

ASD TECHNICAL REPORT

A STUDY OF SUBSTRUCTURE AND CREEP RESISTANCE  
USING NICKEL, WITH PRELIMINARY DATA FOR NIOBIUM

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## FOREWORD

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## ABSTRACT

A correlation obtained between instantaneous creep rate and a quantitative measure of the substructure density at the same time during primary creep of nickel at 1550°F and 3360 psi indicated that the development of substructure was responsible for primary creep. The correlation held for substructures developed at higher stress before testing at 3360 psi. These test conditions were used because substructures were developed which could be clearly delineated by etch pitting. Attempts to use lower temperatures failed because the substructures could not be clearly delineated, due either to incomplete polygonization or to precipitation interfering with the decorator effect of impurities. The results did show that the non-homogeneity of substructures requires both high resolution and the ability to average measurements if meaningful quantitative measurements are to be obtained.

Attempts to correlate substructures with varied creep-rupture properties at 1100°F from hot rolling failed, due to lack of sufficient polygonization for delineation and to interference with the decorator effect by precipitation. Measurable substructures could not be produced for testing at 1100°F by other means due to the interference by recrystallization when the strain rate was reduced sufficiently for polygonization.

Extremely limited data for niobium indicated that creep-rupture properties were influenced by rolling conditions even though there was very little strain hardening in this material.

## PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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## INTRODUCTION

This summary report covers research conducted at The University of Michigan for Materials Central, Wright Air Development Division. The work was authorized under Air Force Contract No. AF 33(616)-5466, and covered the period June 15, 1959 to September 30, 1960.

The general objective of the research was to obtain an understanding of the mechanisms by which hot working can increase the creep-rupture strength of metals and solid-solution alloys. The main effort was expended on the problem of defining the role of substructures induced in nickel by prior rolling and creep. A limited amount of research was carried out to define the effect of hot working on the creep-rupture properties of niobium.

Prior research (Ref. 1) demonstrated that marked increase in the creep-rupture strength of "A" Nickel at 1100°F could be produced by rolling prior to testing. The increase depended mainly on the degree of reduction by rolling, independent of the temperature of reduction up to the point where recrystallization occurred during rolling or during testing at 1100°F. Evidence obtained indicated rapid recovery from strain hardening during testing at 1100°F. The evidence suggested that substructural variation, dependent mainly on the degree of reduction, was a possible explanation of the relationship between rolling and creep-rupture properties. Recrystallization eliminated substructures and thereby removed the strengthening mechanism. In the absence of recrystallization, increased creep resistance was postulated to result from finer substructures developing as the amount of reduction by rolling was increased.

Creep resistance at 800°F had also been shown in Reference 1 to be highly dependent on conditions of prior rolling. In this case, however, strain hardening was an important factor in addition to the mechanism which appeared to control at 1100°F (Ref. 2).

The research of Reference 2 had shown that substructures were visible after etching to develop etch pits only when small reductions by rolling had been made at 1800°F. Creep testing slightly improved

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the delineation of the substructures in those specimens where it was visible to some extent before testing. If a substructure was not visible before testing, it could not be developed after testing. The precipitation of decorator elements, presumably mainly carbon, was indicated by the data as one cause of inability to delineate substructures. The other possible reason seemed to be incomplete polygonization leaving the sub-boundaries too broad and diffuse to properly etch. Other methods of attempting to define substructures, such as X-ray diffraction, and a transmission electron microscopic technique, failed.

Extensive attempts to delineate substructures after varied rolling conditions and after testing at 1100°F offered little hope of progress, wherefore the objective finally turned to finding conditions of creep which would result in substructures which could be delineated by etch pitting and measured to correlate with the creep characteristics.

## EXPERIMENTAL MATERIALS

### Nickel

Two heats of "A" Nickel with different carbon levels (designated here as "higher-carbon" and "lower-carbon" to distinguish them) were supplied gratis by the International Nickel Company. Additional studies used carbonyl nickel vacuum-melted at the University. The reported chemical analyses of all three heats are listed in Table 1.

The 1-inch round bars of the higher-carbon "A" Nickel were rolled 20.1 percent, annealed in air for 1 hour at 1600°F (air cooled), and heated again in air at 1800°F for 1 hour (water quenched).

The lower-carbon "A" Nickel was received as an 0.81-inch round bar. It was cold rolled 66.8 percent, cut into 2-3/4-inch lengths, annealed as a single batch in a salt bath at 1800°F for 2 hours, and water quenched.

The carbonyl nickel was prepared as a 2.5-inch diameter, seven-pound ingot. Type B carbonyl nickel powder was consolidated by induction melting under an argon blanket in a Norton Magnorite (MgO) Crucible. The melt was deoxidized with pure magnesium and cast in a mold of cast iron. The ingot was then remelted in a new MgO crucible under a vacuum of 1-5 microns to remove the excess magnesium and other volatile impurities. Chemical composition (Table 1) was determined using a full-width slug cut from the annealed bar at

a location corresponding to midway between the top and bottom of the ingot. The ingot was hot rolled from 1600°F to a 1-1/2-inch square bar. The bar was then cold rolled 70.1 percent and annealed under the same conditions as the lower-carbon "A" Nickel.

#### Double-Electron-Gun-Melted Pure Niobium

The pure niobium was obtained from the Mallory-Sharon Metals Corporation in the form of 1-inch rounds. A specific chemical analysis was not available for this material, but analyses on other heats of similarly-produced niobium indicate the following impurity ranges: 170-270 ppm of oxygen, 35-100 ppm of nitrogen, and 100-155 ppm of carbon.

The manufacturer reported that fabrication consisted of machining the surface of a 3-inch diameter ingot, forging from argon at 2000°F to a 1-1/4-inch diameter bar, rolling to a 1-inch round, and annealing in vacuo.

## PHASE A: SUBSTRUCTURE STUDIES WITH NICKEL

### EXPERIMENTAL PROCEDURES

The difficulties encountered in delineating substructures in the "A" Nickel, as reported in Reference 2, were reviewed and checked. Based on the results of this step, the conditions required to produce substructures which could be delineated were postulated. Experiments were then carried out to develop substructures which could be measured prior to creep-rupture testing at 1100°F to attempt to define the relation between known substructure and creep-rupture properties.

Due to recrystallization limiting the range of substructures and to other uncertainties introduced by the method of developing substructures, the attempt to relate known prior substructures to creep-rupture properties at 1100°F was not successful. When further experimentation demonstrated the possibility of measuring instantaneous creep rates as a function of instantaneous substructure in specimens exposed to creep at 1550°F, the decision was made to compromise and obtain this information as a guide to the original problem.

The work done for References 1 and 2 utilized an "A" Nickel with impurities including 0.06 percent carbon. Because the impurities could have been a factor, two other grades of nickel were introduced. One was an "A" Nickel with 0.01 percent carbon. The other was made by vacuum melting carbonyl nickel powder in an attempt to obtain nickel with less impurity.

#### Delineation of Substructures

Substructures were delineated by etch pitting and microscopic observation. Specimen preparation consisted of sectioning longitudinally along the center, mounting in bakelite, rough and fine grinding on wet silicon carbide papers, polishing with Linde B levigated alumina on a Syntron vibratory polisher for 6-8 hours, and etching electrolytically in an aqueous, 40-percent  $H_3PO_4$  solution for 6-8 minutes at 2 volts with a current density of 0.2-0.3 amps per square inch. The main function of this etchant under the conditions given was to cause pitting at sites where dislocations were nearly perpendicular to the surface. It has been demonstrated (Ref. 1) that a prerequisite for etch pitting nickel with  $H_3PO_4$  is carbon in the form of Cottrell atmosphere around the dislocations.

## Development of Measurable Preinduced Substructures for Testing at 1100°F.

Analysis of the problem indicated that measurable preinduced substructures could be induced by slow tensile straining at 1600°F. This was checked first by prestraining specimens to about 6 percent at rates of 100, 30, and 10 percent per hour in a tensile machine. Specimens were then prepared for creep-rupture testing at 1100°F by prestraining over the range from 0 to 20 percent at a rate of 20 percent per hour. One series of specimens was cut up and the substructure measured to produce a curve of sub-boundary density versus percent strain. Other specimens were then strained and creep-rupture tested at 1100°F. The original substructure for the latter specimens was estimated from the curve of sub-boundary density versus percent strain.

The specimens used were 0.250-inch diameter. Contaminated surface material was machined off after prestraining before testing at 1100°F.

A counting technique to quantitatively measure substructures had to be developed as detailed in the Results section.

### Creep-Rupture Testing

Creep-rupture tests were carried out both in air and in a high vacuum. Tests at 1100°F and preliminary testing at higher temperatures were done in air. Tests at 1550°F for the correlation of instantaneous substructure with instantaneous creep rate were run in vacuum to avoid the surface cracking and contamination associated with tests in air at this temperature.

All tests in air were conducted in accordance with ASTM Recommended Practices. Specimens were placed in furnaces at the test temperature and four hours allowed to adjust temperature distribution and to obtain precise control over the specimen temperature. The load was applied at the end of this time and creep or rupture time measured as was appropriate. The optical extensometer used for creep measurements had a sensitivity of about 0.000005 inch per inch for the 1-inch gage length of the 0.250-inch diameter specimens used. Measurements were made manually at sufficient intervals to define the creep curves. Rupture times were automatically measured. The individual-specimen creep units feature a simple beam and automatically-controlled furnaces.

The creep tests in vacuo at 1550°F employed a creep unit specially designed to include the high-sensitivity modified Martens

optical extensometer. The vacuum, as measured by a hot-filament discharge gage, ranged from 0.07 to 0.4 microns ( $7 \times 10^{-5}$  to  $4 \times 10^{-4}$  mm) Hg; most frequently, it was about 0.15 microns Hg. The specimen temperature was measured with chromel-alumel thermocouples attached directly to the specimen at the ends and center of the 1-inch gage section. The indicated temperature difference between any two of the three points never exceeded 4°F, and in most cases, did not exceed 1°F. The mean indicated temperature was held to within 3°F of the nominal temperature. New thermocouples were used for each test. Comparisons between new and used couples indicated that the departure from calibration during a creep test was insignificant.

Between 400°F and 1550°F, the heating time was about 2-1/2 hours and the cooling time was about 3-1/2 hours. Usually 3 to 12 hours after the specimen reached 1550°F was required for temperature and vacuum adjustments before the load could be applied.

The stress was applied by direct dead-weight loading. The weights were pre-stacked on a forked extension of a screw-type jack so that the full load could be quickly but gently placed on the weight pan. For stress-reduction tests, the weights were stacked in two tiers so that the forked extension of the jack could be inserted between the tiers to raise the upper tier when it was desired to reduce the stress abruptly.

The load was transmitted through the bottom of the vacuum chamber by means of a sliding vacuum seal whose essential components were a polished stainless steel pull rod and three teflon O-rings held rigidly in a packing gland. No lubricant was used. A constant stress ( $\pm 0.3$  percent of the nominal stress) was maintained at the base conditions by a manual, step-wise decrease of the applied load as the specimen elongated. Pre-creep was done with constant higher load.

Before the use of a vacuum for the tests at 1550°F was selected, preliminary attempts were made to protect the surface of the specimens by using a dynamic atmosphere of dried and gettered helium. Some improvement was obtained, but there was still too much cracking of the specimen surface during a test. Most of this came from the inability to initially out-gas the system. Since proper out-gassing required evacuation of the system, the decision followed to use a vacuum for the tests. The creep rates for tests under vacuum were higher than in air as is the normal effect of a vacuum.

The total strain at the time creep tests were discontinued for measurement of substructures or the amount of strain during slow tensile prestraining was measured in an unusual manner. Three

sets of gage marks were placed on the gage length at 120° intervals using a diamond pyramid hardness tester. The initial and final gage lengths were measured to 0.0002 cm under a microscope with a movable stage, using the corners of the indentations as reference points. The impressions retained identity very well even for tests in air. The optical extensometer used to measure creep was necessarily attached to the shoulders of the specimens. It was therefore necessary to correct for the extension in the shoulders and fillets of the specimens. The overall strain measurements were used to establish and check the correction as well as the conversion factor used to calculate creep from the extensometer readings.

## RESULTS

Data are presented first for the further evaluation of the delineation and quantitative measurement of substructures. Results of the attempt to produce known initial substructures for creep-rupture testing at 1100°F are then presented. Finally the main results show the correlation between instantaneous creep rate and a measure of the mean substructure for creep at 1550°F and 3360 psi stress.

### Delineation of Substructures.

In view of the past difficulties in delineation of substructures, the problem was restudied. Experience indicated that the only practical way at this time to study substructures in relation to creep of nickel was to delineate the substructures by an etch pitting technique which offered the necessary degree of resolution and sensitivity and was able to average the rather non-uniform substructures. The literature reports that successful delineation by etch pitting requires a well-polygonized structure with consequent sharp sub-boundaries. In addition, very few cases of successful delineation are recorded unless impurity atoms are present to "decorate" the sub-boundaries. Etch pitting experiments on nickel by Guard (Ref. 3) showed that carbon had to be available as a decorator element for successful etch pitting with  $H_3PO_4$ .

Samples of the higher-carbon "A" Nickel from the work of Reference 2 were restudied but the causes for inability to decorate substructures in the as-rolled material or in the specimens after creep testing at 1100°F were not definitely established. Apparently both a precipitation reaction removed the decorator effect and also in many cases the polygonization was not sufficient to yield sharp boundaries. The following features were noted:

1. All samples were air cooled from 1600°F prior to rolling. The considerable precipitate after this treatment is assumed to be at least partly responsible for inability to etch sub-boundaries. This was particularly true for specimens subjected to creep without any rolling.
2. As best as could be judged from the experience of this investigation, all samples rolled at temperatures below 1600°F had sufficient precipitation present to interfere with the decorator effect.
3. Experiments demonstrated that the temperature required to dissolve the precipitate in one hour was 1475°F. On this basis the precipitate in the samples rolled at 1600°F and 1800°F should have been dissolved by heating for rolling, and the impurities present in a form suitable for decoration. The only samples in the as-rolled condition in which anywhere near complete delineation of substructure was obtained, however, were those reduced less than 10% at 1800°F. Creep testing at 1100°F somewhat improved delineation, as was shown in Reference 2.
4. Careful re-examination of these specimens indicated considerable precipitation although the precipitate was less than that present in samples rolled below 1600°F or in samples creep tested without rolling. Apparently the rolling partially retarded the precipitation reaction and was most effective in those samples reduced small amounts at 1800°F.
5. The data suggested that if the rolling conditions were such that fairly complete polygonization could occur, the decorator effect was retained. Possibly the diffusion of the decorator element to the sub-boundaries retarded precipitation. If polygonization was incomplete, the consequent precipitation as well as the absence of sharp boundaries prevented delineation. Any tendency for polygonization during testing at 1100°F was apparently masked by the precipitation reaction in the absence of fairly-complete initial polygonization since testing only improved delineation when some substructure could be delineated before testing.

On the basis of these observations it was concluded that:

- a.) Fairly-complete polygonization during rolling would be necessary for delineation of substructures prior to creep



testing. This was realized only for small reductions at 1800°F. Larger reductions at 1800°F or reduction at 1600°F did not yield the necessary degree of polygonization even though decorator elements were in solution.

- b.) The absence of initial polygonization, in combination with precipitation interfering with the decorator effect, prevented delineation of substructures after creep testing at 1100°F. The degree to which incomplete polygonization during testing and the precipitation effect were responsible was not established. Indications were that diffuse sub-boundaries were at least as much a factor as the lack of decorator and could have been the major factor.
- c.) Delineation of substructures sufficiently well for quantitative measurement would require conditions which would result in a well-polygonized structure as well as retarding precipitation. This consideration pointed to the necessity of using slower rates of deformation than rolling, such as creep at sufficiently high temperature for polygonization to occur with the decorator in solution.

At this point the diffusion treatment at 932°F (500°C), reported necessary by Guard (Ref. 3) in his work with "A" Nickel to achieve adequate decoration, was checked for a number of samples. No improvement resulted for those samples showing precipitation. Higher diffusion temperatures were then tried to determine if decoration improved. Delineation was extended somewhat, but was still not satisfactory up to 1500°F when recrystallization interfered with the process. There was also considerable question as to whether or not the higher-temperature treatments were altering substructures. The lack of complete delineation and recrystallization indicated that the diffusion treatments should be abandoned.

The next step was to investigate exposure conditions which would result in substructures which could be delineated. Both slow tensile straining at 1600°F and creep at temperatures higher than 1100°F were studied.

Samples of the higher-carbon "A" Nickel were tensile strained at 1600°F to 5.38 to 6.18 percent at rates of 100, 30, and 10 percent per hour. Substructures could be delineated in all the samples.

To investigate use of creep to develop structures which could be etched, specimens of the higher-carbon "A" Nickel were water quenched from 1800°F to prevent precipitation and then exposed to creep as follows:

- (a) Exposed to a stress of 11,000 psi at 1100°F until the creep strain was 4.1 percent. There was no visible substructure but considerable general precipitation. Presumably the precipitation preventing decoration occurred before the substructure developed sufficiently during creep to maintain the decorator effect.
- (b) Exposed at 1350°F under 5250 psi until the creep strain was 1.42 percent. Bands of areas free from precipitation showed substructure.
- (c) Exposed at 1500°F under 3325 psi until the creep strain was 3.48 percent. The substructure could be clearly delineated but was incomplete along grain and twin boundaries. Precipitation was limited to grain and twin boundaries and presumably prevented decoration in the adjacent matrix.

Three further tests were then undertaken at 1550°F at a stress of 3000 psi to creep strains of 0.56, 1.91, and 3.53 percent deformation. The substructure was well developed after 1.91 and 3.53 percent deformation and extended up to the grain boundaries so that there was only an insignificantly narrow band where the substructure was not delineated. Substructure did not extend throughout each grain after 0.56 percent strain. The number of small subgrains appeared to increase up to 1.91 percent deformation without much change in size. Further straining appeared to cause subdivision of subgrains formed earlier.

The specimens tested to the larger deformations were subject to extensive intergranular cracking at the surface. There was very little cracking when the creep was limited to 0.56 percent. The two larger deformations exhibited about 40 cracks per inch, averaging 0.0018 inch in depth, with some cracks as deep as 0.005 inch. The surface layers were also depleted of decorator atoms so that the substructure would not etch in these areas.

The intergranular cracking and reactions at the surface of the specimens tested at 1550°F indicated that if creep at 1550°F were to be used further in the investigation some means of protecting the surface would be necessary. After preliminary experiments had shown that a dynamic atmosphere of purified helium would not work properly in a standard unit converted from air testing, the decision was made to conduct the creep tests in vacuo. Vacuum testing remedied the cracking and decarburization problems, as demonstrated by Figure 1.

At this point the carbon contents of the lots of nickel used by Guard were noted to be 0.015 and 0.007 percent. These were well below the 0.06 percent carbon content of the "A" Nickel being used in the present studies. To check whether the difficulties encountered from precipitation at lower test temperatures might be due to the relatively high carbon level, a heat of 0.01 percent carbon "A" Nickel was obtained. Creep tests (Fig. 2) using this material were conducted at 1300°, 1400°, and 1500°F. The microstructures after creep (Fig. 3) showed only a small improvement over the higher-carbon "A" Nickel. Careful examination indicated that precipitation was still restricting the delineation of substructures. The delineation became more complete as the creep temperature was raised. By testing at 1550°F the interference from precipitation (Fig. 3d) was limited to extremely narrow bands on either side of the grain boundaries.

These experiments indicated that substructures suitable for measurement could be obtained either by slow tensile straining at 1600° or by creep at 1550°F in vacuo.

#### Measurement of Substructures

With conditions established for producing substructures that could be delineated, attention was directed to the problem of quantitative measurement. In every case it was noted that the substructure was variable within individual grains and between grains. For this reason the measuring method was selected to yield a mean value. In previous work it had been determined that x-ray diffraction methods either had insufficient resolving power or else high resolution techniques presented a nearly unsurmountable task to arrive at a mean value. Some promise was found for transmission electron microscopic techniques giving the necessary resolution; however, not only were they very difficult, but the small volumes involved presented the problem of obtaining a useful average measurement. The achievement of conditions of exposure leading to substructures that could be well delineated by etch pitting indicated that the best procedure would be to measure the substructure thus delineated by microscopic methods, even though the exposure conditions were considerably different from the original rolling.

The procedure selected was to count the number of sub-boundaries using a lineal intercept technique (Ref. 2). The first step was to determine the effect of etching time (Fig. 4). The rate of etching could be controlled and a point was reached beyond which no additional boundaries appeared with further etching.

Further experimentation indicated that reproducible counts of boundaries resulted for individual specimens by using three traverses along parallel lines 0.05 inch apart across the 0.250-inch diameter specimens at an angle of  $22.5^\circ$  to the tension axis. Each traverse covered the full width of the specimen. The observations were made at a magnification of 600 diameters. The total number of boundaries counted, including twin and grain boundaries, was averaged by dividing by the length of traverse and was reported as the mean boundary density in boundaries per inch.

In those cases where partial recrystallization occurred, separate measurements of sub-boundaries in un-recrystallized grains were made. The whole polished surface was then measured to determine precisely the percentage of recrystallized grain area. The overall mean number of boundaries per inch was taken as the weighted mean of the two measurements.

The procedure of counting the number of boundaries and using the mean value results in a number which is very nearly equal to the reciprocal of the subgrain size (distance across the subgrains). The grain and twin boundaries were included in the counts because they should function in the same way as sub-boundaries. In early work before the final procedure was developed only sub-boundaries were counted.

#### Relation of Pre induced Measurable Substructures to Rupture Properties at 1100°F.

A series of specimens of the higher-carbon "A" Nickel with varying amounts of strain was prepared by straining at 1600°F at 30 percent per hour in a tensile machine. Some of the specimens were used to measure the mean sub-boundary density as a function of amount of strain (Fig. 5). The other specimens were remachined and subjected to creep-rupture testing at 1100°F and 20,000 psi. The curve of Figure 5 was used to estimate the initial mean substructure of the specimens subjected to rupture testing. The replicate points on the curve are the results of repeated measurements to show the degree of reproducibility.

The rupture times at 1100°F and 20,000 psi (Table 2 and Fig. 6) increased from 9 up to about 30 hours as the sub-boundary density increased to 800 per lineal inch. This increase was associated with prestrains of about 2 percent. The next higher level of prestrain studied (about 5.5 percent) caused a large increase in the number of sub-boundaries present but no change in rupture time. Beyond 5 percent prestrain, recrystallization began to occur during the

prestraining operation with a resultant decrease in rupture time, to a plateau at about 15 hours. The lack of a corresponding increase in rupture time with increasing sub-boundary density in the range of 800 to 2200 per lineal inch was unexpected.

Partial recrystallization could be identified in all specimens before rupture testing except those prestrained 1.68 and 5.37 percent. The sub-boundary density levelled off for greater strains due to progressive recrystallization with increasing prestrain.

In preparing the data for the curves of Figure 6, the following factors were noted:

1) For a strain of 1.74 percent, subgrains were found mainly at grain corners, in bands parallel to and near grain boundaries, and in bands across grains. The sub-boundary density was high in these areas. Localized areas containing subgrains were, however, visible in all grains along the length and from surface to center of the specimens.

2) Sub-boundaries were visible throughout each grain for a strain of about 5 percent. The subgrain size varied from grain to grain as well as from point to point within a grain.

3) Those grains which did not recrystallize during larger amounts of straining exhibited increasing sub-boundary density with increasing strain.

4) When recrystallization occurred, all traces of the substructure were removed from the grains which had recrystallized. Some recrystallized grains in specimens given the larger strains apparently developed substructure due to continued straining after recrystallization had occurred.

Relative line-breadth measurements established that strain hardening of the type that broadens X-ray lines was not produced by tensile prestraining at 1600°F at a rate of 30 percent per hour. A diffraction pattern was taken in the back-reflection region from a specimen strained 19.7 percent. An annealed specimen was given an identical exposure. To ensure equivalent photographic processing, the two exposures were made on the same sheet of film. Profiles of the (420) diffraction lines were then taken with an automatic recording microphotometer, and the widths of the profiles (Fig. 7) were compared at half-maximum intensity. As indicated by the two-headed arrow in Figure 7, the widths were equivalent.

The results of the rupture tests suggested that the first subgrains to appear during slow straining at 1600°F were effective in increasing

rupture life at 1100°F. Those which developed within the grains during straining to larger amounts were apparently ineffective. The results agreed with those previously obtained showing that if any recrystallization occurred during working (or during testing after working), rupture strength at 1100°F either decreased or levelled off.

Comparison of the present data with that in Reference 1 shows that rupture life at 1100°F and 20,000 psi can be increased much more by hot rolling than by slow tensile straining at 1600°F. In both cases recrystallization limited the amount rupture life could be increased by prior strain. Another difference with slow straining was the absence of an increase in rupture life when the strain was increased from 1.67 to 5.37 percent without recrystallization being detected either before or after testing. Even though no recrystallization was visible after 5.37 percent strain, it was apparently ready to start and this apparently limited strength even in the presence of a fine substructure. The much shorter time periods involved in the rapid deformation of hot rolling allowed considerably-larger strains to be introduced without recrystallization than was the case for slow tensile straining. Furthermore, substructures observed in bars hot rolled at 1800°F were very much finer than those developed during slow straining at 1600°F.

The possibility was recognized that slow tensile straining at 1600°F might be sufficiently similar to the creep process to restrict rupture life by creep damage. For structurally-stable materials, if a certain percentage of creep life is used up at one set of conditions, the subsequent life at another set of conditions is known generally to be reduced. A major factor in the results could have been that loss in rupture life offset the expected increase from substructure formation. This could account for the lack of an increase in rupture life for the increase in substructure in the specimen prestrained 5.37 percent.

In view of the complications due to recrystallization and the uncertainties due to possible creep damage, slow tensile straining was dropped as a means of producing measurable initial structures before testing at 1100°F. An additional major factor in this decision was the very limited range of strains and substructures which could be introduced before recrystallization, compared to those involved in the prior rolling program.

#### Delineation of Substructural Changes During Creep

At the same time the tests were being carried out at 1100°F on specimens prestrained at 1600°F, tests were also conducted to determine if changes in substructure during creep were properly reflected

in the substructures visible after creep. Specimens were prestrained at 1600°F at 30 percent per hour and exposed to creep as follows:

<u>Prestrain at</u> <u>1600°F</u>	<u>Subsequent Creep Exposure</u>			
	<u>Temp.</u> <u>(°F)</u>	<u>Stress</u> <u>(psi)</u>	<u>Time</u> <u>(hrs)</u>	<u>Strain</u> <u>(%)</u>
6.06	1200	6950	93	1.37
5.93	1350	3325	70	1.42
5.32	1500	3325	18	1.20
0	1500	3325	20	3.48

When examined after creep the following changes in substructure were noted:

- (1) After creep at 1200°F, the substructure was not visibly changed from the initial condition. Counts of sub-boundaries, however, indicated a boundary density reduction of 23 percent during creep.
- (2) The prestrained specimens exhibited noticeable increase in sub-grain size after testing at 1350° and 1500°F. The final sizes were the same for both temperatures.
- (3) The material without prestrain tested at 1500°F had developed a substructure similar to the prestrained specimens tested at 1350° and 1500°F under the same stress.
- (4) There was little evidence of general precipitation interfering with decoration in the prestrained samples. Precipitation in the specimen creep-tested at 1500°F without prestrain was limited to grain and twin boundaries.

Apparently the decorator elements did move with the changes in substructure during creep and continued to permit delineation of the changed substructure by etch pitting. It seemed evident from the data that changes during testing at 1100°F in an originally well developed substructure would be at best very slow and probably slight. The similarity of substructures with and without prestrain, after testing at 1500°F, suggested that prior induced substructures would not be sufficiently stable to have much effect at such high temperatures and that substructures characteristic of the creep conditions would control. The structures being similar after tests at 1350° suggested the main dependence on stress of substructures formed by creep.

## Instantaneous Creep Rate versus Substructure During Creep at 1550°F.

Based on the prior reported experience, the decision was made to attempt to produce measurable substructures by creep at 1550°F. The lower carbon "A" Nickel then available was substituted for the higher carbon material previously used on the basis that it might show less effect from impurities and less interference with decoration as a result of precipitation. The vacuum-melted carbonyl nickel also became available and was included in an effort to obtain some indication of the effect of impurities. A temperature of 1550°F was selected as the lowest at which reasonably complete delineation of substructures could be obtained.

The difficulty from recrystallization in specimens tensile pre-strained at 1600°F to produce measurable initial substructures, plus the limited range of substructures, indicated that the same limitations might be present in specimens in which creep was used to produce known prior substructures for subsequent creep testing. In addition, the probability that prior creep would be using up rupture life had not been resolved for rupture testing at lower temperatures. In view of these uncertainties and the fact that the substructures developing during creep at 1550°F seemed closely related to the creep at that temperature, the relations between creep and substructures were chosen for study at 1550°F. The choice seemed to afford about the best conditions for obtaining a quantitative measure of creep as related to substructures and would yield useful results even though the specimens might not be suitable for lower-temperature investigations. The data offered a unique opportunity to investigate the relation between instantaneous creep rate and the substructure at that time, with the possibility of correlating substructure formation to decrease of creep rate with time.

The stress selected for the creep exposure at 1550°F was 3360 psi. This was fairly arbitrary. The tests had to be carried out under high vacuum with limited equipment. It was therefore desirable to keep the time short. The prior experiments had indicated that creep at this temperature and stress would in the relatively short times result in substructures well defined by etch pitting. Because considerable emphasis was to be placed on the influence of substructures on creep at 1550°F, it was desired to minimize any contribution to creep by grain boundary sliding or migration. A fairly short rapid test would be a step in this direction. Likewise it was desirable to avoid excessive deformation on loading. The selected stress was a compromise to meet these requirements.



Tests were terminated at varying amounts of creep strain covering the range from decelerating through minimum into accelerating creep. The several tests with no prior deformation gave creep curves which could be represented by one curve (the lowest curve of Fig. 8 for the lower-carbon "A" Nickel and of Fig. 9 for the carbonyl Nickel). Specimen numbers adjacent to the curves indicate the point at which tests were terminated for substructure measurement with the results listed in Table 3 and shown as a band on Figure 10.

A straight line correlation in the form of a narrow band (Fig. 11) resulted when the log of the instantaneous creep rate was plotted against the mean boundary density at the same time. The creep rates decreased with increasing boundary density. Creep rate at any time in the tests at 3360 psi at 1550°F was associated with a definite value of the mean number of boundaries measured. The correlation applied equally to the "A" Nickel and the carbonyl nickel. It should be noted that the correlation held for the period of decreasing creep rate and even held during the increasing creep rate when recrystallization was occurring.

#### Stress Reduction Check of Correlation Between Instantaneous Creep Rate and Substructure.

The correlation between instantaneous creep rate and substructure density suggested that the development of a substructure was the cause of varying creep rate with time. If so, a method of producing different substructures and then measuring the creep rate at 3360 psi ought to show adherence to the correlation. This was done by producing substructures at a higher stress and then reducing the stress to 3360 psi and measuring the creep rate immediately after the stress was reduced. This procedure varied both the time and strain associated with a given substructure from that of the constant stress tests at 3360 psi. Because finer substructures would be produced at the higher stress it also extended the range of the correlation toward lower creep rates.

Tests were started at 5360, 4360, and 3860 psi. When the strain attained predetermined values, the stress was abruptly reduced to 3360 psi and the creep rate measured immediately after the reduction. Replicate specimens were exposed at the higher stress in the same way but cooled directly to room temperature and the sub-boundaries counted. The tests at 3360 psi after creep at a higher stress were terminated when the creep rate began to increase and the sub-boundary density in the specimens was then counted. The creep curves obtained are included in Figures 8 and 9. The replicate tests are shown as single curves. The creep rates immediately after the stress was reduced (Table 3) were measured in a way intended to eliminate or minimize any effects of substructure changes after the stress was reduced to 3360 psi. Straight lines were drawn through the plotted creep-time data (Fig. 12) immediately after the stress was reduced. The first point was

ignored if it did not fit on the straight line. These deviations did not appear to be related to recovery because they were erratic.

The replicate tests at the higher stresses could not always be terminated at exactly the same strain. The actual measurements were plotted at the top of Figure 10 and then corrected to the strain level of the stress-reduction creep test by a line parallel to the band for 3360 psi. Arrows in Figure 10 and parentheses in Table 3 indicate the corrected values. The corrections were small.

The creep rates and sub-boundary densities immediately after the stress was reduced fitted the correlation (Fig. 11) of the instantaneous creep rates and sub-boundary densities for constant-stress tests at 3360 psi. The creep rates and structures measured at the termination of creep at 3360 psi in the stress-reduction tests also fell on the correlation band. Although the range in substructures for a given pre-strain was small, the adherence to the correlation indicates that the substructure was controlling instantaneous creep rate, and not some function of time or strain. The mean number of boundaries was associated with a definite creep rate at 3360 psi and 1550°F, independent of stress, testing time and total strain used to produce sub-boundaries under the limited range of conditions investigated.

In the stress-reduction tests on carbonyl nickel the creep rates at 3360 psi tended to converge with increasing time at 3360 psi to about the same value that was being approached in the longest creep test run at 3360 psi without prior strain (Fig. 13). The associated substructures, however, were all so near the value for the largest strain of the constant-stress test at 3360 psi that difficulty was encountered in detecting substructural differences. The creep rates after stress reduction changed quite rapidly, indicating that the substructures were changing rapidly to those characteristic of the creep condition. This indicates that the constant-stress test was approaching an equilibrium value for secondary creep. The finer substructures of the stress-reduction tests apparently bracketed the equilibrium substructure. In the stress-reduction tests on lower-carbon "A" Nickel, partial recrystallization prevented development of data related to equilibrium creep rates and substructures.

#### Description of Creep Characteristics and Substructures

The creep curves for the "A" Nickel at 3360 psi show decreasing creep rates to strains of about 4 percent (Fig. 13). At this point the creep rates increased as the result of the start of recrystallization. Recrystallization also caused the stress-reduction tests to increase in creep rate after creep at 3360 psi was allowed to continue (tests 16, 13, and 18 in Fig. 8). For this reason true secondary creep was not attained in the tests.

The carbonyl nickel had a slightly lower creep rate for a given strain (Fig. 13) than the "A" Nickel, presumably due to different amounts of yielding on loading. It was less subject to recrystallization and as discussed previously approached a constant creep rate at the highest strains studied. At this point it also was beginning to recrystallize. Due to its greater resistance to recrystallization, it did attain a higher sub-boundary density than the "A" Nickel.

The development of substructure in the lower-carbon "A" Nickel during creep at 1550°F and 3360 psi is summarized as follows:

- (1) Sub-boundaries appeared first at grain corners and other points which presumably were regions of localized strain concentration within the grain structure. For a total strain of 1.15 percent these areas could be seen in all the grains (Fig. 14 a).
- (2) When the total strain reached 2.04 percent (Fig. 14b) subgrains could be seen throughout most of each grain. The density was highest at the points where they first became visible and was relatively low in the remainder of each grain.
- (3) Additional creep (Fig. 14 c, d, e) resulted in a further increase in the mean boundary density (Table 3). This apparently occurred mainly in those parts of the grains which originally had low sub-boundary density. The subgrain in the areas which originally exhibited a substructure apparently attained a minimum size and then increased in size (Fig. 15).
- (4) At about 4 percent strain or a little less, recrystallization began to appear. The recrystallization wiped out the substructure in the recrystallized area. The unrecrystallized grains continued to increase in boundary density (Table 3) while other grains were recrystallizing. After the larger deformations, new sub-boundaries could be seen to be forming in some of the recrystallized grains. Evidently, continued creep after recrystallization introduced enough new strain into the recrystallized grains to reform a substructure. Figure 14e shows unrecrystallized grains at the left, a new recrystallized grain with no substructure at the upper right and a recrystallized grain with reformed substructure as a result of creep subsequent to its formation at the right of the photograph.

The structures in the stress-reduction tests followed the same qualitative trends discussed for the constant-stress tests at 3360 psi. Figure 16 shows the microstructures developed during 3.66 to 3.88 percent strain in varying times under varying stresses. The structure became noticeably finer with increasing stress and strain rate. Conceivably, the longer times of the lower-stress tests produced coarser structures through subgrain growth.

The carbonyl nickel exhibited substructures similar to the lower-carbon "A" Nickel. The main difference noted was the larger strains required to induce recrystallization. The sub-boundary density appeared to slightly higher for a given strain, although the overlap with the data for the lower-carbon "A" Nickel was sufficient that a real difference was not established. The curves (Fig. 10) suggest that an equilibrium boundary density was being approached. This corresponded with the creep data which suggested that a constant creep rate was also being approached. Apparently the greater resistance to recrystallization of the carbonyl nickel permitted a closer approach to secondary creep than for the "A" Nickel.

As may be seen in Figure 14 the substructure was angular. After considerable creep (or after the longer times for creep) there was a tendency for the subgrains to approach an equiaxed form and be more uniform. There also seemed to be less variation in substructure between grains at this stage.

All of the observations of substructure as developed by etch pitting with  $H_3PO_4$  emphasized the problem of obtaining a meaningful measure of the substructure. The number of boundaries varied widely from place to place within individual grains and between grains. The size of the substructure was such that measurement required a resolution of better than 0.0003 inch of a non-uniform distribution. The method of measurement however had the necessary resolving power and provided a means to obtain a meaningful average.

#### Interpretation of Correlation Between Substructure Density and Creep Rate at 1550°F and 3360 psi.

The correlation suggests that as creep proceeded it occurred by a mechanism which caused a substructure to form. This substructure reduced the ease of creep so that creep rate decreased with increasing substructure density. The adherence of the stress-reduction specimens to the correlation suggests that the substructure was the controlling factor. Furthermore the correlation held for two heats of nickel made from different raw materials and melted under quite different conditions.

The restriction of the data to one stress and temperature is clearly recognized to be a serious limitation on the generality of the results. Certainly a wider range of test conditions should be checked before the generality of the correlation can be accepted. The theory and experimental results in the literature indicate that a constant substructure should be produced leading to the constant creep rate of secondary creep. Unfortunately the occurrence of recrystallization prevented a check of this for the experiments conducted. The data for the carbonyl nickel indicated that this was being approached but was not sufficient for verification. It would have been very useful to have been able to verify the method of measuring the substructure by showing a constant substructure and creep rate for secondary creep.

In the stress-reduction tests, there was not a period of low creep rate or continued decrease in strain after the load was reduced. In many cases where an applied stress is reduced, a period of decreasing strain is encountered and represents a period of recovery. Evidently, significant dislocation pile-up was not occurring during the high-stress exposures. Presumably under these conditions of creep, all the dislocations moved to the sub-boundaries and reacted in a way which resulted in the substructure being the main factor influencing creep. There was consequently no period of low creep rate immediately after the stresses were reduced to 3360 psi while piled-up dislocations were being reduced by climb to the proper level for the stress and temperature. The correlation therefore represents a case where dislocation pile-up was not a factor in the creep rate, and the creep rate immediately after stress reduction fell on the curves of Figure 11. Presumably at lower temperatures (or faster rates of strain) where polygonization could not occur as completely, the dislocation pile-up during creep would be an additional factor.

The creep rate according to the correlation of Figure 11 was very sensitive to the changes in substructure. A two-fold change in the mean number of boundaries was associated with approximately a 100-fold change in creep rate. The correlation indicated an exponential relation between the mean number of boundaries (or the reciprocal, which is the mean interboundary distance.) If creep is considered as a thermally activated process, an expression of the following form (Ref 4) applies:

X

$$\dot{\epsilon} = Ae^{-\Delta H/RT} Z(\sigma, S) \quad (1)$$

where  $\dot{\epsilon}$  = creep rate  
 $\Delta H$  = energy of activation  
 $R$  = gas constant  
 $T$  = absolute temperature  
 $A$  = strain per unit time per activation  
 $Z$  = functional dependence of creep rate on stress,  $\sigma$ , and structure,  $S$ .

The activation energy for creep in a given material has been shown to be independent of temperature in the high temperature range. Thus, in the present study, where temperature and stress were constant Equation (1) can be written as

$$\dot{\epsilon} = a Z(S)_{\sigma} \quad (2)$$

where  $a = Ae^{-\Delta H/RT}$  for the conditions of the tests.

The correlation of Figure 11 may now be used to provide an empirical value for  $Z(S)_{\sigma}$  in Equation (2). The plot fits an exponential relationship, giving  $Z(S)_{\sigma}$  the following form:

$$Z(S)_{\sigma} = e^{-\delta/d} \quad (3)$$

where  $d$  = mean inter-boundary distance  
 $\delta$  = a constant of correlation with units of length.

The relationship between creep rate and structure (Equation 2) thus takes the form

$$\dot{\epsilon} = ae^{-\delta/d} \quad (4)$$

The values of  $a$  and  $\delta$  can be calculated for the line on Figure 11. The value of  $a$  is the intercept the line would have if extrapolated to zero boundaries. From the curve shown in Figure 11,  $a$  is about 1530 %/ hour

Analysis of the slope of the correlation shows that  $\delta$  has a value of  $3.2 \times 10^{-3}$  inch or about 100 micron. Equation (4) can thus be written:

$$\dot{\epsilon} = 1530e^{-3.2 \times 10^{-3}/d} \quad (5)$$

*is expressed in per cent per*  
 where  $\dot{\epsilon}$  ~~has units~~ <sup>hour</sup> and  $d$  is measured in inches.

The attachment of physical significance to the values for the constants  $\alpha$  and  $\delta$  seems unwarranted without considerably more study than was possible under this investigation.

The results as previously discussed suggested that essentially all the dislocations were moving to and being assimilated by the sub-boundaries to form a well polygonized structure. This is supported by the low number of etch pits within the subgrains (Fig. 13, 14, and 15). It is likewise considered unlikely that grain boundary sliding was a significant factor. Otherwise the correlation between constant-stress creep and stress-reduction tests would not have been as good. Again it would be recognized that the correlation would not be expected to hold under conditions where grain boundary creep was a major factor.

In the present study, the sub-boundaries in many grains were observed to be largely parallel and lying at an angle of 45 degrees to the tension axis (Figs 15 and 16). This suggests a very close relationship between the sub-boundaries and the planes of coarse slip in the material.

The role of impurities in the heats of nickel studied was not clear. The impurities could be particularly important. The carbonyl nickel heat was included in the present studies to provide a material with the lowest practical level of impurities for comparison with the lower-carbon "A" Nickel being studied. Although the results from these two materials did not indicate that any difference existed between their response to substructure formation, impurities were not ruled out as a contributing factor. This resulted largely from the rather small differences in purity level actually represented by the two materials. Work on a material of still higher purity would be necessary to define with certainty the relative role of substructure and impurities on the creep process.

## GENERAL DISCUSSION

The original objective of this investigation was to explain increased creep-rupture strength from hot working, with emphasis on strengthening at temperatures when recovery from cold work appeared to take place quickly. In more detail, an explanation was sought for the

increase in creep-rupture strength of "A" Nickel at 1100°F with amount of reduction by rolling, virtually independent of the temperature of rolling so long as recrystallization did not occur during working or during testing. The investigation had been undertaken under the hypothesis that creep-rupture properties at 1100°F were probably controlled by recovery effects at 1100°F leading to substructures dependent on amount of reduction.

Extensive effort failed to produce a technique which would delineate the substructures sufficiently well either prior to or after creep at 1100°F to yield a test of the hypothesis. Attempts failed to avoid the difficulties of delineating substructure by preinducing well-polygonized substructures with decoration impurities available by slow straining above the solution temperature of a precipitate. When the strain rate was reduced, recrystallization limited the amount of strain which could be introduced without recrystallization to rather small amounts. Lower temperatures of straining introduced either precipitation which interfered with decoration or incomplete polygonization. Specimens with preinduced substructures formed during creep at 1550°F were not tested at 1100°F for two reasons: The range of substructures was almost identical to those produced by the slow tensile straining at 1600°F which were unsuccessful. Secondly, time was not available to do this and complete the more promising correlation between substructures and creep-rate at 1550°F. In addition to the limited range in substructures, slow straining resulted in substructure sizes which were about 10 times as large as those in samples reduced 10 percent by rolling at 1800°F. The assumption is that the sizes after the larger reductions by rolling which were of much interest were even smaller. In spite of extensive experimentation, no way was found to introduce measurable prior substructures equivalent to those formed during rolling or during subsequent creep at 1100°F.

The correlation of creep rate at 1550°F and 3360 psi to subboundary density represents a marked step forward in the understanding of the relation of substructures formed during creep to the creep resistance. Hypotheses in the literature (Ref. 4 for instance) have indicated that at high temperatures where the activation energies of creep and self-diffusion are equivalent, the deceleration of creep during primary creep was due to substructure formation. The results of this investigation verify this hypothesis. Verification was possible for two reasons. Most important was the combination of high resolution plus the ability to average to a meaningful quantitative measure of the very non-uniform substructures. Secondly, the experimental conditions resulted in rather complete polygonization with sharp sub-boundaries being the predominate factor influencing creep.



The reason why the role of substructure in creep-rupture properties at 1100°F after hot rolling could not be established is still uncertain. In so far as the experience of this investigation is concerned, it now appears that:

- (1) The recovery effect at 1100°F, while it removed the usual manifestations of cold work, probably did not result in well-polygonized, sharp sub-boundaries which could be etched. Precipitation of decorator impurities appeared to be another major factor and possibly the controlling factor. The successful experience in developing substructures by creep seems to be the strongest evidence that incomplete polygonization was the major reason that substructures in hot-rolled nickel could not be delineated by etch pitting.
- (2) If incomplete polygonization was the controlling factor, the inference would be that diffuse boundary substructures would be more effective in resisting creep than sharp boundary structures. Thus the degree of perfection of the substructure as well as the size of the subgrains would be a major factor.
- (3) The completeness of polygonization seems to be allied with stability of substructure effects. If a material is cold worked this is very effective in inhibiting creep so long as the cold work is stable. If recovery can occur rapidly during testing or if working takes place under conditions where partial polygonization can occur, the diffuse substructures are effective to higher temperatures due to greater stability. Apparently in this latter state the main factor is the amount of strain from which the substructure forms. At lower creep temperatures the amount of cold work is an added factor. At higher creep temperatures, polygonization occurs during testing. At sufficiently high temperatures the characteristic substructure for the creep conditions becomes the controlling factor and prior history (or substructure) should no longer be a factor other than its effect on primary creep. If the prior history should introduce an initial substructure characteristic of the creep conditions, the creep test should show constant creep rate from the start of the test. If a finer structure were introduced before creep, there ought to be an initial period of accelerating creep rather than decreasing creep while the substructure grew to the characteristic or equilibrium size. All of these hypotheses assume that recrystallization would not interfere.

- (4) On the basis of the inferences from the results obtained, the temptation is to guess at the role of impurities in creep (as separate from a decorator effect). Many results of creep experiments outside of the present study suggest that there can be a strong interaction between alloying elements and substructures which multiply creep resistance. Heretofore the investigation had been carried out with the concept that stabilization of substructure would be a logical explanation. At the present time it seems more probable that the alloying elements might inhibit polygonization and stabilize the diffuse boundary condition which seems to have high creep resistance.

Actually the experimental results simply showed that any substructure condition developed by prior hot working or by recovery at 1100°F would be very stable at 1100°F. The temperatures for creep had to be increased considerably before there was a significant change in preinduced substructures. Unfortunately the conditions under which measurable substructures could be developed before testing at 1100°F were extremely limited so that a worth-while check of the influence of prior substructure could not be made at 1100°F.

The indications are that the measurement of substructure used was at least a reliable relative measure of the true substructure. Without definite proof, it is thought that this measure approached the true, absolute values for the specimens exposed to creep at 1550°F. One may recognize, however, in view of the problems of obtaining clear delineation of substructures that measurements could be in error due to lack of proper conditions for delineation when the substructures appeared to be incomplete. In other words, when delineation was obviously incomplete no proof was obtained whether this was due to the absence of a substructure or inability to delineate the structure by the etch pitting.

The influence, if any, of grain size on the results was not noticed. The lower-carbon "A" Nickel had a slightly larger grain size (mean diameter 0.012-inch) than the carbonyl nickel (mean diameter 0.008-inch.) The main difference between these two materials seemed to be less deformation during loading and more creep strain at 1550°F to induce recrystallization for the carbonyl nickel.

The results of this investigation of substructures and creep properties of "A" Nickel point to the need for a more detailed study of the type of structure with emphasis on imperfectly polygonized structures as important in the engineering creep temperature range. This type of an approach seems to be the way to establish the influence of prior history and compositional effects which are not now understood.

## CONCLUSIONS

The decreasing creep rates of primary creep of nickel at 1550°F under 3360 psi appeared to be due to the formation of a substructure. Increasing completeness and fineness of substructure correlated with decreasing creep rate. Recrystallization prevented a check on the constancy of substructure during secondary creep.

Because the correlation between instantaneous creep rate and substructure size held for a limited range of substructures induced at higher stresses, the indications are that the substructure was the controlling function for creep rate.

Substructures were found to be very fine and non-uniform. The success of the correlation was due to a method of measurement which combined high resolution with meaningful averaging.

The original objective, to establish the mechanism by which hot rolling increased creep-rupture strength at 1100°F, was not attained. Apparently both incomplete polygonization to sharp sub-boundaries and suppression of the decorator effect of impurities by precipitation was responsible for the inability to delineate substructures. Attempts failed to induce measurable substructures prior to creep at 1100°F. Such substructures were only produced by slow straining at rather high temperatures where only relatively coarse substructures in a limited range of size could be produced before recrystallization interfered. Certain inferences from the data are included in the discussion which may be important to the problem of the influence of prior history in the engineering creep range.

## PHASE B - REFRACTORY METAL STUDY

### EXPERIMENTAL PROCEDURES

The objective of the refractory metal study under the present contract was to determine the influence of hot working on the structure and high-temperature properties of pure niobium. Also, a comparison of the hot-working response of niobium to that of nickel (Ref. 1) was expected to be informative concerning the behavior of BCC metals versus FCC metals.

To prevent contamination of the pure niobium bar stock during processing, the University of Michigan rolling mill was equipped for inert atmosphere operation and provided with a means for heating in an inert atmosphere. Specimens from the bar stock so processed were then tested in vacuo.

#### General

In establishing the technique to be used for the rolling, many decisions were necessary among available alternatives. Only very limited experience was available as a guide. In fact, the opinions of qualified individuals, whose counsel was sought, often conflicted.

The method selected as being the best compromise involved an enclosed chamber around the rolls and an integrally attached furnace for heating. As a guide for evaluation of the efficiency of the system prior to its use on the niobium, plain carbon steel was used as an indicator. When such a piece could be successfully processed without significant oxidation, the system was deemed adequate.

The rolling mill enclosure and furnace described in the following sections were able to meet this requirement. Many difficulties were encountered, however, which had not initially been anticipated. For example, trial runs demonstrated that to prevent oxidation during heating prior to rolling the furnace system had to be vacuum tight to prevent back-diffusion of air through small leaks which might otherwise have seemed insignificant. The necessity also existed of heating the furnace to about 600°F under vacuum to remove the adsorbed gases from the walls. Similar difficulties were encountered in the construction of the chamber, although to a lesser degree, due to the shorter time of contact between the work pieces and the atmosphere in the chamber.

## Modification of Rolling Mill for Inert-Atmosphere Rolling

The rolling mill was sealed from the atmosphere with a sheet steel enclosure which surrounded the rolls. This enclosure was provided with "glove boxes" through which the operators work. The enclosure was provided with a 6" vacuum slide valve which permitted complete sealing from the furnace during purging.

To remove the air from the enclosure, an air-operated ejector pump was used. This pump was capable of attaining a vacuum of about 27-28 in Hg. To purge the chamber, successive evacuation and back-filling with argon was employed until the final atmosphere had reached the ultimate purity possible. The optimum evacuation conditions for each cycle were computed from the known pumping speed and leak rates as a function of pressure, so that the ultimate purity of the atmosphere could be reached with the minimum number of evacuations. The chamber was back-filled each time using tank argon. Argon was then flushed through the chamber for 3-4 hours at a rate of about 70 cu. ft. per hour to provide additional purification.

The argon employed was Liquid Carbonic Co. tank argon. Analysis of the gas in the University of Michigan mass spectrometer indicated a purity of about 99.9 percent. Using this directly from the tanks, the estimated final purity of the atmosphere in the rolling mill chamber was about 99.8-percent argon.

## Furnace for Heating

In order to adequately cover the range of heating temperatures anticipated for both pure niobium and a niobium alloy, a graphite resistor furnace was chosen. An order was placed with the Hevi-Duty Electric Company for a 4-inch diameter furnace with a 24-inch heated length. This furnace was designed to provide a relatively uniform hot zone over the center 12 inches of its length. A 40-KW saturable reactor power supply was chosen to provide stepless control of the temperature over the entire working range of the furnace (1500°-4000°F). Pieces up to about 2-1/2 inches in diameter and 12 inches long can be accommodated by the furnace.

Several of the design features of the graphite resistor furnace were unusual and resulted in delays during construction of the furnace. When, as a result of these delays, the available time in which to complete the program became short, an electric resistance-wound furnace was designed and put into service for rolling the pure niobium. The

furnace was constructed by winding a coil of Hoskins 875 wire around a stainless tube which attached to the rolling mill chamber in the same way as the graphite tube furnace.

The atmosphere provided to the furnace consisted of the same tank argon as was used in the rolling mill chamber. However, before admission to the furnace, the argon was purified by passing through a dry ice-acetone cold trap and then through a tube furnace containing titanium chips at 1650°F.

Successful operation of the heating furnace required several precautions and a relatively elaborate technique. The furnace and the gas purifying system had to be vacuum tight. The entire system was evacuated to about 30 microns and held overnight. The chip furnace was then heated to 1650°F and the pressure allowed to drop back to 30 microns. Finally, the furnace for heating for rolling was heated to about 600°F and once more the pressure allowed to return to the 30 micron level. After this sequence of operations, the entire system was slowly filled with argon.

A steady flow of argon was employed to provide a continuous supply of pure gas. The gas was allowed to pass through the entire length of the furnace and then escape through a bubbler containing mineral oil. A back-pressure of about two inches of oil was maintained at the outlet.

When the gas flow was established, the furnace was heated to the desired temperature. Temperature distribution was obtained in the furnace by independent adjustment of the heating coils at the center and the ends. With this adjustment, the temperature could be held to within  $\pm 10^\circ\text{F}$  of the desired working temperature.

Due to the many unforeseen difficulties in providing an "adequately-inert" atmosphere for rolling using the small resistance-wound furnace, the available time did not permit installation of the graphite tube furnace. All of the rolling completed was done using the smaller furnace.

### Procedure for Rolling

The previous two sections describe the way in which both the rolling mill enclosure and the heating furnace were prepared for use. In conducting the rolling of the niobium, the furnace and mill enclosure were concurrently brought to operating conditions as described previously. Throughout this period, the two were separated by a six-

inch vacuum slide valve. The pieces were placed in the mill enclosure until the heating furnace was on temperature and the enclosure completely purged. The valve was opened long enough to insert a piece for heating, and then immediately closed until the heating period was concluded. The bars were allowed to heat for 1 hour and were then removed from the furnace and rolled.

No reheating was used for any of the rolling reported for this investigation. All rolling was done in two passes. The initial size of the stock determined the final reduction of area obtained.

After rolling the bars were placed in a metal box which was immediately covered to provide a stagnant atmosphere, thereby minimizing the opportunity for contamination.

This system permitted rolling of a plain carbon steel bar at 1800°F without any visible discoloration of the surface. Niobium bars were rolled at both 1800° and 2000°F. All of the bars had clean surfaces after rolling and showed no noticeable contamination. Although the purity of the atmosphere in the rolling mill chamber was definitely lower than was desirable, the procedure employed was successful due to the high purity of the atmosphere in the heating furnace and the short time periods (about 60 to 70 seconds) during which the bars were in the rolling mill enclosure before cooling.

### Creep-Rupture Testing

The creep rupture testing of the niobium bars was carried out in vacuo at 1800°F using a test unit quite different from that in which the nickel was tested. The design consisted of an evacuated shell containing a tantalum wound resistance furnace. Four molybdenum radiation shields provided insulation against heat loss. The furnace was wound from wire and operates at 110 volts and about 1500 watts. The load was transmitted to the specimen through a water-cooled O-ring seal, by direct dead-load application. The friction in the seal was measured experimentally and found to be negligible. In computing actual loads, compensation was made for the force of the atmosphere against the cross-section of the pull-rod.

The pressure during testing never exceeded  $5 \times 10^{-4}$  mm and after the first few hours of a test, the pressure remained below  $10^{-4}$  mm. Temperatures were measured with chromel-alumel thermocouples attached to the sample. Checks on the calibration of these thermocouples after 30 hours, indicated that no significant change had occurred.

Deformation was measured during the tests using a dial indicator with a sensitivity of  $5 \times 10^{-5}$  inches.

## RESULTS AND DISCUSSION

The double-electron-gun-melted pure niobium from Heat No. 6133 was rolled at 1800° and 2000°F with reductions varying from 3 to 40 percent. Room temperature hardness values were obtained after rolling, and the creep and rupture properties evaluated with tests at 1800°F.

### Effect of Rolling on Hardness

The hardness variation with rolling at either temperature (Fig. 17) was rather small. The hardness increased with the first few percent reduction and then remained relatively constant with further reduction. Although the data suggest that the hardness after rolling at 2000°F was less than that obtained for the same reduction at 1800°F, the slight differences indicated by the data may not have been significant.

Comparison of these hardness changes with those for "A" Nickel from Reference 1 (Fig. 17) shows that the hardness level and hardness increase for the niobium was the same as that obtained for the nickel after rolling at 1800°F. This temperature is, however, relatively high for nickel representing a homologous temperature ( $T_h$ ) of 0.74, while for the niobium  $T_h$  at 1800°F is only 0.48. Data for nickel rolled at lower temperatures (Ref. 1) showed that considerably larger hardness increases resulted.

### Effect of Rolling on Creep-Rupture Properties

Only a limited amount of data could be obtained on the niobium stock within the time available under the present contract. The material rolled 19.0 percent at 1800°F was selected for the first tests since four samples were available in this condition.

Based on the best information available from the published literature, an initial stress of 6240 psi was selected to give rupture in 10 to



100 hours at 1800°F This sample ruptured (Table 4) in 0.3 hours. A second test was started at 5740 psi which gave rupture in 0.55 hours. For the third test, the stress was reduced to 3940 psi giving a rupture time of 29.2 hours. This stress was then selected for further comparison testing of the material in varying conditions. These data (Table 4 and Fig. 18) indicate the following:

(1) The rupture time of the material rolled 19.0 percent at 1800°F was slightly longer (Fig. 18) than that for the material simply heated to 1800°F with no reduction.

(2) The minimum creep rate was about the same for the material rolled at 1800°F and the material simply heated to 1800°F (Fig. 19). The amount of first-stage creep in the rolled sample was considerably lower.

(3) The sample rolled 20.5 percent at 2000°F was considerably more creep resistance than either of the two above mentioned samples. Very little primary creep occurred (Fig. 19) and the minimum creep rate at 3940 psi was much lower than for either of the other two materials. This sample was discontinued at 60 hours, more than double the rupture time of the material after rolling at 1800°F.

## Discussion

Due to the limited data, no definite conclusions can be drawn. The change in hardness of the niobium with amount of reduction was small, as was to be expected for niobium. The rupture strength was assumed to be low in comparison to the data used as a guide for selecting stresses because the material tested contained a lesser amount of contaminants.

The limited rupture-test results give no clear indication of the effect of rolling. The apparent similarity of rupture strength of material simply heated at 1800°F to that of material rolled at 1800°F suggested that rolling would have little effect on rupture strength. On the other hand, rolling at 2000°F resulted in a marked increase in rupture strength. The rolling at 1800°F did virtually eliminate prior creep and somewhat prolong rupture life. It would seem that rolling conditions could have a fairly pronounced effect on creep rupture properties even though there is practically no strain hardening. Presumably the main mechanism for such an effect would be substructural variations governed by the rolling conditions.

The apparent pronounced increase in strength from increasing the rolling temperature from 1800° to 2000°F indicates that unlike nickel, the creep-rupture properties of niobium are influenced by the temperature of rolling. Possibly there is a difference in the substructures arising from rolling, recovery, and testing that could be clarified by further work.

## 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 (July, 1958).
2. A. P. Coldren, J. E. White, R. K. Bowen, and J. W. Freeman, "Studies of Heat-Resistant Alloys," WADC Technical Report 59-606 (February, 1960).
3. R. W. Guard, "The Role of Polygonization in Creep," WADC Technical Report 59-279 (March, 1959). Appendix A.
4. J. E. Dorn, "The Spectrum of Activation Energies for Creep," Creep and Recovery, p. 255ff., American Society for Metals, Cleveland, Ohio (1957).

Table 1

## Chemical Composition of Nickel Heats

Type and Heat Number	Weight Percent							PPM						
	<u>Ni</u>	<u>C</u>	<u>Mn</u>	<u>Si</u>	<u>Fe</u>	<u>Cu</u>	<u>Mg</u>	<u>Zr</u>	<u>Ti</u>	<u>B</u>	<u>S</u>	<u>O</u>	<u>H</u>	<u>N</u>
High-Carbon "A" Ht. N9500A	Bal.	0.06	0.27	0.06	0.09	0.03	40	<50	240	30	80	-	-	-
Low-Carbon "A" R. L. Stock 02808	Bal.	0.01	0.03	0.10	0.032	-	100	<50	-	<5	50	10.8	0.4	1.6
Carbonyl* (U of M)	Bal.	0.006	-	<0.005	0.02	-	200	<50	-	8	30	3.7	1.2	1.7

\* Qualitative spectrographic analysis of the carbonyl nickel indicated the presence of trace amounts of Cu, Al, and Pb. No other metallic elements were detected.

Table 2

Results from Samples of High-Carbon "A" Nickel Prestrained  
at 1600°F and Rupture Tested at 1100°F and 20,000 psi

Prestrain at 1600°F (%)*	Substructure Density (sub-boundaries per inch)**	Rupture Data at 1100°F and 20,000 psi		
		Rupture Life (hrs)	Elongation (% in 4D)	Red. of Area (%)
0	--	12.0	35.5	61.5
0	--	7.4	33.9	56.6
1.67	830	27.4	41.8	58.0
1.69	840	19.8	39.1	53.9
5.37	2,240	26.0	33.8	51.3
10.80	2,700	15.3	39.8	70.8
23.21	2,230	15.1	46.4	72.9

\* At a strain rate of 30 percent per hour.

\*\* Densities obtained by interpolation from Figure 5.

Table 3

## Structure Measurements on Low-Carbon "A" Nickel and Vacuum-Melted, Carbonyl Nickel After Creep at 1550°F

Specimen Number	Stress (psi)	Creep Test Data				Lineal Analysis of Microstructure			
		Time (hr)	Plastic Strain (percent elong.)	Instantaneous Creep Rate (percent/hr)	Volume Recrystallized	Mean Boundary Density (boundaries intercepted/lineal inch) <sup>a</sup>		Weighted Mean	
						In Recrystallized Portion	In Unrecrystallized Portion		
Base Condition									
LCN-20	--	--	--	--	--	--	--	--	120
LCN-7	3360	0.025	1.15	27.4	0	1232; 1283*	1232; 1283*	1774	1232; 1283*
LCN-8	3360	0.083	2.04	7.3	0	1774	1774	1967	1774
LCN-17	3360	0.233	2.69	3.5	0	17.2 <sup>b</sup>	17.2 <sup>b</sup>	2205	1967
LCN-11	3360	0.85	3.66	1.1	1.1	357.0	357.0	2550	2180
LCN-19	3360	1.90	5.18	2.1	25.6			2673	1987
LCN-18	3860	0.25	3.49/0.57	4.7 <sup>c</sup>	0	20.0 <sup>d</sup>	20.0 <sup>d</sup>	(2565) <sup>c</sup>	2673
LCN-12	3860/3360	0.196/0.837	3.49/0.57	5.7/0.54 <sup>c</sup>	24.6 <sup>d</sup>			(2770) <sup>c</sup>	(2565) <sup>c</sup>
LCN-13	4360	0.10	3.80	9.1	0	2690	2690	2690	2690
LCN-14	4360/3360	0.067/0.683	3.52/0.55	12.9/0.26 <sup>c</sup>	34.6 <sup>d</sup>	260.0 <sup>d</sup>	260.0 <sup>d</sup>	(2770) <sup>c</sup>	(2770) <sup>c</sup>
LCN-16	5360	0.022	3.68	45.2	0	954.0 <sup>d</sup>	954.0 <sup>d</sup>	(2990) <sup>c</sup>	2940
LCN-16	5360/3360	0.024/0.926	3.91/0.57	44.3/0.12 <sup>c</sup>	65.1 <sup>d</sup>			(2990) <sup>c</sup>	(2990) <sup>c</sup>
Vacuum-Melted Carbonyl Nickel									
Base Condition									
HPN6-11	3360	0.050	1.47	10.3	0	--	--	1547	145
HPN5-4	3360	0.133	1.96	4.3	0	--	--	1753	1547
HPN6-8	3360	0.30	2.76	3.0	0	--	--	2100	1753
HPN5-1	3360	0.75	3.19	1.2	0	--	--	2291	2100
HPN5-2	3360	1.85	4.06	0.62	0	--	--	2357	2291
HPN8-12	3360	4.30	5.16	0.31	trace	--	--	2508; 2570*	2357
HPN7-7	3860	0.217	3.52	5.7	0	--	--	2550	2508; 2570*
HPN8-13	3860/3360	0.25/3.88	3.34/1.32	4.7/0.41 <sup>c</sup>	trace	--	--	(2530) <sup>c</sup>	2550
HPN7-6	4360	0.10	3.44	12.9	0	--	--	2543	(2530) <sup>c</sup>
HPN6-9	4360/3360	0.10/1.60	3.76/0.55	13.3/0.35 <sup>c</sup>	0	--	--	(2620) <sup>c</sup>	2543
HPN7-5	5360	0.017	3.57	57.7	0	--	--	2825	(2620) <sup>c</sup>
HPN6-10	5360/3360	0.017/2.23	3.58/0.58	57.7/0.12 <sup>c</sup>	0	--	--	(2830) <sup>c</sup>	2825

a - Grain, subgrain and twin boundaries intercepted per inch over total traverse distance of about 1.9 inches, crossing from 150 to 250 grains.

b - Grain boundaries between new and old grains were arbitrarily counted as part of the unrecrystallized structure, permitting this value to be lower than the base-condition value.

c - At beginning of creep at correlation stress, 3360 psi.

d - At termination of creep at correlation stress, 3360 psi.

\* - Repeated measurements.

\*\* - Data separated by a diagonal mark (/) pertain to stress-reduction tests; the values to the left and right of the diagonals correspond to the high- and low-stress portions of the tests, respectively. The creep rates to the left of the diagonals occurred at the end of the high-stress part of the test, immediately preceding the lowering of the stress.

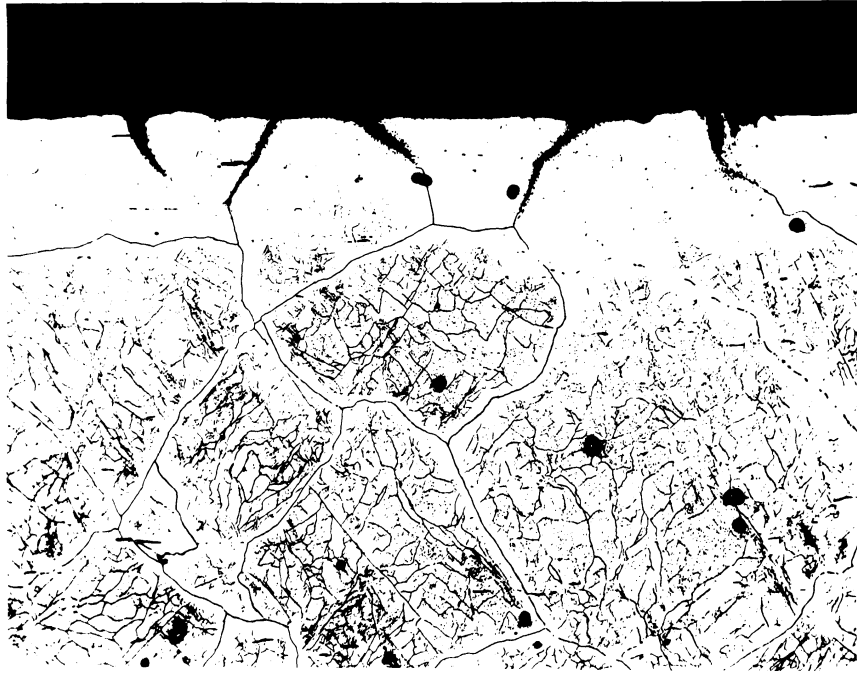
TABLE 4

Results of Creep-Rupture Tests on Niobium  
(all tests at 1800°F)

<u>Temp(°F)</u>	<u>Rolling Condition</u>		<u>Stress</u> psi	<u>Rupture</u> <u>Time, hrs.</u>	<u>(1) Elong.</u> <u>(%)</u>
		<u>Amount(%)</u>			
1800		19.0	6240	0.3	32
1800		19.0	5740	0.55	29
1800		19.0	3940	29.2	29
1800		0	3940	21	30
2000		20.5	3940	Discontinued at 60 hrs.	

---

(1) Reduction of area could not be measured due to fibrous appearance of fracture. All values were estimated as >90%.



(a) Photomicrograph of the Edge of a Specimen  
Creep Tested in Air at 1550°F. Mag. 100X.



(b) Photomicrograph of the Edge of a Specimen  
Creep Tested In Vacuo at 1550°F. Mag. 100X.

**Figure 1** Illustration of Surface Cracking and Decarburization  
Associated with Creep Testing Nickel in Air and the  
Effective Elimination of These Problems by Testing  
In Vacuo.



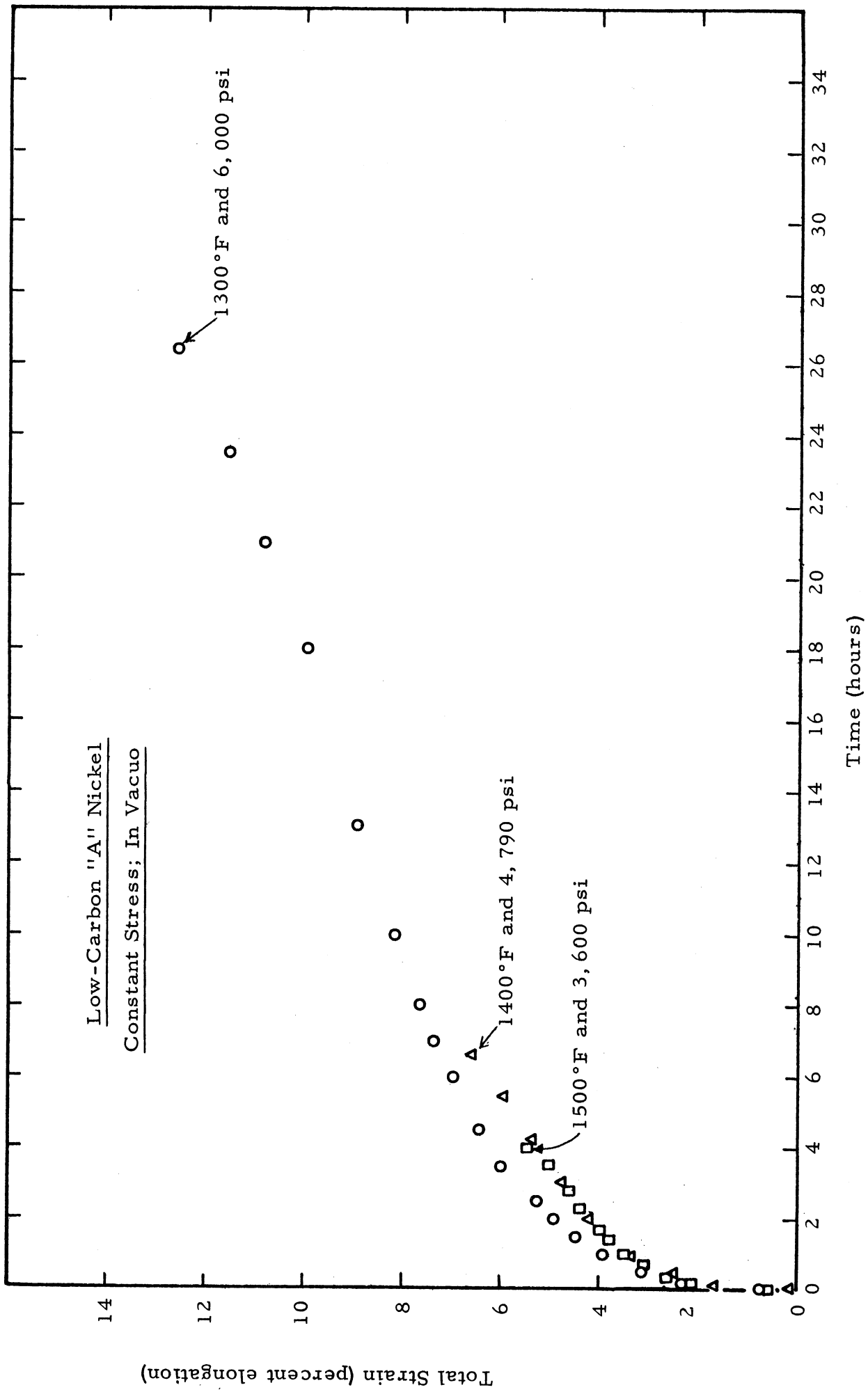
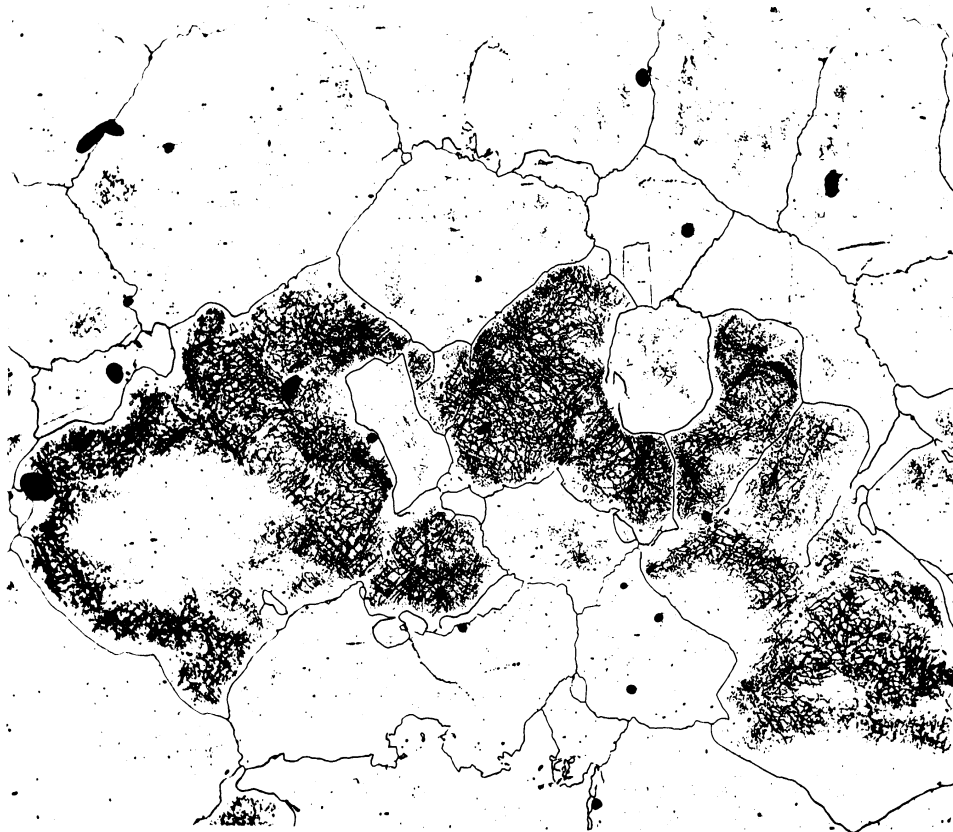


Figure 2 Creep Curves from Preliminary Tests Conducted to Ascertain the Effect of Creep Temperature on the Clarity of Substructures as Revealed by the Etch-Pit Technique

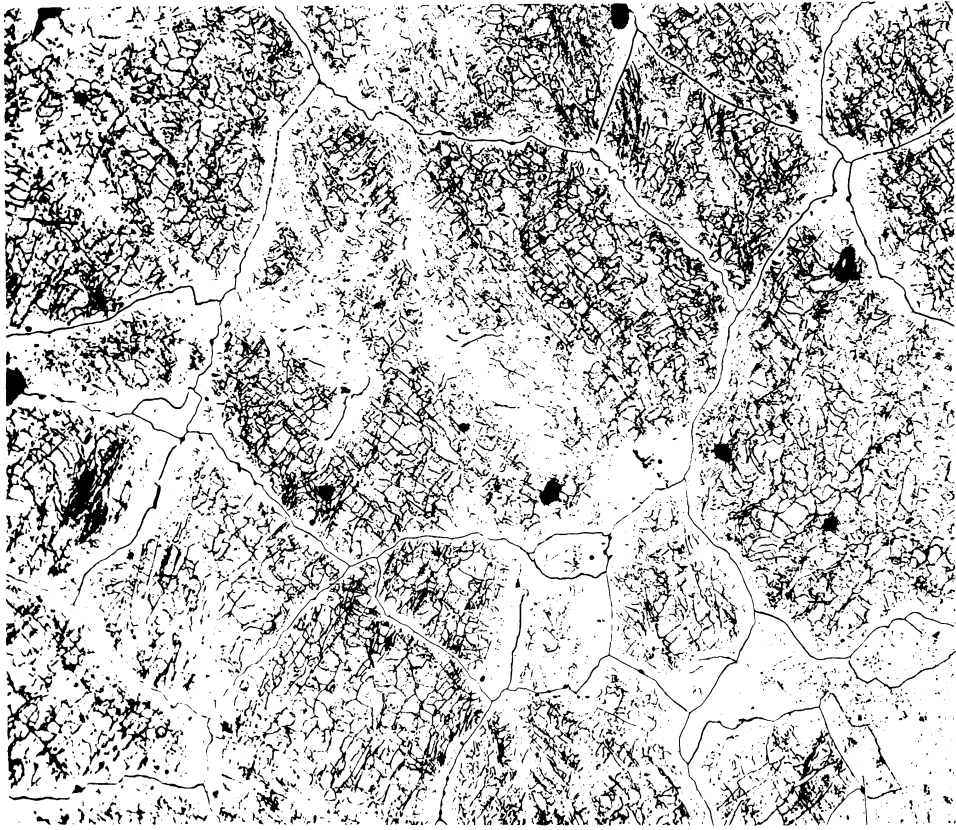


(a) Creep Tested at 1300°F and 6,000 psi to 12.7 Percent Elongation in 26.4 Hours. Mag. 100X.

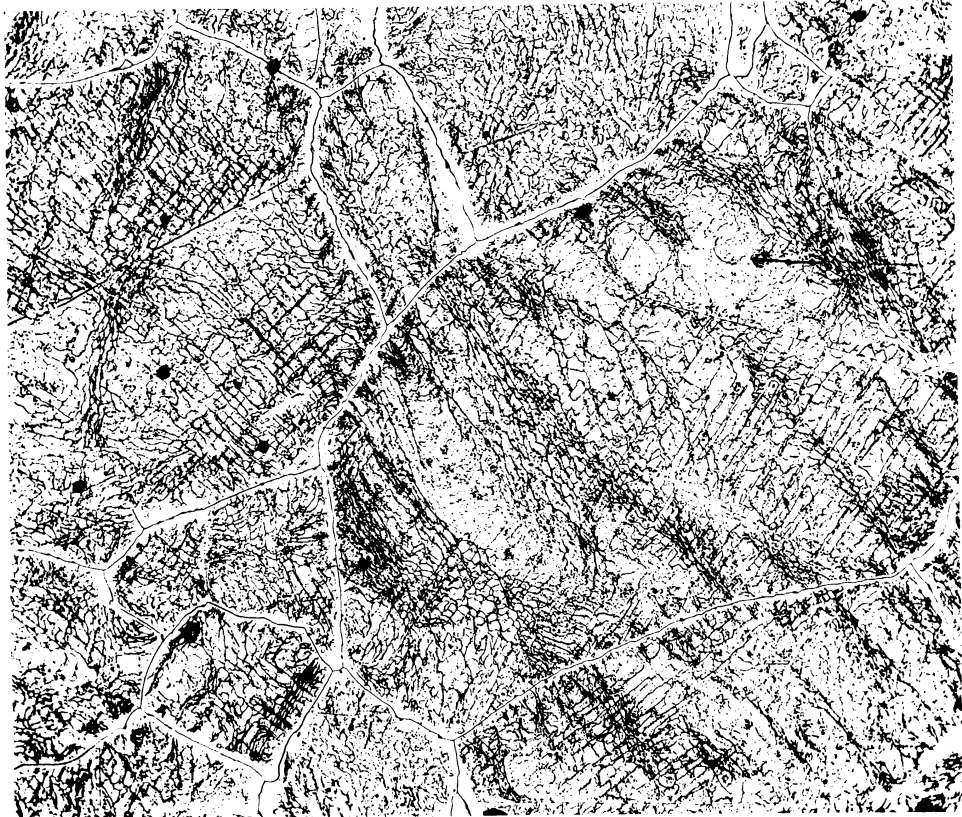


(b) Creep Tested at 1400°F and 4,790 psi to 6.60 Percent Elongation in 6.6 Hours. Mag. 100X.

Figure 3 Influence of Creep Test Temperature on Delineation of Substructure in Low-Carbon "A" Nickel by Etch Pitting.



(c) Creep Tested at 1500°F and 3,600 psi to 3.03 Percent Elongation in 4.0 Hours. Mag. 100X.



(d) Creep Tested at 1550°F and 3,360 psi to 2.04 Percent Elongation in 0.083 Hours. Mag. 100X.

Figure 3 Concluded.

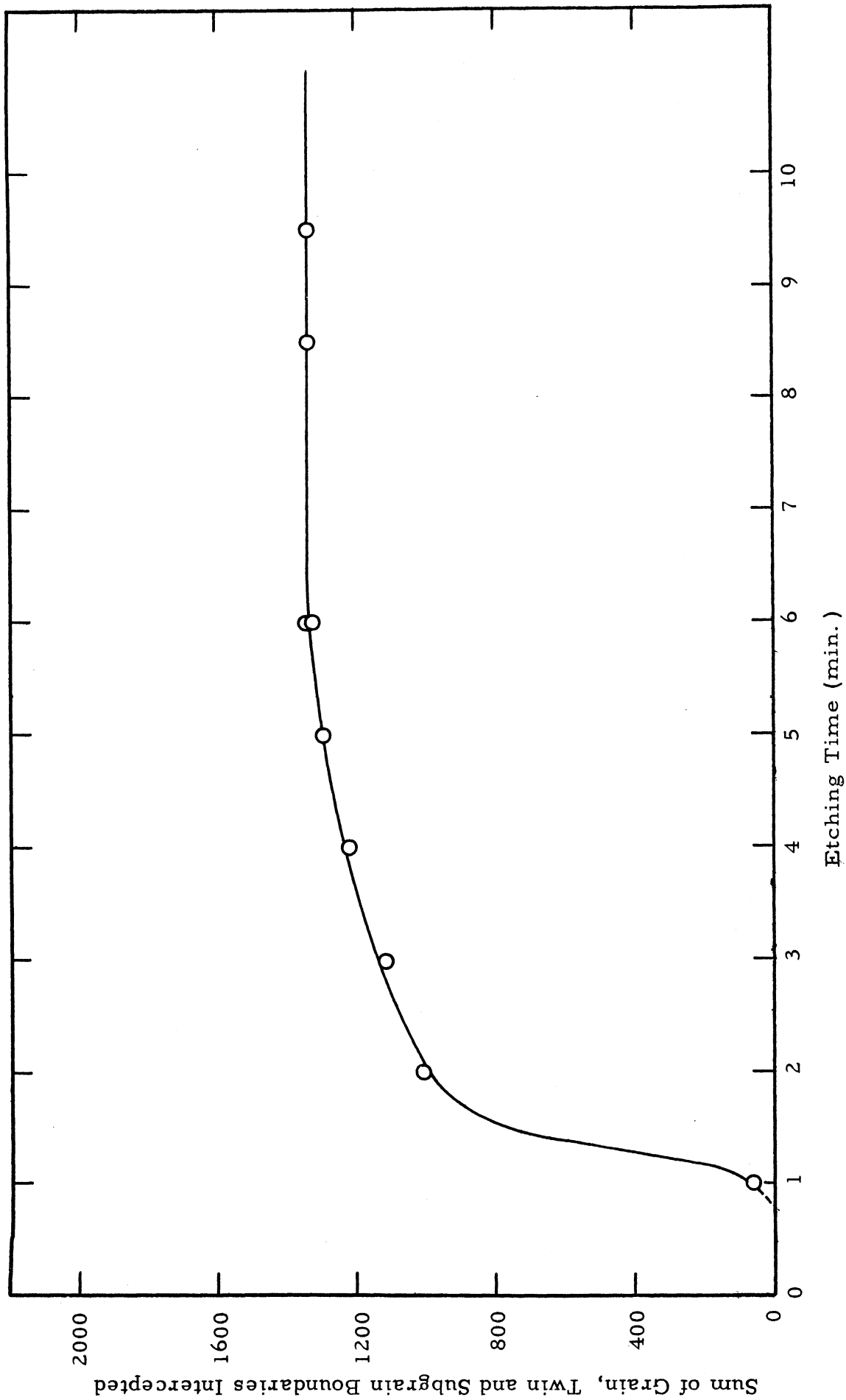


Figure 4 Effect of Etching Time on the Sum of Grain, Twin and Subgrain Boundaries Observed to Intersect a Straight Line Connecting Two Given Points in a Specimen of Low-Carbon "A" Nickel Creep Tested at 1550 °F to 3.83 Percent Elongation.

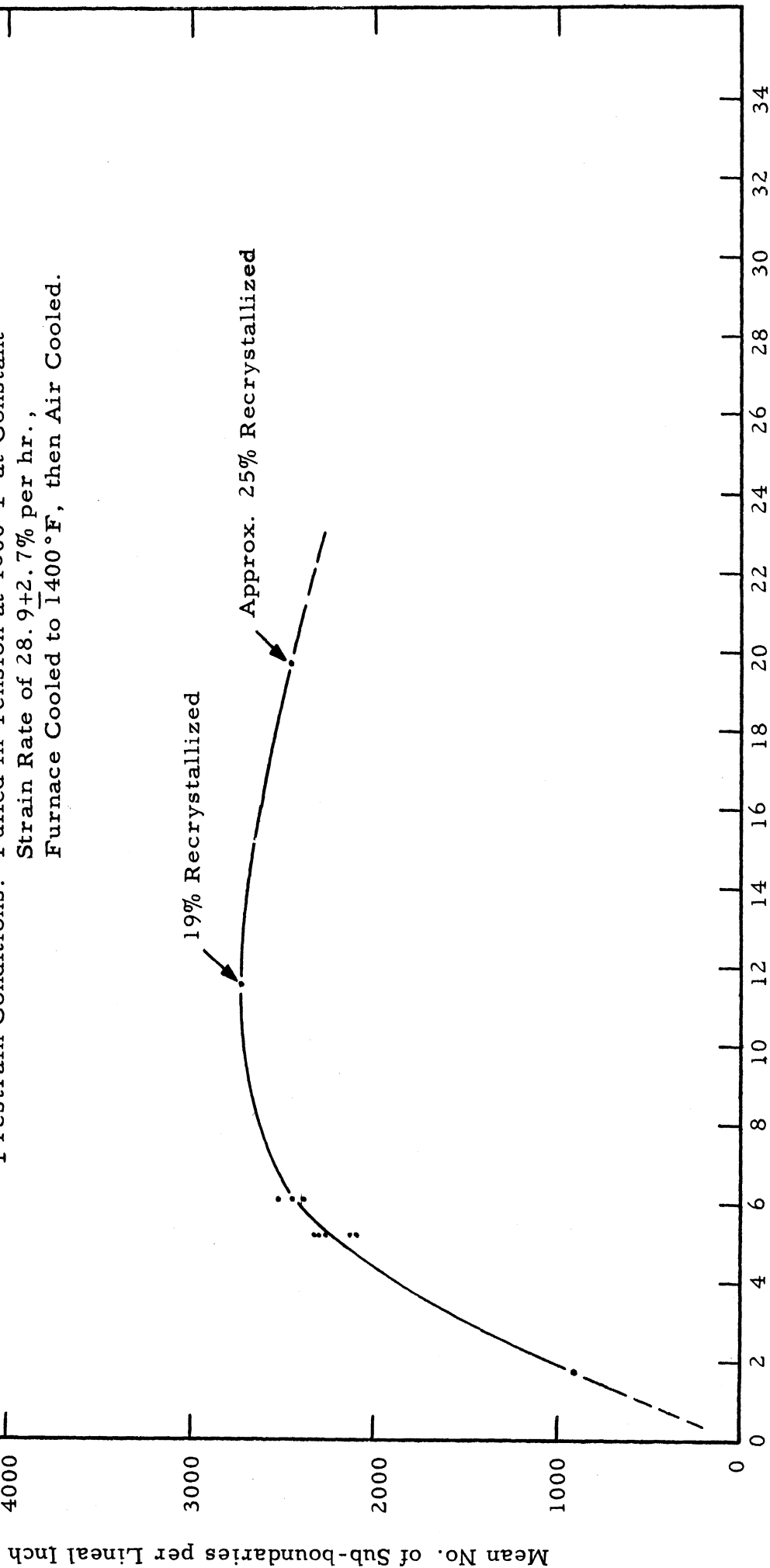
High-Carbon "A" Nickel

Heat Treatment: 1 hr. 1800°F, W. Q.

Prestrain Conditions: Pulled in Tension at 1600°F at Constant

Strain Rate of 28.9±2.7% per hr.,

Furnace Cooled to 1400°F, then Air Cooled.



Prestrain (percent elongation)

Figure 5 Metallographic Counts of Subgrain Boundaries in High-Carbon "A" Nickel. (Sub-boundary densities were obtained by lineal intercept method. Each point is an average for a total length of traverse of about 3 inches, crossing about 350 grains.)

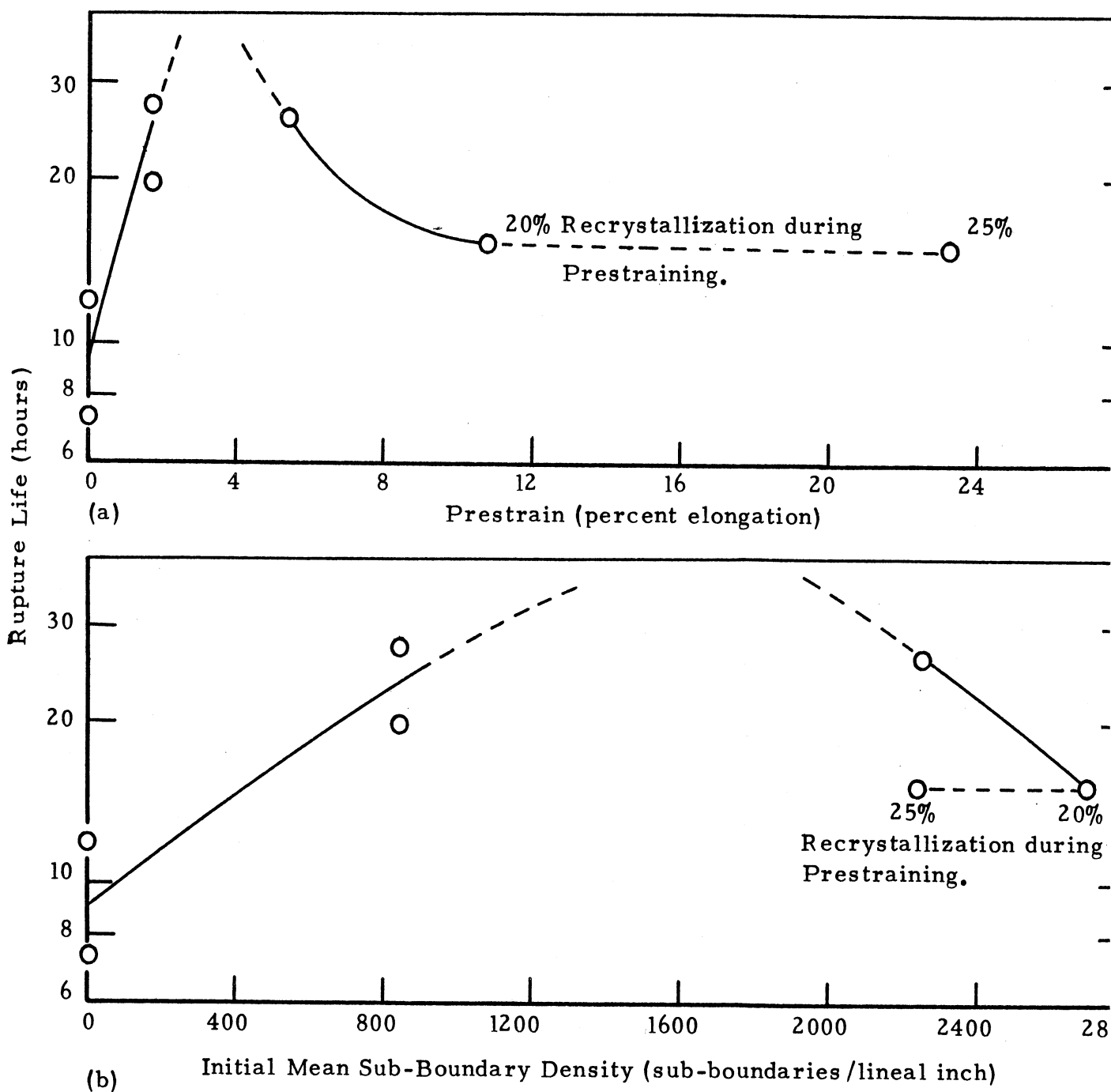


Figure 6 Effect of Percent Prestrain and Initial Sub-Boundary Density on the Rupture Life of the Higher-Carbon "A" Nickel at 1100°F and 20,000 psi.

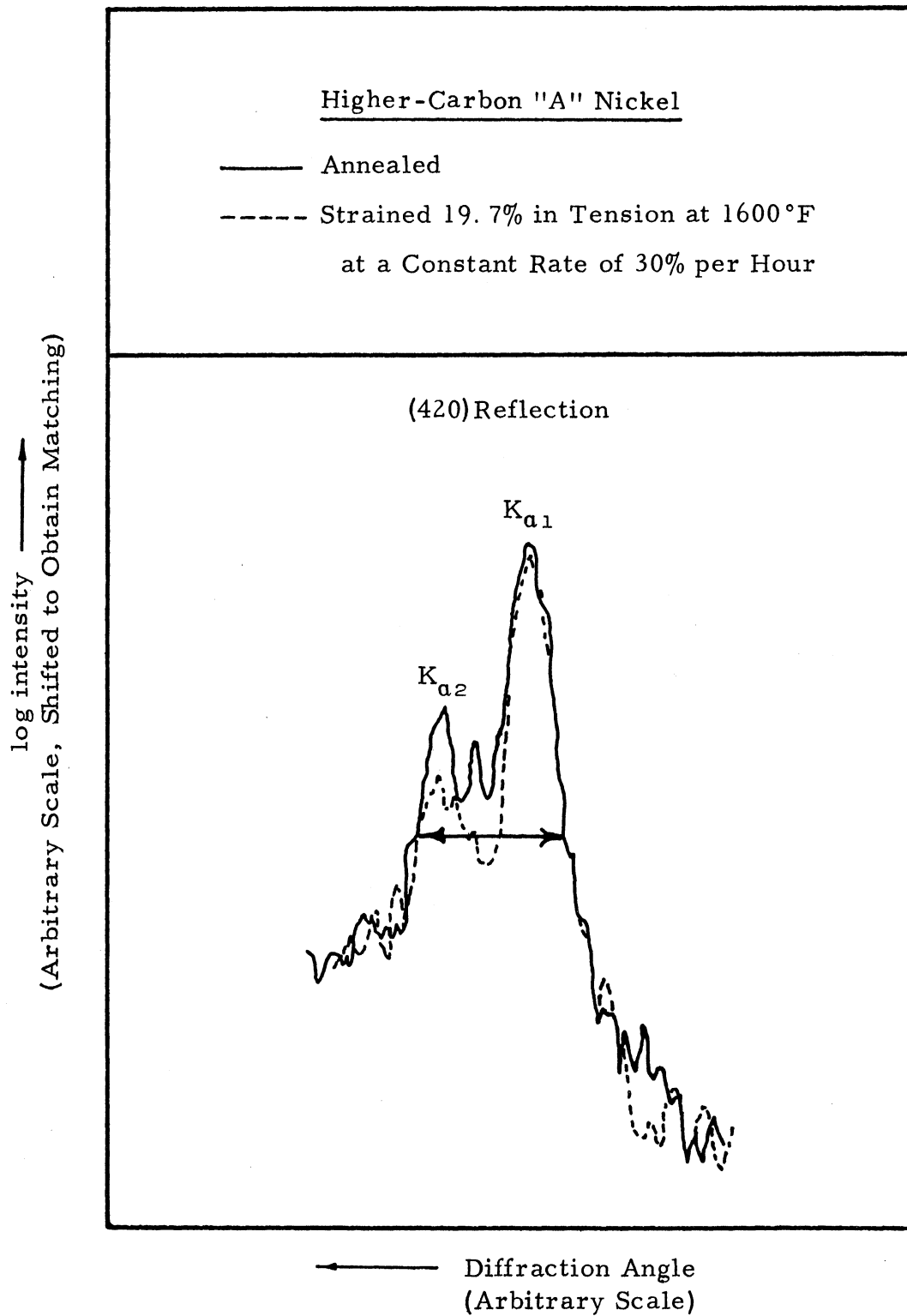


Figure 7 Profiles of X-Ray Diffraction Lines from (420) Planes in Annealed and Hot-Strained "A" Nickel. (Note that the widths of the lines are equal, indicating an absence of lattice stresses of the type found in cold-worked metals. The two photograms were taken on the same sheet of film so that photographic processing would be identical.)

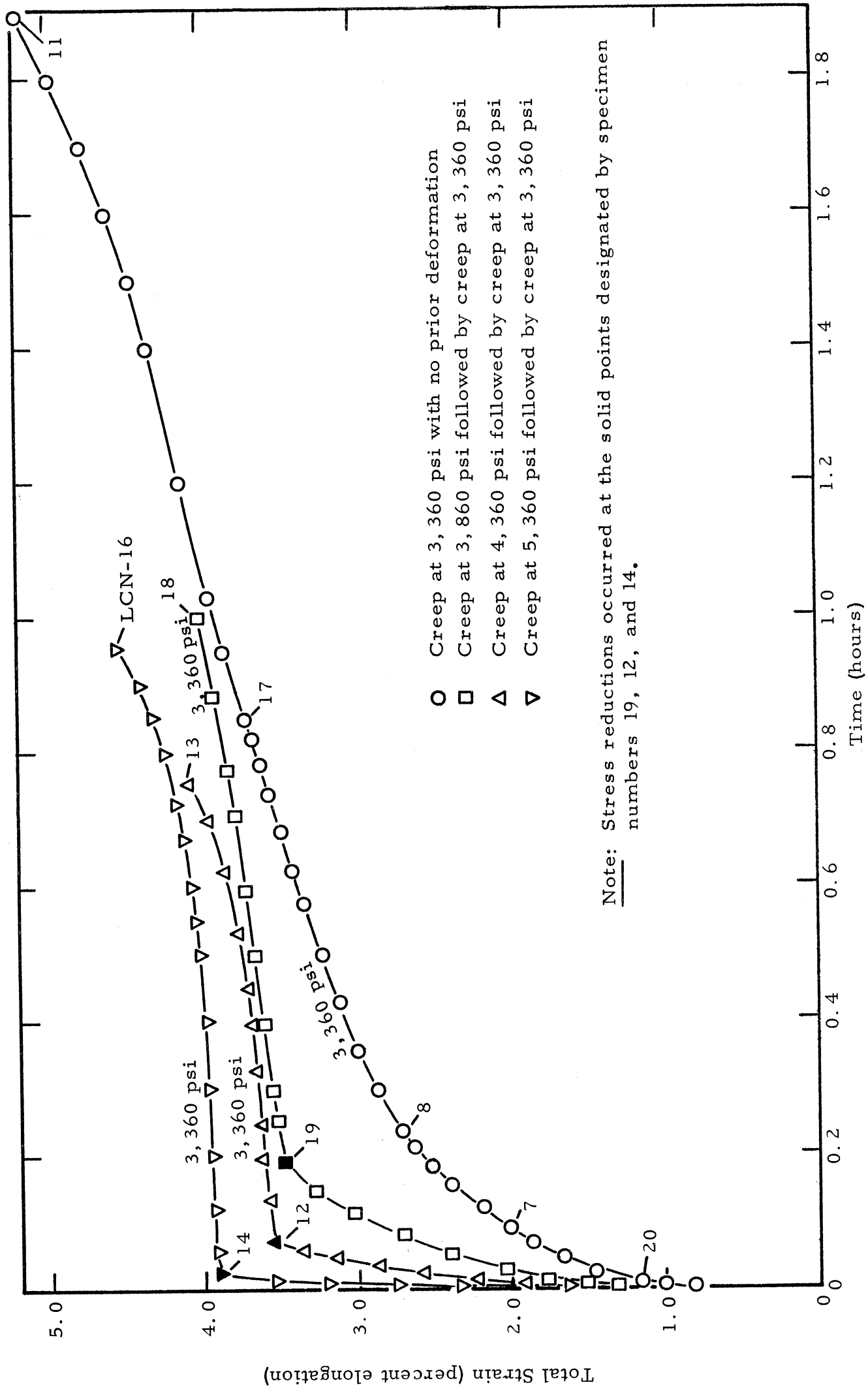


Figure 8 Creep Data for Low-Carbon "A" Nickel Tested in Vacuo at 1550°F. (The creep curve at 3,360 psi for specimens with no prior deformation is the average of several curves.)



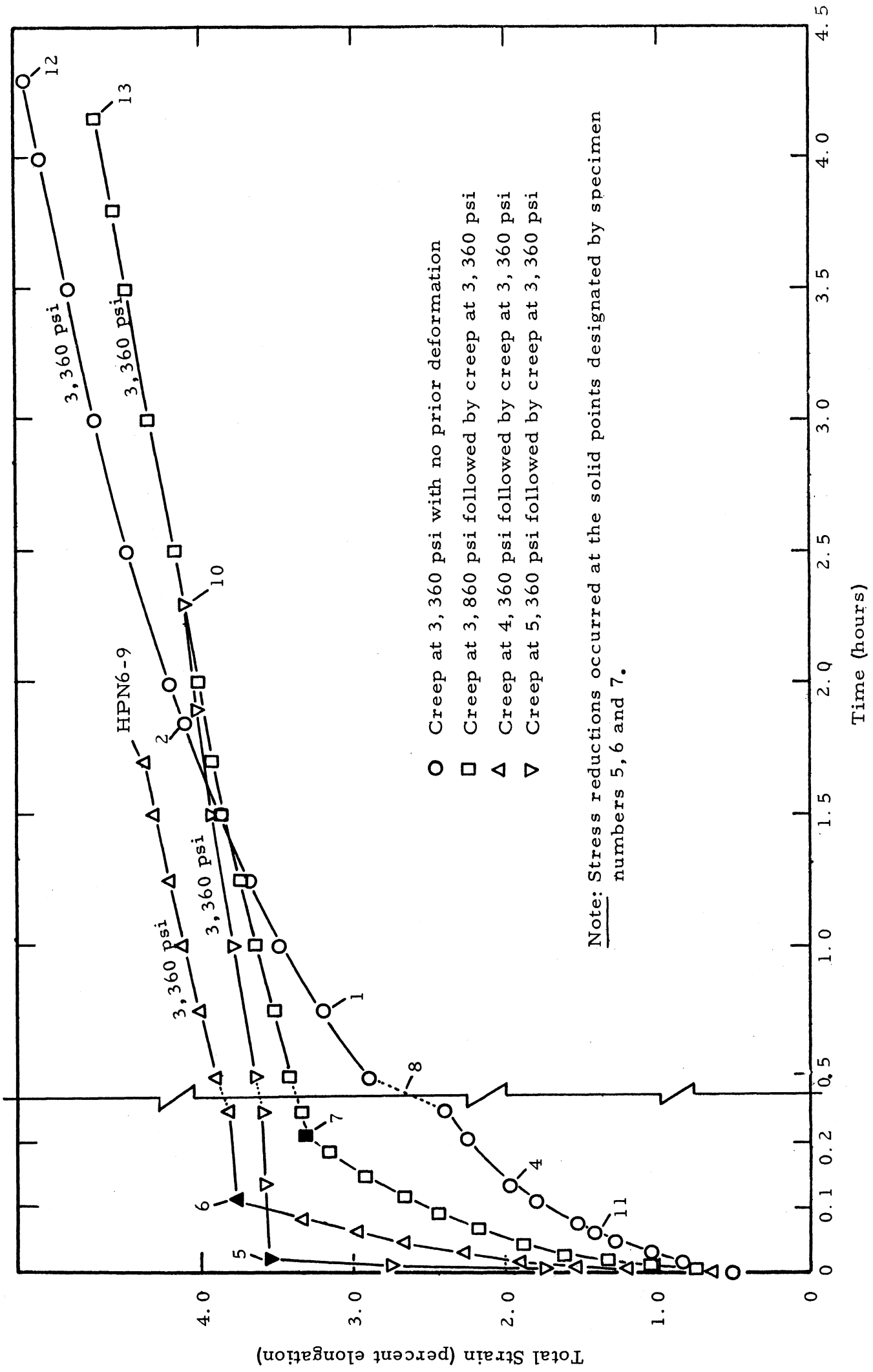


Figure 9 Creep Data for Vacuum-Melted Carbonyl Nickel, Tested in Vacuo at 1550°F. (The curve at 3360 psi for specimens with no prior deformation is an average from several curves).

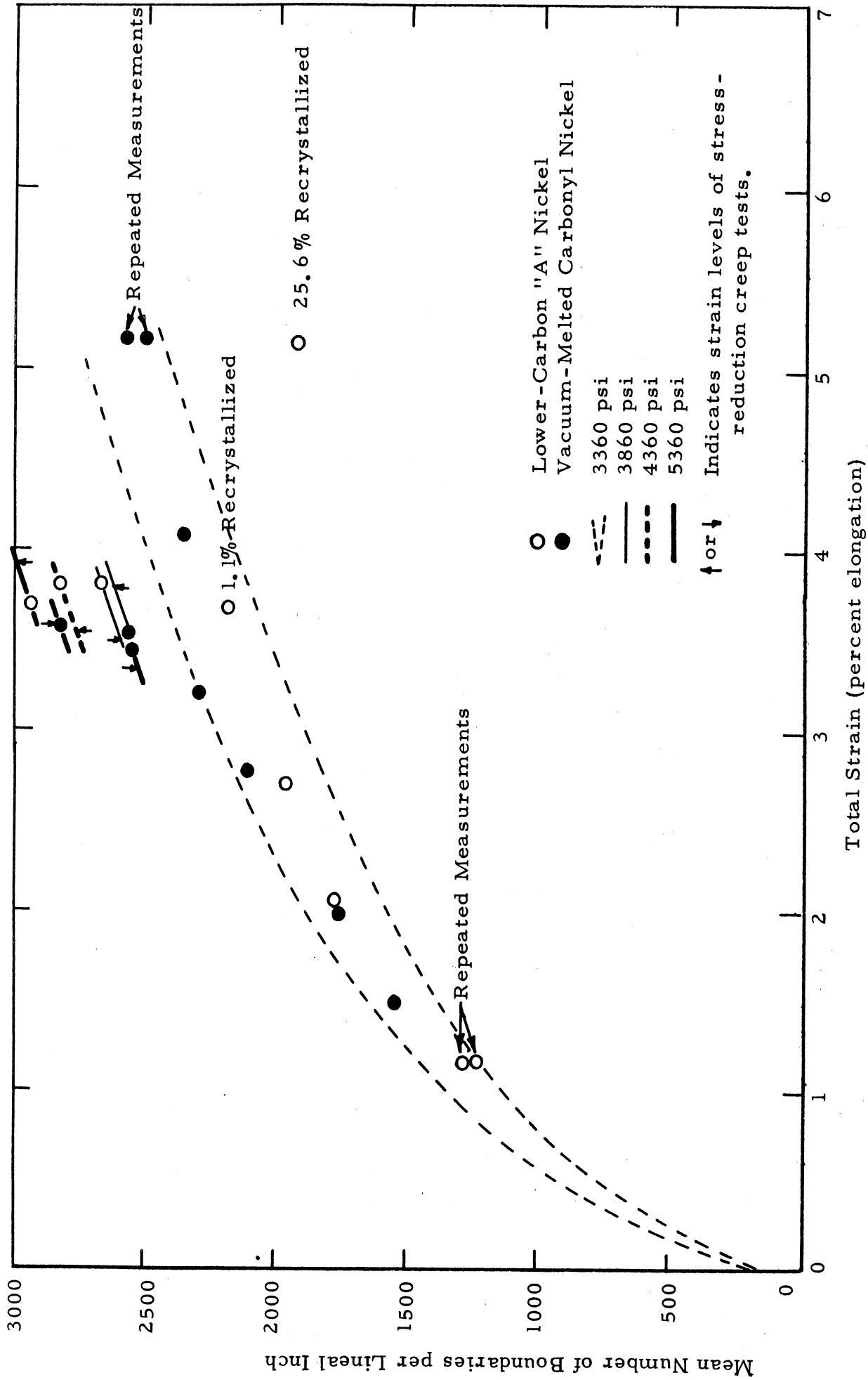


Figure 10 Influence of Strain in Vacuo at 1550°F on the Mean Number of Subgrain, Grain, and Twin Boundaries per Lineal Inch in the Lower-Carbon "A" Nickel and the Carbonyl Nickel.

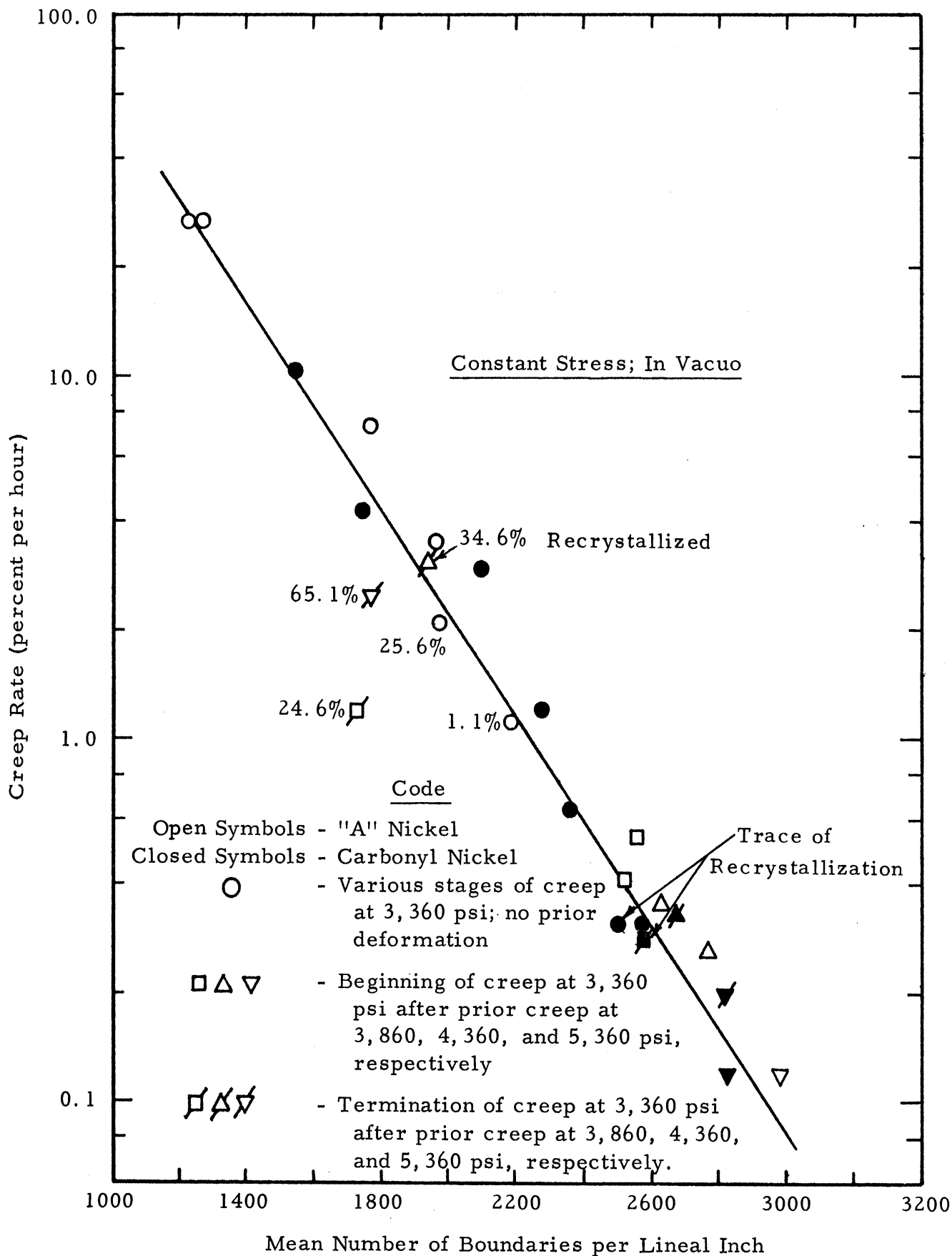


Figure 11 Correlation of Creep Rate at 3,360 psi and 1550°F with Mean Number of Subgrain, Grain, and Twin Boundaries per Lineal Inch in Low-Carbon "A" Nickel and Carbonyl Nickel

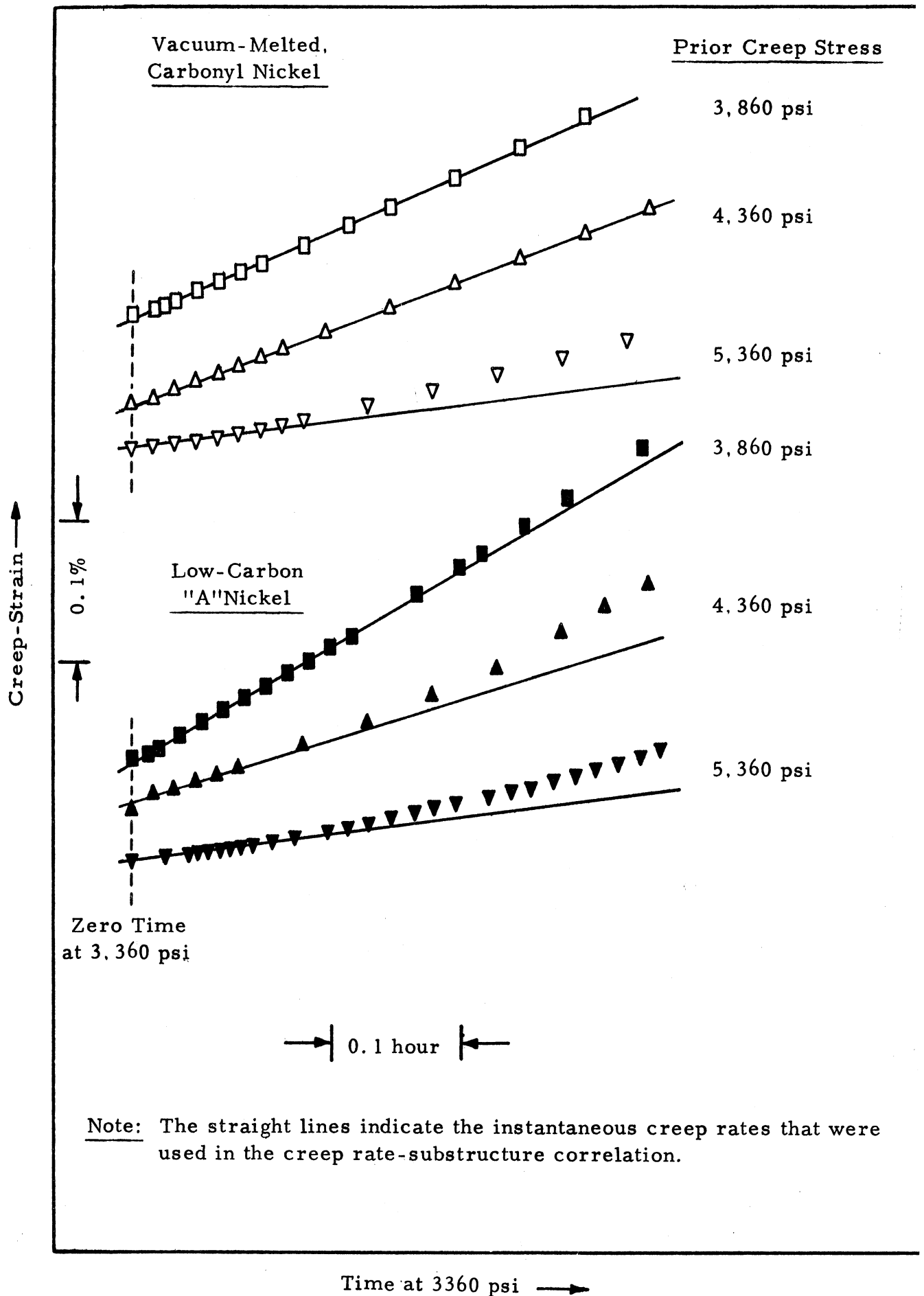


Figure 12 Strain-Time Curves for Carbonyl Nickel and "A"Nickel at 1550°F Show the Initiation of Creep at 3,360 psi Immediately Following Prior Creep Higher Stresses.

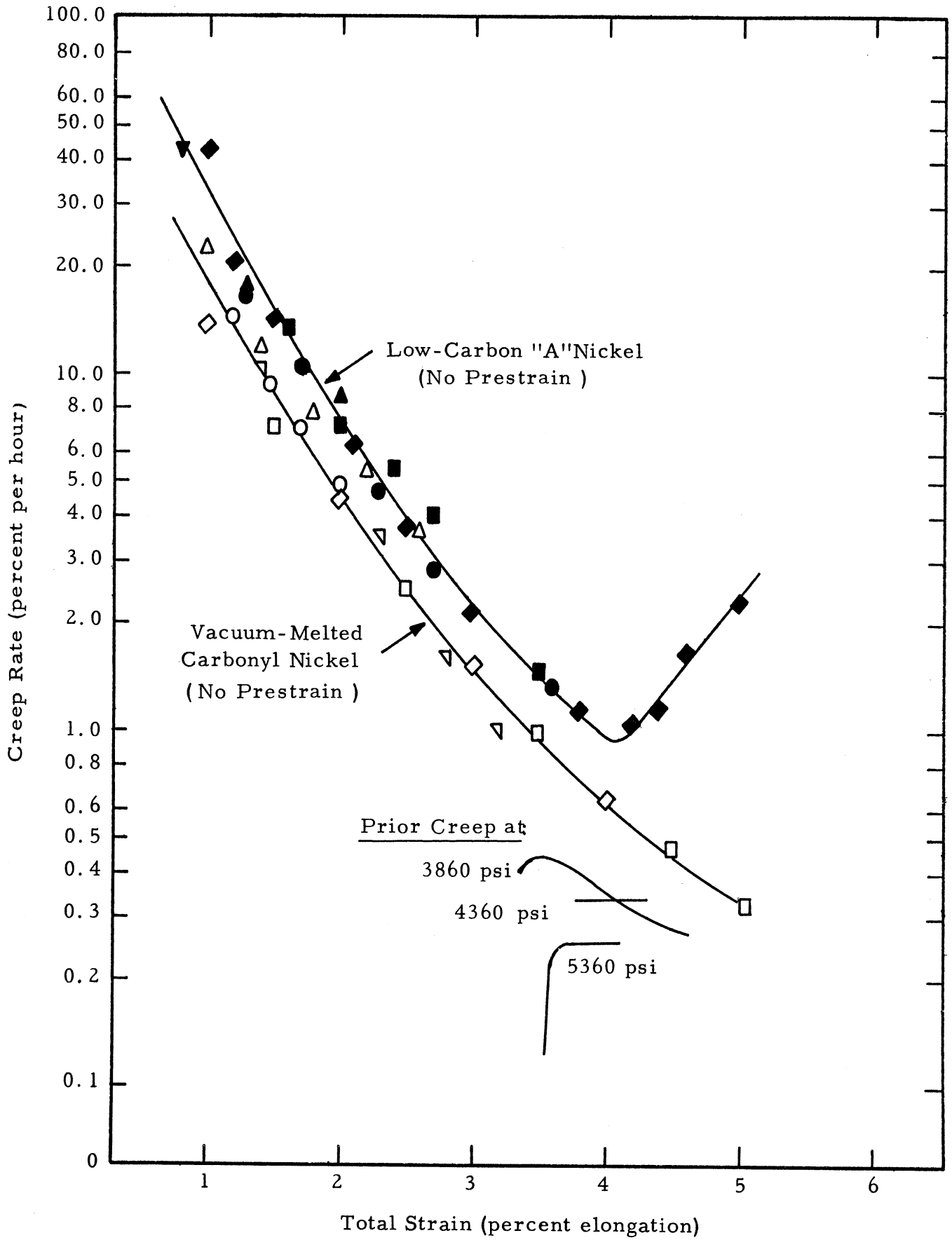
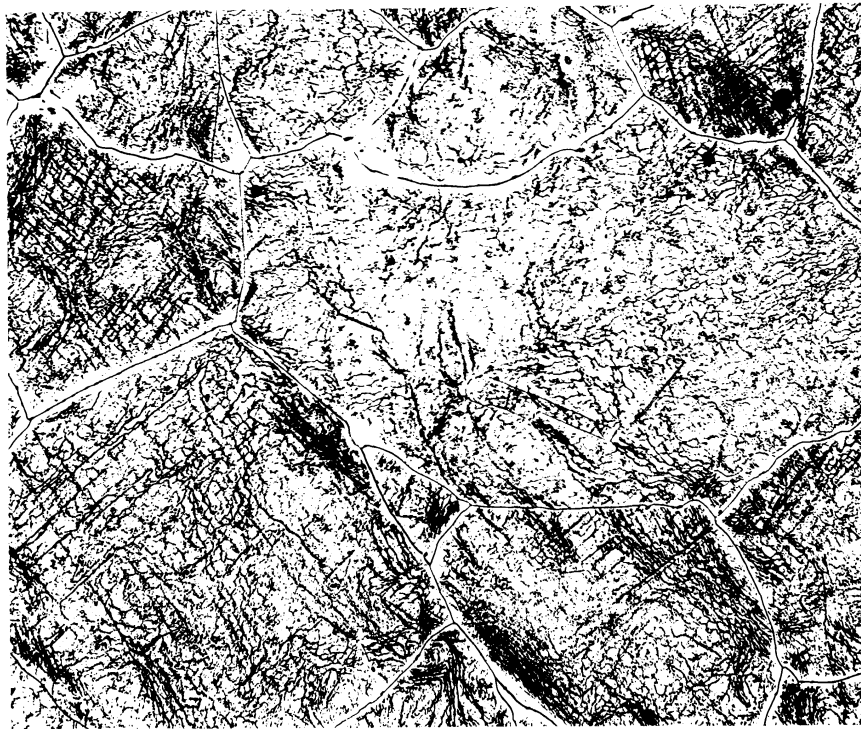
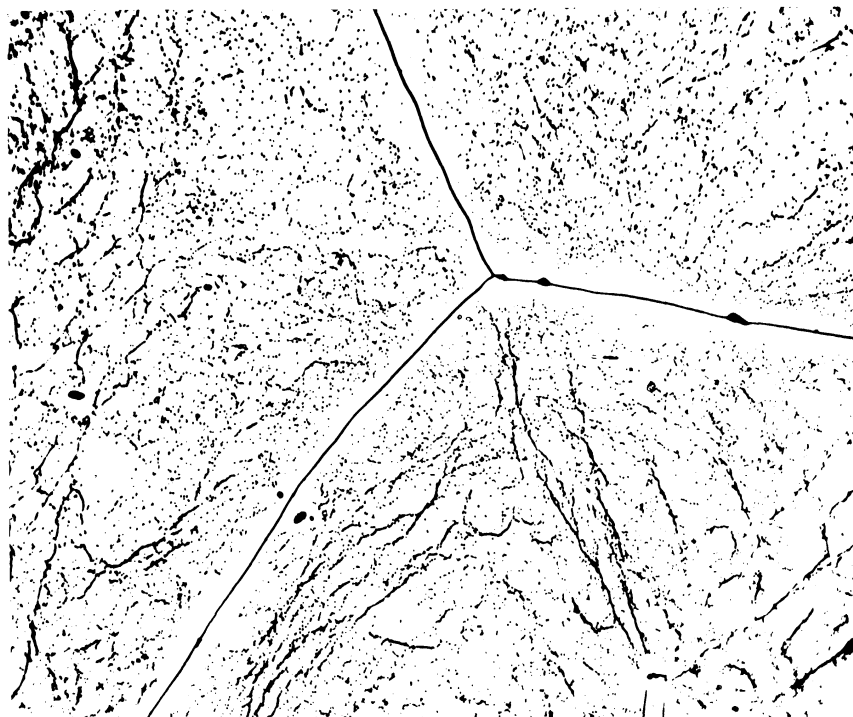


Figure 13 Relation of Creep Rate to Total Strain in Low-Carbon "A" Nickel and Carbonyl Nickel Creep Tested in Vacuo at 1550°F under a Constant Stress of 3360 psi.



(a) 1.15 Percent Elongation; Decelerating Creep.  
Mag. 100X.



(a) 1.15 Percent Elongation; Decelerating Creep.  
Mag. 500X.

Figure 14

Typical Substructures in Low-Carbon "A" Nickel Produced by Creep Testing at 1550°F and 3,360 psi to (a) 1.15, (b) 2.04, (c) 2.69, (d) 3.66, and (e) 5.18 Percent Total Elongation. The tension axis is horizontal in all cases.

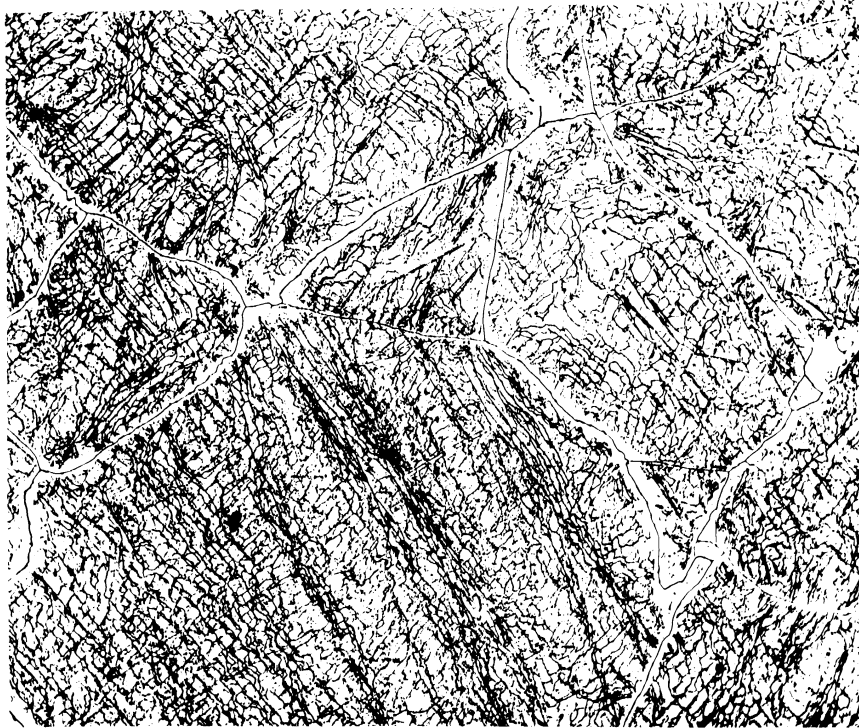


(b) 2.04 Percent Elongation; Decelerating Creep.  
Mag. 100X.



(b) 2.04 Percent Elongation; Decelerating Creep.  
Mag. 500X.

Figure 14. Continued



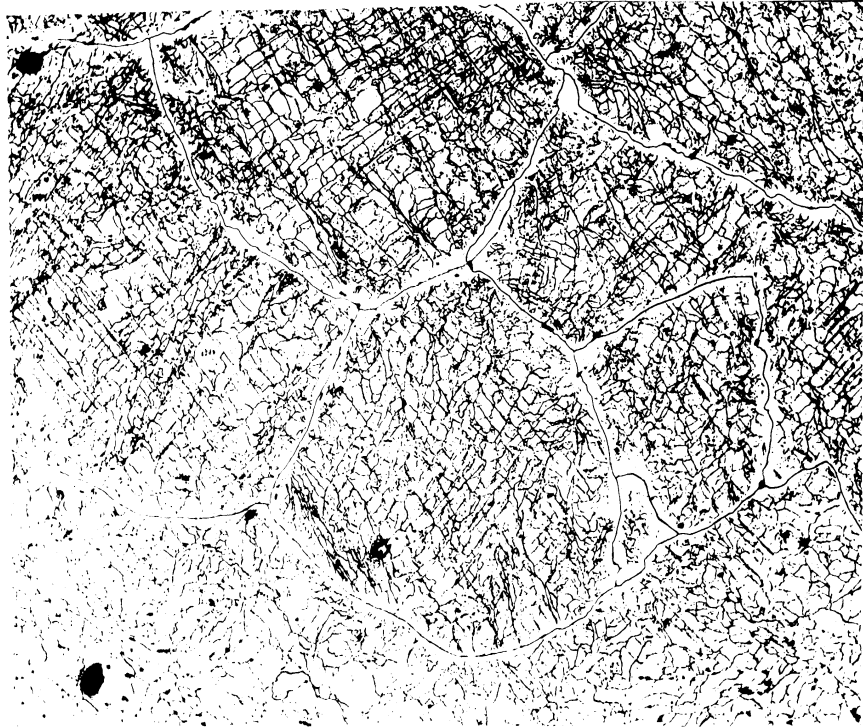
(c) 2.69 Percent Elongation; Decelerating Creep.  
Mag. 100X.



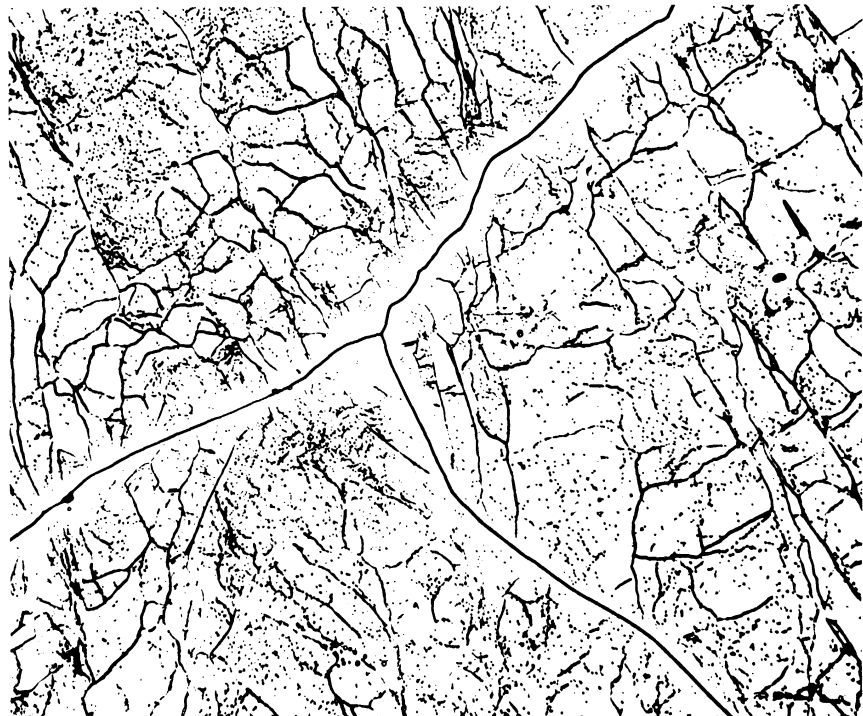
(c) 2.69 Percent Elongation; Decelerating Creep.  
Mag. 500X.

Figure 14 Continued.





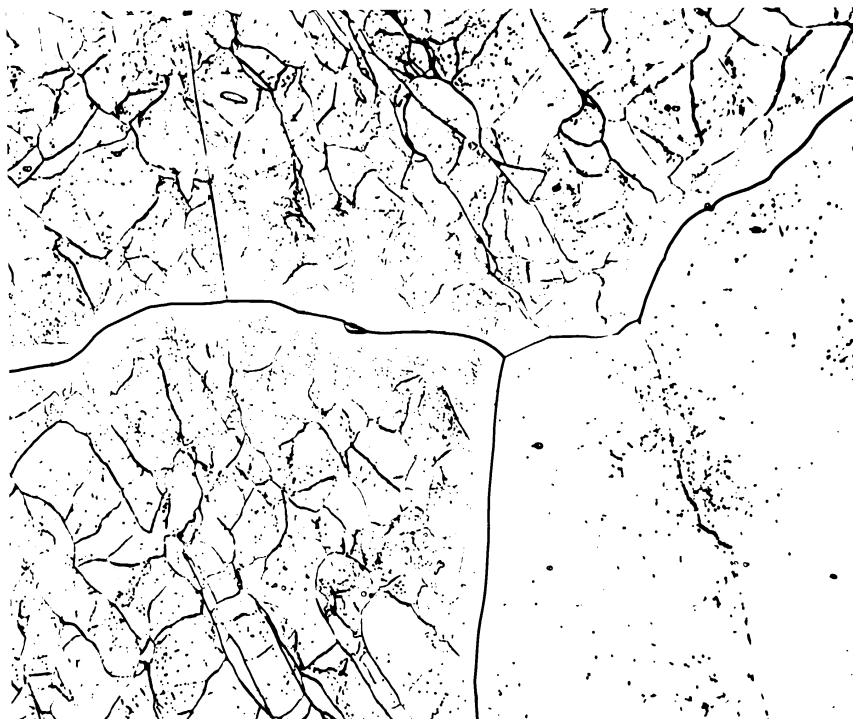
(d) 3.66 Percent Elongation; Minimum Creep Rate.  
Mag. 100X.



(d) 3.66 Percent Elongation; Minimum Creep Rate.  
Mag. 500X.



(e) 5.18 Percent Elongation; Accelerating Creep.  
Mag. 100X.

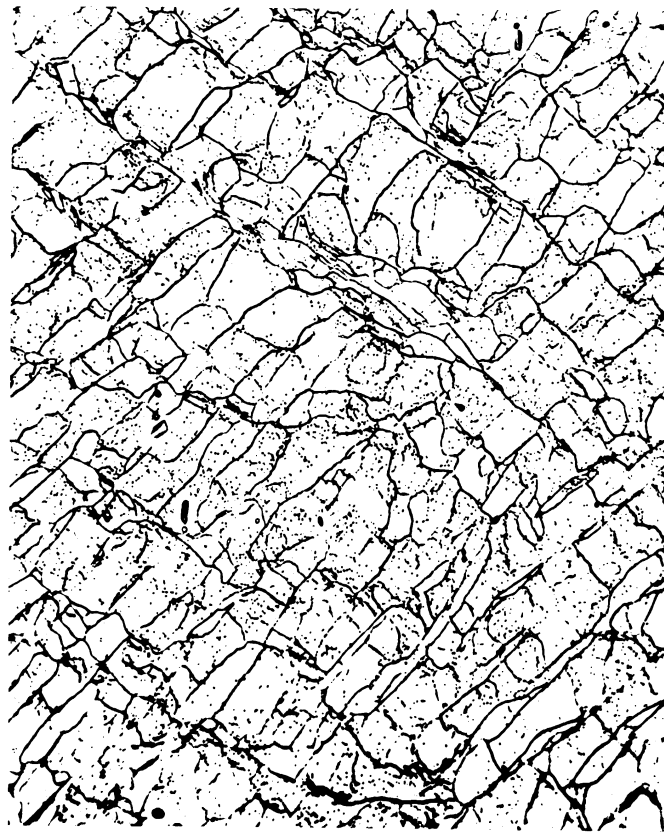


(e) 5.18 Percent Elongation; Accelerating Creep.  
Mag. 500X.

Figure 14 Concluded.



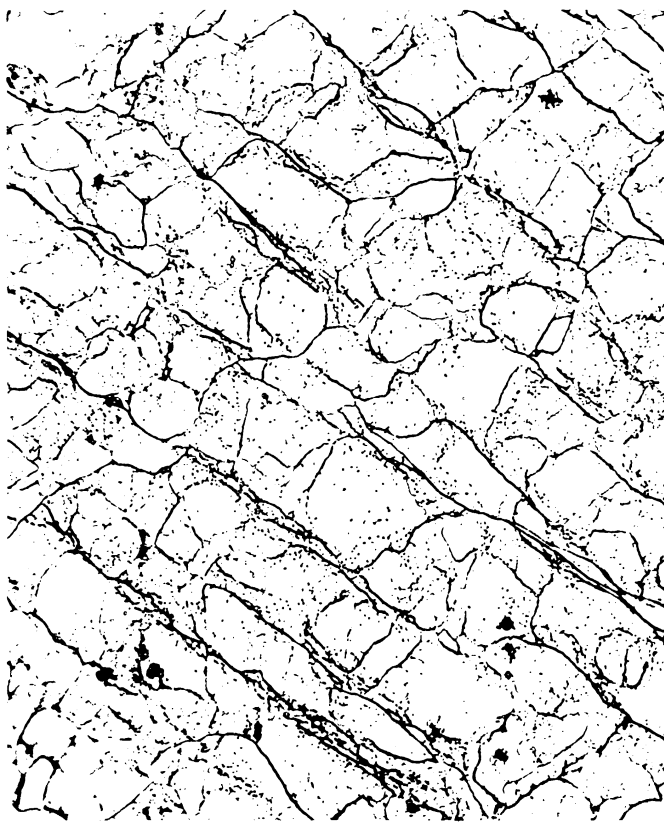
(a) 2.04 Percent Strain in 0.083 hr.



(b) 2.69 Percent Strain in 0.23 hr.

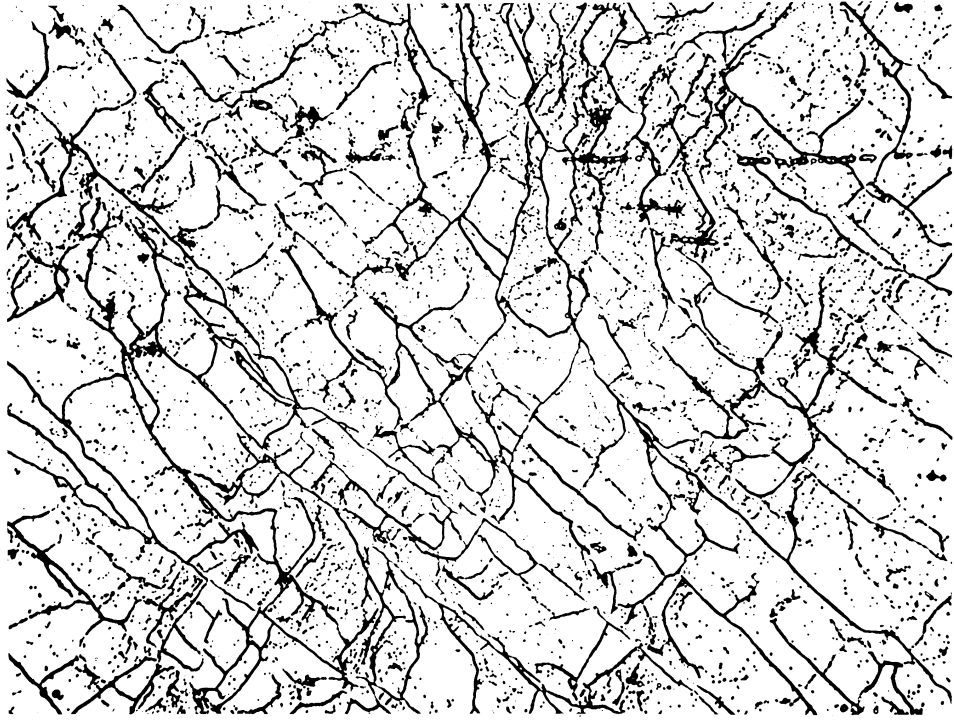


(c) 3.66 Percent Strain in 0.85 hr.

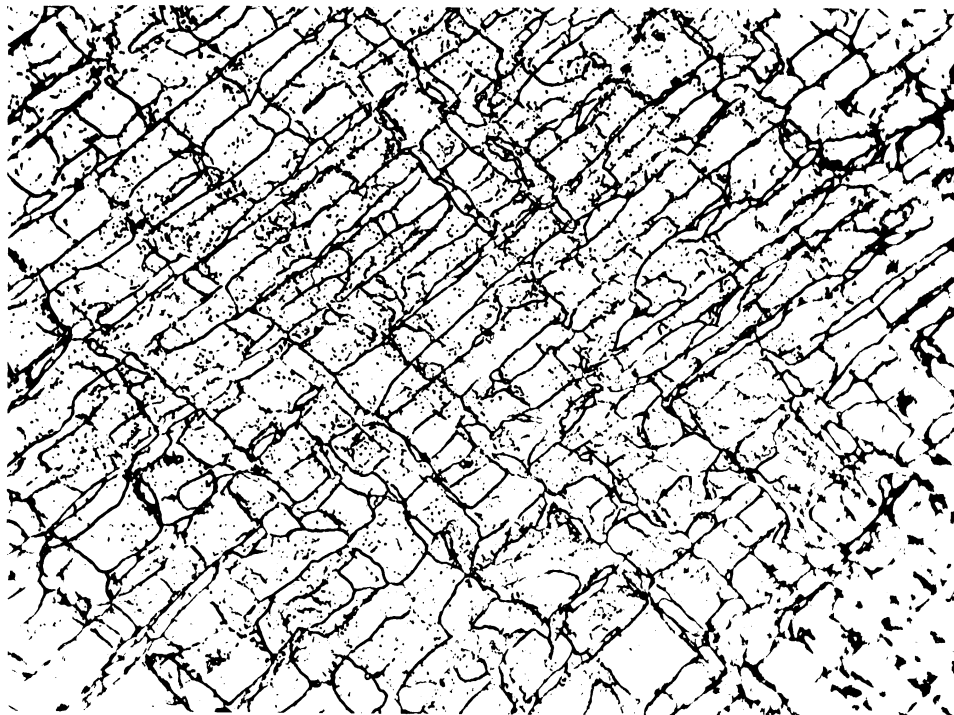


(d) 5.18 Percent Strain in 1.9 hr.

Figure 15 Subgrain Growth Within Areas of Maximum Sub-boundary Density in Low-Carbon "A" Nickel During Creep at 1550°F and 3360 psi. Tension Axis Vertical. Magnification 500X.

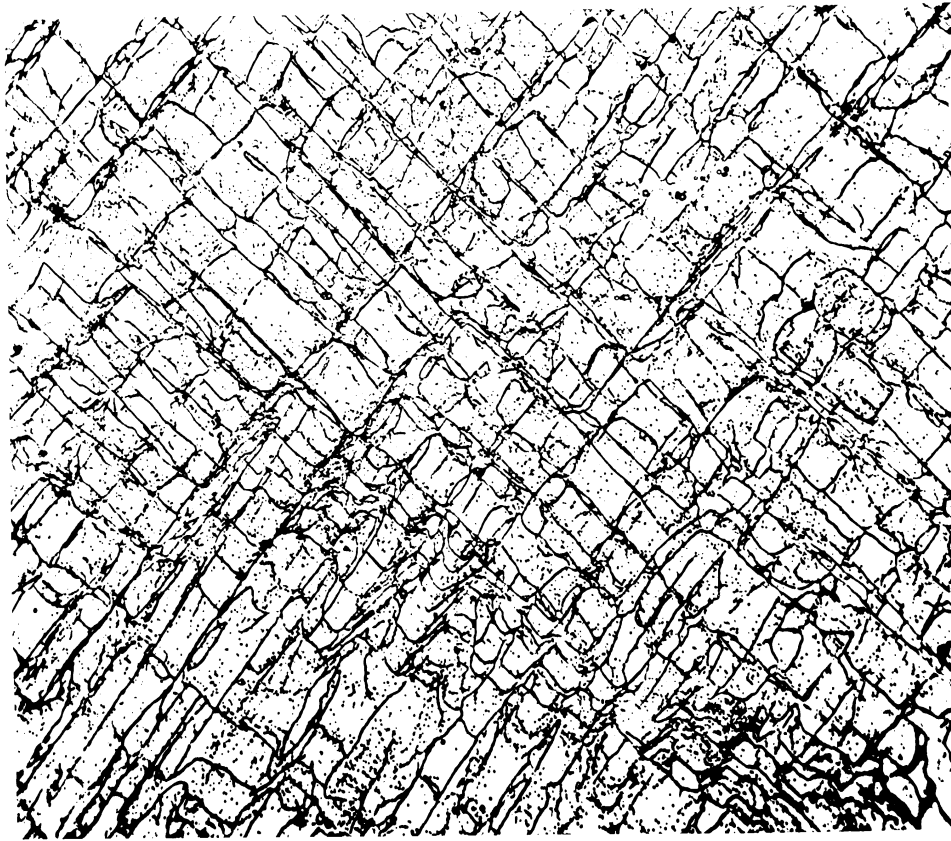


(a) Tested at 3,360 psi to 3.66 Percent Strain in 0.85 hours

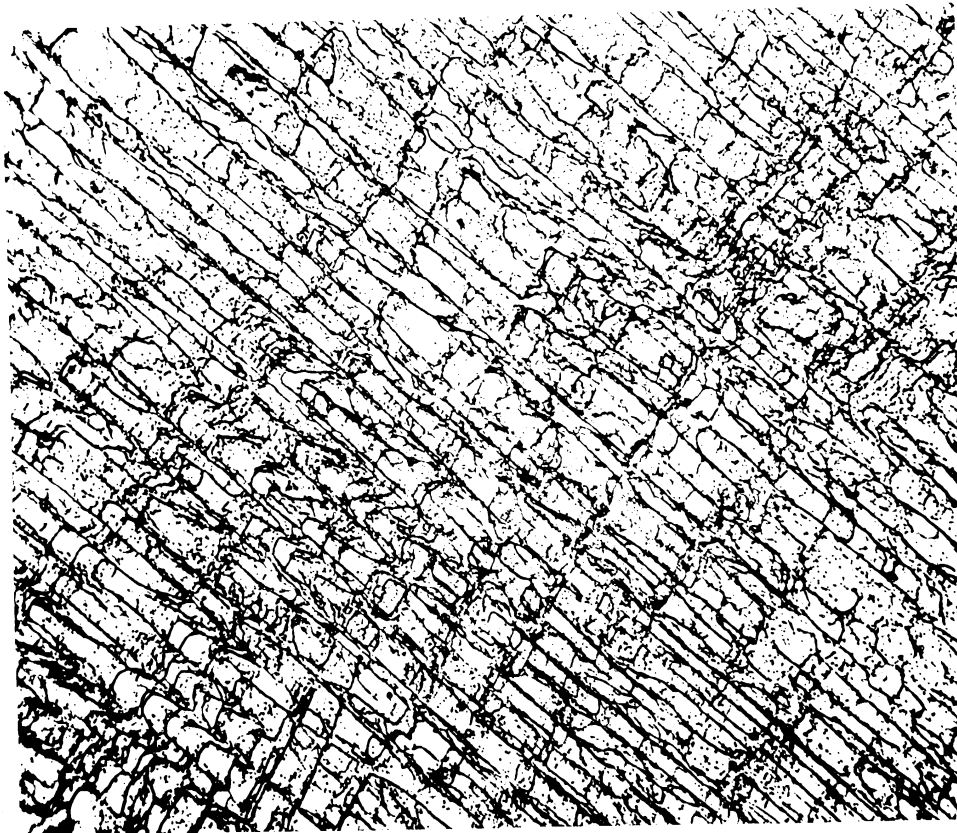


(b) Tested at 3,860 psi to 3.80 Percent Strain in 0.25 hours

Figure 16 Influence of Creep Stress on the Appearance of Areas of Maximum Sub-boundary Density in Low-Carbon "A" Nickel Creep Tested at 1550°F. Tension Axis Horizontal. Mag. 500X.



(c) Tested at 4,360 psi to 3.80 Percent Strain in 0.10 hours



(d) Tested at 5,360 psi to 3.68 Percent Strain in 0.022 hours

Figure 16 Concluded.

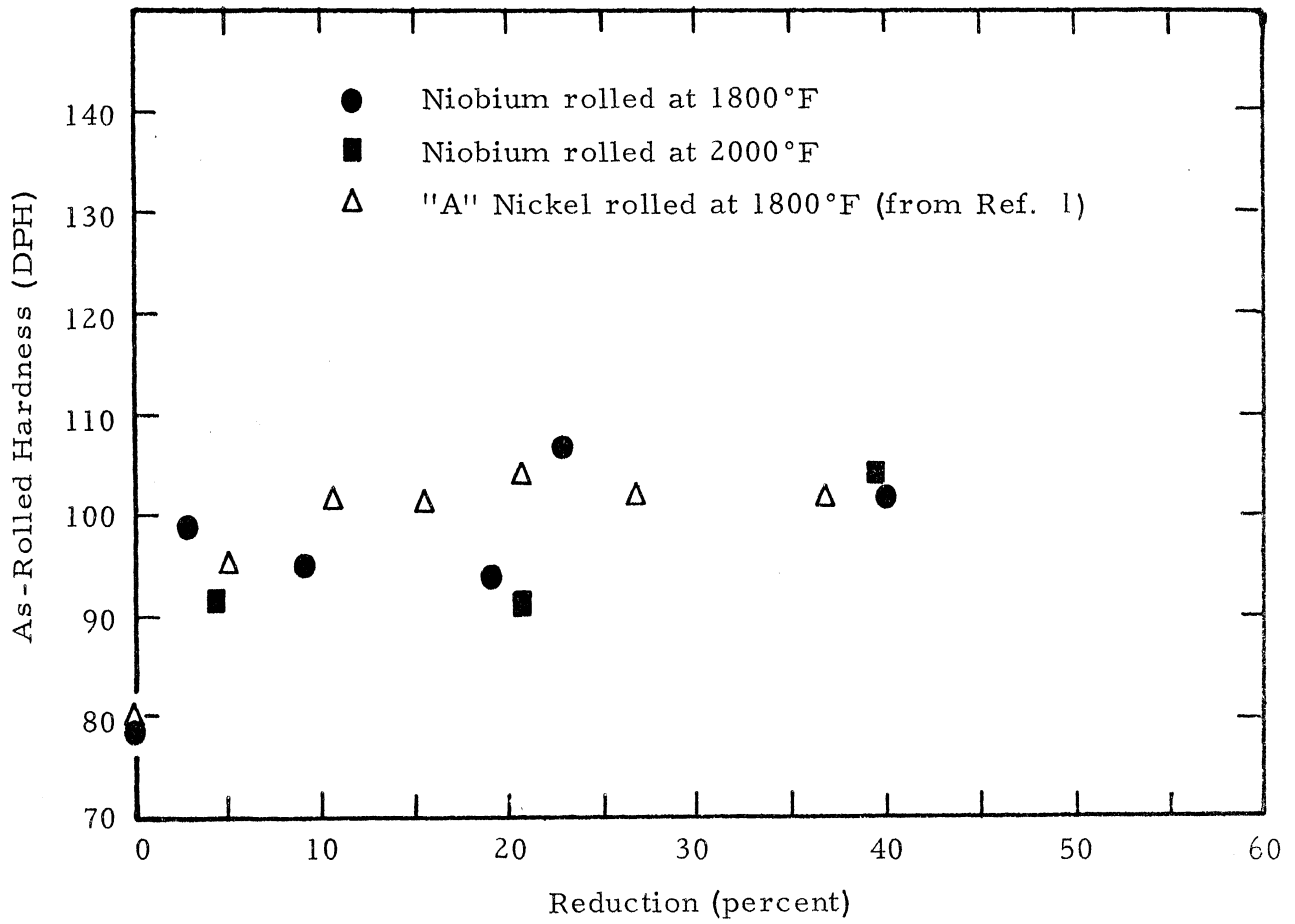


Figure 17 Effect of Amount of Reduction at 1800° or 2000°F on the As-Rolled Hardness of Pure Niobium

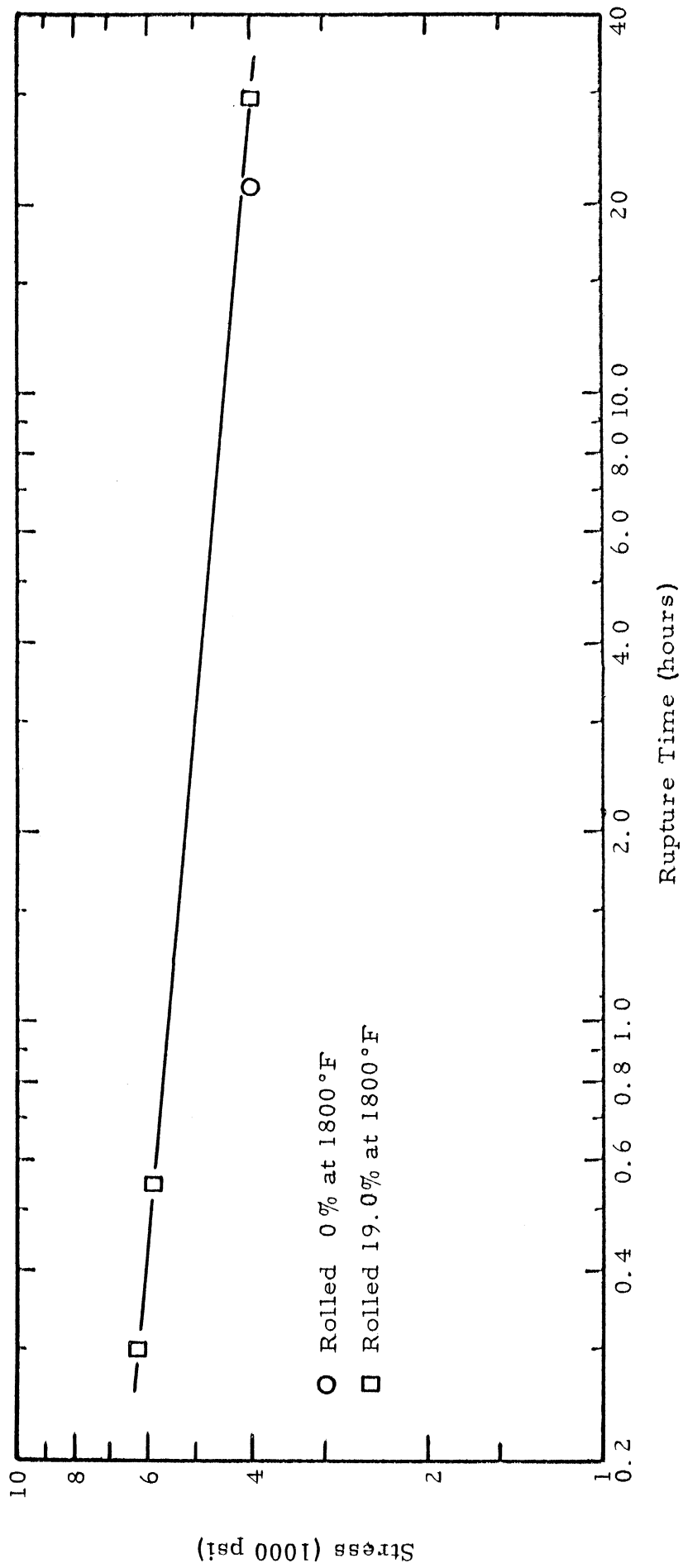


Figure 18 Stress-Rupture Time Data at 1800°F for Double-Electron-Gun-Melted Niobium

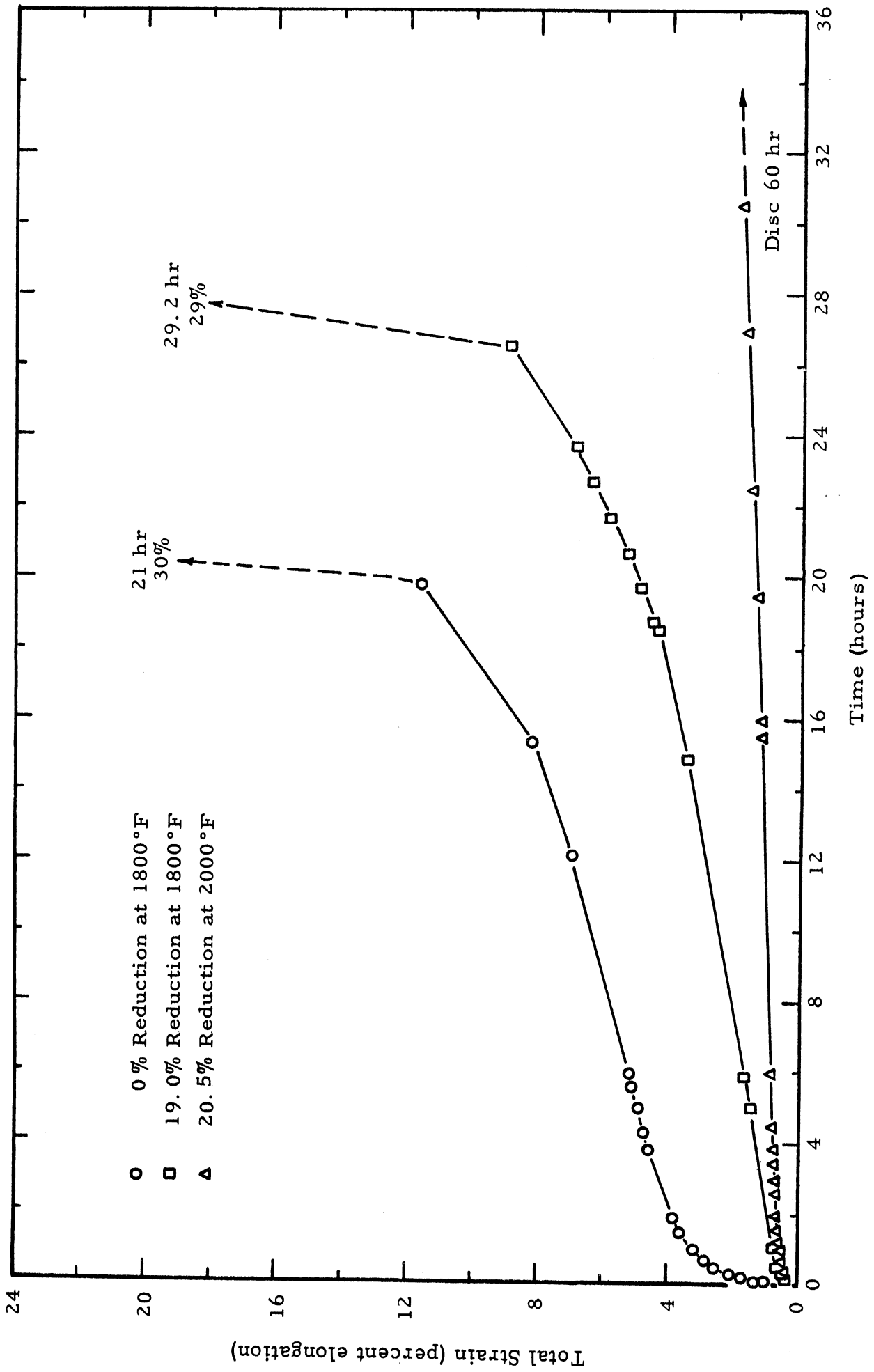


Figure 19 Time-Elongation Curves at 1800°F and 3940 psi for Double-Electron-Gun-Melted Niobium in the Indicated Conditions. Numbers on curves indicate rupture time and elongation.



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