ASD TECHNICAL REPORT

EFFECT OF CREEP-EXPOSURE ON MECHANICAL PROPERTIES OF 80Ni - 20Cr AND TZM MOLYBDENUM ALLOYS

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FOREWORD

This report was prepared by the University of Michigan, Department of Chemical and Metallurgical Engineering under USAF Contract No. AF 33(616)-6462. This contract was conducted under Project No. 7381, "Materials Application", Task No. 73810, "Exploratory Design and Prototype Development". The work was administered under the direction of the Materials Central, Deputy for Advanced Systems Technology, Aeronautical Systems Division, with Mr. W. H. Hill acting as project engineer.

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ABSTRACT

An extensive investigation was made of the influence of creep-exposure at 1000° to 1800°F on the mechanical properties of a structurally stable 80Ni - 20Cr alloy. Strain hardening and residual stresses raised strength and lowered ductility for creep at the lower temperatures. With increasing temperature of creep, this factor diminished. Internal microcracking in the grain boundaries, however, then occurred increasingly with exposure temperature and creep strain. A remarkably large amount of microcracking was required to reduce ductility and, when more severe, to reduce ultimate tensile strength. Two grain sizes were investigated. The fine grained material was less damaged by cracking than the coarse grain material for a given exposure. It was, however, subject to grain growth at intermediate temperatures with a loss in strength.

The results suggest that internal cracking had to be extensive for appreciable damage. This is probably due to the low strength and high ductility of the alloy. A higher-strength alloy would almost certainly show more damage to the properties from increased notch sensitivity. In other alloys studied previously, thermally-induced structural changes have been a major factor. Other than the instability of the fine grained material, there was little evidence of this for the 80Ni - 20Cr alloy. There were, however, impurities and minor phases present which could have been a factor.

Due to uncertainties of material and experimental difficulties, no conclusive results were obtained with TZM molybdenum alloy, 0.55Ti-.064Zr-.017C.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

W. J. Trapp
Chief, Strength and Dynamics Branch
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INTRODUCTION

Studies of the effects of elevated temperature creep-exposure on the short-time mechanical properties of aircraft structural metals have been conducted at the University of Michigan for several years under the sponsorship of Materials Central, Aeronautical Systems Division, under the present contract AF 33(616)-6462 and its predecessor AF 33(616)-3368.

Earlier studies (Refs. 1-6) were conducted on alloys of established creep resistance whose properties depended on some mechanism of strengthening, i. e. precipitation hardening or martensite transformation, which could be unstable under exposure to temperature and creep. These materials included 2024-T86 aluminum, C110M and Ti-16V-2.5Al titanium alloys, 17-7PH precipitation-hardening stainless steel, and Rene' 41 nickel-base alloy.

It was deemed essential that more insight be gained into the aspects of damage due to creep itself, relatively independent of microstructural changes. This is most easily performed through experiments on a material which is as free from thermally-induced changes as possible. Ideally, the material should undergo a transition in the characteristic creep fracture from transcrystalline to intercrystalline as the test temperature and/or time period of exposure is increased. It was expected that this would result in a transition in the effect of prior creep on mechanical properties from effects due to strain hardening to those due to cracking.

A binary alloy of 80Ni - 20Cr was selected as the material most nearly meeting the requirements of structural stability and simplicity for the requirements of the investigation. In addition, this composition is the basis for the large family of complex nickel-base alloys of which Rene' 41 is an example. Two conditions of grain size were selected for study; small grains and large grains prepared by heating the small grain material. Differences in the creep properties were expected between large grain and small grain material prepared in this manner. The effects of creep-exposure on short-time mechanical properties were to be evaluated by room temperature tensile and impact tests and supporting metallographic studies.

In addition to the 80Ni - 20Cr alloy, tests were to be conducted on an alloy representative of the high melting-point or "refractory" alloys gaining in importance for future Air Force usage. This was tentatively selected to be a binary Mo-0.5Ti alloy, however, the Mo-0.55Ti-0.064Zr (TZM) alloy was later substituted as being of more promise. These tests were to be conducted in vacuum on uncoated specimens or in air on coated specimens.

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TEST MATERIALS

80Ni - 20Cr ALLOY

Alloys of the basic 80Ni - 20Cr composition are produced commercially for use as resistance heating elements. Because of their ready availability, it was decided to procure one of the commercial compositions.

Approximately 112 pounds of Chromel-A alloy were received from the Hoskins Manufacturing Company in the form of hot-rolled 1/2-inch diameter bar stock with the designation of Heat No. 1067. The following chemical analysis was furnished:

Element	Percentage (wt.)
Chromium	20.44
Nickel	77.48
Carbon	0.01
Silicon	1.41
Manganese	0.15
Iron	0.31
(Others - by difference)	0.20

The test stock was cut into 4-inch long specimen blanks and annealed in groups of 24 blanks each to produce either small grain or large grain material according to the following schedule:

Small grains (ASTM Size 10.4) -- 1550°F - 4 hours
plus air cool (Treatment coded "C1")

Large grains (ASTM Size 4.1) -- 2100°F - 1 hour
plus air cool (Treatment coded "C2")

The experiments leading to the selection of these heat treatments are described in a later section of this report (see pages 7-10).

TZM MOLYBDENUM ALLOY

The procurement of the molybdenum alloy was delayed until availability of a vacuum testing unit. Originally, it was intended to study the binary Mo-0.5Ti alloy, however, in the interim, communications with ASD representatives indicated the considerable promise of a ternary alloy Mo-0.5Ti-0.08Zr (TZM alloy) -- with the provision that carbon content be 0.02 percent or lower -- and when it was determined that stock of this alloy was available, it was procured.

Approximately 69.5 pounds were purchased from the Climax Molybdenum Company of Michigan. The material was identified as being all from Heat TZ-6001 and was received in the form of 8 5/8-inch diameter stress-relieved bars, each approximately 6 feet long, and one bar 30-inches long. The Diamond Pyramid Hardness was reported to range from 283-293. The reported chemical analysis follows:

Element	Wt. Percent
Titanium	0.55
Zirconium	0.064
Carbon	0.017
Oxygen	0.00048
Hydrogen	0.000005
Nitrogen	0.0009
Aluminum	0.0008
Calcium	0.0002
Iron	0.0016
Silicon	0.0026
Tin	<0.0010
Lead	<0.0010
Tungsten	<0.0100
Cobalt	<0.0005
Copper	<0.0001
Nickel	<0.0001
Manganese	<0.0001
Magnesium	<0.0001
Chromium	<0.0001
Molybdenum	balance

The material was tested in the stress-relieved condition with no additional heat treatment. No information was available for the stress-relief conditions, however, this is usually accomplished by heating for one hour at 2100°-2200°F (Refs. 19, 20).

TEST SPECIMENS

Details of the test specimens for studies of the 80Ni - 20Cr alloy are shown in Figure 1. The test specimens used for studies of the TZM molybdenum alloy are discussed separately (pages 22-23 and Fig. 64)

Creep-exposure of the 80Ni - 20Cr alloy was conducted on specimens with a 0.350-inch diameter gage section and a reduced section of approximately 2 inches. The specimens for subsequent mechanical property testing were designed to be machined from the gage section of the creep-exposure specimens. In order

to eliminate the effect of surface attack in the tensile tests, approximately 0.025-inches were machined from the gage section diameter of the exposed creep specimens.

The ASTM Type-W notched sub-sized specimen was used for impact tests. The dimensions, 0.197-inches square by 2.16-inches long, were such that it could be conveniently machined from the reduced section of a creep specimen. The notches were roughed in with a special notch cutter to establish the angle and root radius and then ground out to the final dimensions.

EQUIPMENT AND PROCEDURES

CREEP-EXPOSURE AND CREEP-RUPTURE TESTS

The creep-exposure and preliminary creep-rupture tests on the 80Ni - 20Cr alloy were conducted in individual University of Michigan creep-testing machines. Equipment and procedures for the TZM molybdenum alloy are discussed elsewhere (page 21). Stress was applied through a third-class lever system having a lever-arm ratio of about 10 to 1. The specimen was gripped by threaded holders that fitted into a universal joint assembly for uniaxial loading. Heating was provided by a wire-wound resistance furnace fitting over the specimen and holder assembly. Strain measurements were made with a modified Martens optical extensometer system which permitted the detection of a specimen elongation of approximately 10 millionths of an inch. Correction factors were used to account for the diminished contribution of the specimen shoulders and fillets to the observed deformation (Ref. 3).

Temperature measurements were made from three chromel-alumel thermocouples wired to the gage section. Asbestos cord was used to shield the thermocouple beads from direct furnace radiation. Prior to starting a test, the furnace was heated to within 50°F of the test temperature. The specimen was then placed in the hot furnace and was brought up to proper temperature and distribution in a period of four hours before the load was applied. ASTM Recommended Practices were used in controlling both temperature and distribution.

Strain measurements were made as each weight was applied during loading and then periodically throughout the test. At the end of the exposure period, a final reading was made and the power to the furnace turned off. The specimen was then cooled under load in order to minimize the effects of creep recovery.

The fracture time in creep-rupture tests was measured by an automatic timer, accurate to one-tenth of an hour, that was actuated by the fall of the specimen holder.

TENSILE TESTS

Short-time tensile tests were conducted in a hydraulic testing machine. Elongation was measured with a microformer strain gage and recorded automatically as a curve of load versus elongation. A strain pacer was used to maintain a strain rate of 0.005 inches per inch per minute. The data determined in the tensile tests were the ultimate strength, 0.2-percent offset yield strength, elongation, reduction of area, and the modulus.

IMPACT TESTS

Izod notched bar impact tests were run on the ASTM Type-W sub-sized specimens. In this test, the specimen is gripped at one end and mounted as a vertical cantilever. A special holding fixture was constructed to accommodate the sub-sized specimen. The impact machine was set to produce a striking energy of 120 foot-pounds. Prior to running a test, the measuring scale of the machine was zeroed by allowing the pendulum to swing through its cycle with no specimen in place and noting the height to which the indicator was raised.

In addition to measuring the impact energy, the ductility characteristics of the fractured specimens were determined by measuring the lateral contraction at the base of the notch and the lateral expansion at the surface opposite the notch.

STRUCTURAL EXAMINATION

The techniques used for structural studies of the 80Ni - 20Cr alloy included optical microscopy, electron microscopy, x-ray diffraction of extracted residues or of filings from the specimens, and hardness tests.

Specimen Preparation

Specimens for microscopic examination and hardness testing were sectioned longitudinally with a water-cooled cut-off wheel and then mounted in Bakelite. The mounted samples were wet ground on a rotating lap through a series of silicon carbide papers finishing at 600-mesh grit. Final polishing was carried out on a cloth-covered rotating lap using fine diamond compound and then on a vibratory polisher in an aqueous media of Linde "B" polishing compound. The samples were cleaned ultrasonically in a detergent solution.

Etchants

Similar etching techniques were used for both optical and electron microscopy. The 80Ni - 20Cr alloy was etched by swabbing in Marble's reagent for

3-5 seconds. The TZM molybdenum alloy was etched in a similar fashion in Murakami's reagent. The composition of the etchants follows (Ref. 7):

Marble's Reagent		Murakami's Reagent	
$CuSO_4$	20gm	$K_3Fe(CN)_6$	10gm
HCl (conc)	100ml	KOH	10gm
H_2O	100ml	H_2O	100ml

Electron Microscopy

Electron microscopy was carried out in an RCA EML Electron Microscope. For examination in the microscope, collodion replicas were prepared from the surface of the etched specimens and mounted on nickel grids. The replicas were shadowed with palladium to increase the contrast and reveal surface contours. Polystyrene latex spheres of either 2580 or 3400 Å diameter were placed on the replicas prior to shadowing to indicate the angle and direction of shadowing. The micrographs reproduced in this report are direct prints from the original negatives; consequently, the polystyrene spheres appear black and the shadows appear white. Since the spheres are raised from the surface of the replica, a particle casting a shadow opposite to that of the spheres is in relief in the metal specimen; conversely, areas casting shadows in the same direction as those cast by the latex spheres are depressions in the surface of the metal specimen and correspond to material that was attacked or eaten out by the etchant.

X-ray Diffraction

X-ray diffraction analysis of the 80Ni - 20Cr alloy was used both for the determination of the lattice parameter and for the identification of minor phases.

Samples of the as-treated C1 and C2 conditions were studied for differences in parameter. Very fine filings were made from each sample using a thin file. The filings were sifted with a magnet to remove any iron particles that might have come from the file and then sifted through a 200-mesh screen. The resultant fine powder was rolled into a thin filament using a Duco cement binder. This was exposed to unfiltered cobalt radiation for 4 hours in a 114.6 mm diameter Debye camera. The lattice parameters were determined by the extrapolation method of Bradley and Jay (Ref. 8).

The identification of minor phases was made from analysis of the powder patterns from residues extracted from the solid samples by immersion in a bromine-methyl alcohol solution. The extraction procedures were described detail previously (Refs. 1, 6). The extracts were formed into thin filaments and exposed to nickel-filtered copper radiation for 4 hours in a 114.6 mm diameter Debye camera. The line positions were determined on an optical comparator

and "d" values were calculated. The patterns were analyzed by comparison with standard patterns available from the ASTM X-ray Powder Data File.

Hardness Measurements

Diamond Pyramid Hardness measurements were made on a Vickers Hardness Machine using a 50 kilogram load. Both diagonals of each of three impressions were measured and the six measurements averaged. Knoop microhardness traverses were run on several samples using a Tukon Superficial Testing machine at 1000 grams load.

RESULTS AND DISCUSSION

Creep-exposures followed by room temperature tensile tests were used to evaluate creep-damage to mechanical properties of the 80Ni - 20Cr alloy and the TZM molybdenum alloy. The exposures of the 80Ni - 20Cr alloy were conducted in air, and the few exposures conducted on TZM molybdenum were run in vacuum. Metallographic examination was directed towards delineation of cracking.

STUDIES OF THE 80Ni - 20Cr ALLOY

After initial studies to establish the heat treatments, the 80Ni - 20Cr alloy was tested in either a large grain or a small grain condition. The large grains were produced by heating the small grain material. Survey creep-rupture tests established the relative properties of the two conditions.

Creep-exposures were conducted generally from 5 to 100 hours at 1000°-1800°F. Both room temperature tensile tests and impact tests on notched sub-sized specimens were used to evaluate properties. Metallographic studies were made to study the cracking of the material and the possible role of minor phases.

Establishment of Heat Treatments for 80Ni - 20Cr Alloy

The desirability of studying a range of grain size was evident from data of Kozyrski, Kononenko, and Okrainets (Ref. 9) showing an appreciable difference in the creep of an 80.2Ni - 19.8Cr alloy when the average grain diameter was increased from 0.02-0.03 mm to 0.5-0.6 mm (approx. ASTM 8 to -1) by annealing the small grain material for 64 hours at 2140°F. The coarse grained material then exhibited considerably greater creep resistance in tests at 1290°F and 14,200 psi. For the present investigation, grain sizes of ASTM 1-2 and ASTM 7-8 were tentatively selected to allow comparable variation in creep resistance.

After consultation with the supplier, it was decided to obtain the 80Ni - 20Cr alloy in the hot-rolled condition with their recommendation for the final annealing treatment to produce the desired grain sizes. In this way, it was possible to avoid cold straightening required after coil annealing in the supplier's furnaces.

In a series of preliminary experiments, the supplier annealed hot-rolled samples for one-half hour each at temperatures between 1600° and 2000°F. Both the as-hot rolled material (Fig. 2) and the sample annealed at 1600°F had fairly uniform fine grains, however, difficulty was encountered in producing uniform large grains at higher annealing temperatures. In a sample annealed at 2000°F (Fig. 3), a mixture of two widely different grain sizes was produced with a coarse zone part way between the surface and center of the sample. A similar effect was noted in a sample pre-annealed one-half hour at 1600°F before final annealing at 2000°F.

These effects were probably due to germination caused by a critical amount of residual strain as a gradient from the surface to the center of the rolled bar.

The presence of such a gradient in the as-hot rolled bar stock was later confirmed by a Knoop microhardness survey (Fig. 5a), which showed a decrease in the hardness from the surface to the center.

A preliminary sample of the hot-rolled stock was then obtained from the supplier for further experimentation at the University. One sample was annealed for an hour at 2100°F (Fig. 4) and another was pre-annealed for 24.2 hours at 1550°F prior to annealing one hour at 2100°F. In both samples, large grains were obtained throughout the cross-section without an excessive gradient in grain size. The average grain size produced by various annealing conditions follows:

Heat Treatment	Average ASTM Grain Size No.	Average Grain Diameter (mm)
As-Hot Rolled	10.4	0.0109
1600°F - one-half hour	10.4	--
1550°F - 24.2 hours +2100°F - 1 hour	4.4	--
2100°F - 1 hour	4.1	0.098

These data are plotted in Figure 6 to show the effect of annealing temperature on grain size. This figure also includes additional data taken for another investigation on samples which had been cold-worked prior to annealing. The purpose of the cold work was to remove the critical strain gradient by increasing the over-all level of strain. Apparently, more than 24 hours annealing was required to produce large grains at temperatures below 2000°F.

Several samples were also annealed for various times at 1550°F and

Vickers hardness readings were made at a point midway between the surface and the center with the following results:

Sample	Vickers Pyramid Hardness
As-Hot Rolled	198
1550°F - one-half hour	204
- one hour	202
- two hours	206
- four hours	199
- 24.2 hours	193

In addition, Knoop microhardness surveys were run on the samples annealed for one, four, and 24 hours at 1550°F (Fig. 5b, c, d).

These data indicate that the four hour anneal removed the original surface-to-center hardness gradient although the over-all hardness level was apparently increased slightly by the one and four hour anneals and then decreased by the 24.2 hour anneal. This observation agreed with the trend of the Vickers hardness data and suggested that the material aged slightly with a peak effect occurring in two hours. The grain size produced by the four hour anneal at 1550°F was ASTM No. 10.3, while the 24.2 hour anneal produced a grain size of ASTM No. 9.5.

Based on these observations, it appeared desirable to anneal the hot-rolled material for at least four hours at 1550°F and use this condition as the fine-grained material for the creep-exposure studies. This caused a negligible amount of grain growth but did remove the strain gradient.

With a size of ASTM No. 10.3 as the fine grain condition, it was felt that the coarse grains of ASTM Size No. 4.1 produced by the 2100°F anneal would provide a sufficient variation for the purposes of the investigation. This would be a difference of six ASTM numbers, a spread comparable to that intended under the tentative specifications of ASTM 1-2 and ASTM 7-8.

However, before finally fixing the annealing conditions, cognizance was taken of the slight over-all hardening produced by the 1550°F anneals. Tests were made to determine if the tensile properties were affected by annealing for one, four, or 24 hours at 1550°F. The results of room temperature tensile tests are summarized in Table 1 and plotted as a function of annealing time in Figure 7. Figure 7 also includes the Vickers hardness data taken on the original metallographic slugs.

The results indicate that increasing the annealing time caused a slight decrease in the ultimate tensile strength and the tensile yield strength. There was little, if any, effect on the ductility. The principal change in strength occurred in the first four hours, with little further effect resulting from increasing the time to 24 hours.

The Vickers hardness data did not appear to correlate with the mechanical property changes.

Replicas for examination under the electron microscope were made of an as-hot rolled sample and the sample aged four hours at 1550°F. These indicated the presence of a minor phase or phases in the alloy. An increase in the number of discrete precipitate particles in the grain boundaries occurred as the annealing time was increased (Figs. 8 and 9). A rough estimate of the particle density was made by counting the number observed at 2200x in one grid of the nickel screen on which the replica rested. The following results were obtained:

Sample	No. of particles at 2200x per unit area
As-hot rolled-center	66
1550°F - four hours - edge of bar	98
- center of bar	120

The increased particle density appeared related to increased hardness.

On the basis of the mechanical properties, the microstructure, and the microhardness data, the four hour anneal at 1550°F was then selected to provide relatively strain-free, fine-grain material of ASTM Size No. 10.3 for the creep exposure tests. This treatment was coded "C1". The one hour anneal at 2100°F was chosen to provide the large grain material of ASTM Size No. 4.1 and was coded "C2".

Base Properties of 80Ni - 20Cr Alloy

Short-Time Properties

Tensile tests at room temperature (Table 1) were run on specimens from different heat treatment lots to establish base properties for comparing the effects of creep-exposure. The large grain "C2" material had a somewhat lower ultimate strength, a considerably lower yield strength, and an appreciably increased ductility in comparison to the small grain "C1" material.

Creep-Rupture Properties

Creep-rupture tests were run on material in both heat treatment conditions to obtain data for selection of creep-exposure conditions and to provide a further basis for comparing the effects of grain size. In order to provide variations in strain rate and exposure temperature, the test stresses were selected to cause rupture in approximately 5 to 100 hours at temperatures between 1000° and 1800°F. The stresses were selected with the aid of a master rupture curve derived from

data of Shahinian and Achter (Ref. 10) for tests of sheet material of similar composition and an average grain diameter of 0.09 mm (ASTM 4.3) produced by annealing for 20 hours at 2000°F. This grain size was extremely close to the 0.098 mm diameter (ASTM 4.1) of the present "C2" material. In the initial tests, the same stresses were used for both the "C1" and "C2" conditions.

The test data show (Table 2) that the large grain "C2" material had appreciably better rupture strength than the small grain "C1" material in contrast to the better room temperature tensile properties of the small grain material. On the other hand, the elongation of the "C2" material in the rupture tests was less than that of the "C1" material.

The master rupture curve (Fig. 10) for the large grain material agreed quite well with the curve derived from the data of Shahinian and Achter for material of almost the same grain size. The curve for the small grain material was displaced towards lower parameter values (shorter time for rupture at a given stress and temperature) and the individual test points also had a considerably greater scatter at parameter values corresponding to the higher temperature, longer time tests. In several of the tests reported in Table 2, the elongation on fracture exceeded the reduction in area. This indicated that extensive interior cracking had occurred.

The creep curves in Figures 11-15 show that the large grain material possessed appreciably higher creep resistance than the small grain material at all temperatures of testing. Master curves (Fig. 16) show this for 5-percent creep. Such variations in creep resistance are expected when large grain material is prepared by simply heating the small grain material (Refs. 11, 12). For both conditions, the creep was rapid and extensive under the conditions studied. With the exception of a few tests at 1000°F, the creep rate accelerated continuously through the test period.

Effect of Creep-Exposure on Tensile Properties of 80Ni - 20Cr Alloy

Creep-exposures were conducted primarily for 5, 10, 50, or 100 hours at temperatures between 1000° and 1800°F. Following exposure, 0.025-inches were machined from the gage section diameter and the specimens were tensile tested at room temperature. In addition to the elevated temperature exposures, several specimens were pre-strained various amounts at room temperature in the tensile machine before tensile testing for comparative purposes. Most of the short-time creep-exposures were conducted for 5 hours as a convenience.

The test data are summarized in Tables 3 and 4 for the small grain and large grain materials, respectively. Individual plots of total plastic strain versus residual room temperature mechanical properties for each exposure temperature are included in Figures 17 and 18. Comparisons of the effect of creep on the two grain sizes are presented in Figures 19 and 20, while the time-elongation curves from the creep-rupture tests and creep-exposures are plotted

in Figures 11-15. In most cases, the creep rate accelerated continuously from the start of the test.

The influence of creep-exposure on the tensile properties at room temperature was qualitatively the same for both grain sizes (Figs. 17 and 18). Exposure at the lower temperatures raised both ultimate and yield strengths. The increase in yield strength relative to the original strength (Fig. 19) was less for the fine grain than for the coarse grain material. (In using Figure 19, it is necessary to recognize that the ratios are relative to the strength before exposure. For instance, the lower relative yield strength of the fine grained material is due in part to the initially higher yield strength.) As the temperature of exposure increased, the increase in ultimate tensile and yield strengths became less and finally the strengths were reduced rather than increased by prior creep-exposure. Large amounts of creep at 1600° or 1800°F were required to reduce the strengths below that of the original material with the coarse grained material requiring more severe exposure than the fine grained. In no case were the yield strengths of the coarse grained reduced below the original. Exposure time did not have much effect on yield strength, except to produce somewhat lower values after the longer exposures at 1200° and 1400°F. The larger strains at 1600° and 1800°F reduced the tensile strength of the fine grained material to very low values. The tensile strengths of the larger grained material were less after prolonged exposure than for short exposure at 1400° to 1800°F.

The relationships of the actual strengths, as influenced by exposure temperature and amount of creep, are compared on a more quantitative basis for the two grain sizes by Figure 20. For 5 hours hours of exposure, the fine grained material fell off in strength for lower exposure temperatures the larger the amount of strain and became lower than the coarse grained material for the higher exposure temperatures. There was less evidence of this for exposure in 100 hours and the strengths stayed above the coarse grained material except for exposure at 1800°F.

Ductility was reduced below that of the original condition by all creep exposures (Figs. 17 to 20). In most cases, the reduction was somewhat more for the longer exposures at a given strain. The ductility of the coarse grained material was reduced more by a given amount of strain than that of the fine grained material except for 1800°F exposure. Exposure temperature had little effect on the fine grained material until the temperature was raised to 1800°F. The amount of strain was the controlling factor. For the coarse grained material, increasing the temperature of exposure above 1200°F reduced the ductility markedly for a given strain.

The plastic pre-strain at room temperature was included in the investigation to help clarify the observed effects on properties by providing a comparison condition where there were no temperature or time effects.

It will be noted that in general the effect of a given amount of strain at room temperature was about the same as at 1000° or 1200°F. Exposure temperature

had to be higher before temperature of exposure was much of a factor. The major exceptions were large strains in prolonged time periods.

Limited data (Tables 3 and 4) indicate that exposure without creep had little effect on properties. The fine grained material at 1800°F was an exception due to the reduced strength and increased ductility. Plastic strain otherwise was required to change tensile properties.

The creep curves for exposure at elevated temperatures are included in Figures 11 to 15. In most cases, the creep rate increased from the start of the tests under the exposure conditions used. In the exposures of the fine grained material at 1000°F and the coarse grained at 1000°F and 1200°F, the plastic yielding during loading was an appreciable part of the total strain. At the higher temperatures, creep strain was predominant. Because previous research (Ref. 6) had indicated that there was no difference from short-time strain and creep strain on these properties, the total plastic strain was used in plotting Figures 17 through 20.

The creep curves for Specimens C1-28, C1-55, and C1-15 of fine grained material at 1800°F were unusual. There was a period of relatively slow creep followed by acceleration for a time and then the curve levelled off again before the exposures were interrupted. Figures 21 and 22 show the extensive surface cracking on these specimens. When tensile tested at room temperature without re-machining, they exhibited negligible ductility. The fracture surfaces were nearly completely oxidized and the specimens were riddled internally with cracks. A similar creep curve was reported in Reference 10 for creep in longer time periods at lower stress levels.

Effect of Creep-Exposure on Impact Properties of 80Ni - 20Cr Alloy

Izod impact tests on sub-sized notched specimens were used to evaluate the properties of the large grain and small grain material at room temperature after 5 hours prior creep at temperatures between 1000° and 1600°F. The test data are included in Tables 3 and 4 and plotted in Figure 23.

In the as-treated condition, the small grain material had slightly higher impact strength than the large grain material (11.5 ft-lbs versus 10 ft-lbs), however, the lateral expansion and contraction at the notches was about the same.

Creep-exposure reduced both the impact strength and ductility with fairly substantial amounts of creep required to effect a significant reduction. In the specimens exposed at 1000° and 1400°F, approximately 20-25 percent creep was required, while in the exposures at 1600°F approximately 8 percent creep was sufficient. After careful study of the data, it was concluded that there was no significant difference between the small grain and large grain materials in the response of their impact properties to prior creep. At first inspection, Figure

23 appears to indicate that the small grain material was more susceptible to decreased properties, however, comparisons up to about 10 percent strain (which was the limit of the available data for the large grain material) revealed no difference between the conditions that could not also be attributed to experimental scatter. The decreased strength for small grain material strained 20 percent at 1000°F and 25 percent at 1400°F and for both materials at 1600°F is substantial and beyond the range of possible scatter. On this basis, it appears that the impact properties were a less sensitive indicator of creep damage than the tensile properties -- particularly with respect to the behavior of the tensile elongation and reduction of area.

Microstructural Studies of 80Ni - 20Cr Alloy

Metallographic examination of the 80Ni - 20Cr alloy was made to study the extent of grain growth and cracking during creep-exposure and also to determine if minor phases were a factor. Optical microscopy was used for the grain growth and cracking studies and x-ray and electron microscope examination were used for minor phase studies.

Optical Examination of 80Ni - 20Cr Alloy

Specimens used for optical examination had been crept to rupture at elevated temperatures or creep-exposed and then tensile tested at room temperature.

Micrographs of a representative group of ruptured specimens are presented in Figures 24-29. For both the small grain and large grain material, extensive intergranular cracking occurred. In the small grain material, there was a tendency for the cracks to link together that was not as apparent in the large grain material. Grain growth occurred in the small grain material during testing at 1600° and 1800°F. There appeared to be little, if any, additional grain growth in the large grain material. The cracks occurred in boundaries transverse to the tension direction of the specimens. The fractures were intergranular.

In both materials, surface cracking was also observed. An example of this is shown for the large grain material in Figures 30 and 31 for two specimens tensile tested after exposure to approximately 14 percent creep in 10 hours at 1400°F. Figure 30 shows the specimen tensile tested without having the surface re-machined. The ultimate strength and ductility were both somewhat lower than the corresponding re-machined specimen of Figure 31. The surface condition thus had an effect on tensile properties even in the presence of extensive internal cracking. This result confirmed the need for re-machining test specimens in order to restrict the mechanical property effects to those due to internal changes in the specimens.

Micrographs of the small grain material after creep-exposure and tensile testing are presented in Figures 32-43. Grain growth during creep-exposure appeared to start at 1600°F (Fig. 36), with 50-100 hours exposure required

before it became appreciable. The grain growth was fairly uniform for the 50 hour exposures, however, in a 100 hour exposure (Fig. 38) bands of much larger grains were observed in the longitudinal direction of the specimen. Fairly uniform large grains were found in the specimens creep-exposed at 1800°F (Figs. 40-43). While exposure without stress for 100 hours at 1800°F also caused grain growth (Fig. 42), the grain sizes were mixed. For the specimens exposed under stress for 5 or 50 hours (Figs. 40 and 41), the grain size appeared to be larger and more uniform than in the specimen exposed without stress.

Cracks were observed in the transverse boundaries of small grain specimens exposed to creep at 1400°F and above. For the specimens exposed 50 hours at 1600°F (Figs. 36 and 37), an increase in the strain from 11 percent to 41 percent caused the cracks to link together. In the specimen with a mixed grain size (Fig. 38), the cracking was concentrated at the transverse boundaries of the coarse grains. In a specimen exposed to 5 hours creep at 1600°F and then sectioned for metallographic examination without tensile testing (Fig. 39), the appearance was little different from analogous samples after tensile tests. Tensile tests caused some elongation of the grains in the tension direction, and perhaps opened cracks slightly. This rendered the cracks somewhat more detectable, but it is doubtful that it increased their number or extent. Lineal analysis indicated that the structure shown in Figure 39 contained about 7 percent voids.

Micrographs of the large grain material after creep-exposure are presented in Figures 44-53. Cracking at transverse boundaries was noted for almost 23 percent strain in 50 hours at 1000°F (Fig. 45). However, 8-10 percent strain at 1200°F (Fig. 46) did not cause cracking. If the exposure temperature was increased to 1400°F, approximately 6 percent strain caused visible cracking (Fig. 47) and a greater number of cracks were evident for strains of 14-15 percent (Figs. 30-31). Increased cracking occurred for creep at 1600°F (Figs. 48-50) and 1800°F (Figs. 51-53), however, there was little or no evidence of cracking in a sample strained to only 0.6 percent in 100 hours at 1800°F (Fig. 53).

The relative ease of crack detection in the large grain material made it possible to treat the results from a quantitative standpoint. The fraction of cracked boundaries in a group of samples exposed to creep at 1400°, 1600°, or 1800°F was determined by lineal analysis and expressed in Figure 54a as the ratio of the number of cracked boundaries to the total number of boundaries intersected by random straight lines on the plane of polish. This procedure was used in preference to determining the relative area occupied by voids because it eliminated the possible effect of the tensile test in further opening up the cracks. There was a fair correlation (Fig. 54b) between this measure of cracking and the ductility of coarse grained specimens exposed above 1400°F. For the group of specimens studied, the exposure time did not appear to be a significant factor in the fraction of cracked boundaries. As the exposure temperature was increased, a smaller amount of deformation was required to institute cracking.

Examination of 80Ni - 20Cr Alloy for Minor Phases

Inasmuch as the two grain size conditions of the 80Ni - 20Cr alloy were produced by differing heat treatments, the possibility existed that the properties were affected by some other factor than merely the grain size and its means of production. In addition, the studies leading to the establishment of the heat treatments indicated that a slight amount of aging occurred when the as-rolled material was heated at 1550°F, and electron micrographs (Figs. 8 and 9) showed an increase in the number of discrete particles at the grain boundaries as the annealing time was increased.

Electron micrographs of representative specimens before or after creep-exposure are presented in Figures 55-62. The apparent structure in the matrix of these specimens is due to etching effects which are sensitive to grain orientation. In the as-treated small grain condition (Fig. 55), discrete particles of a minor phase occurred at isolated portions of the grain boundaries. In the large grain condition (Fig. 60), although there is an isolated particle within the grain, the principal occurrence of minor phases is a fine, apparently continuous network at the boundaries. The matrix lattice parameters of these samples were determined by analysis of the x-ray diffraction pattern from filings (Fig. 63 and Table 5). A fair degree of scatter was present in the data, however, it appears that the lattice parameter of the large grain material was about 0.004 Å larger than the small grain condition. This suggests that solutioning may have occurred at the 2100°F annealing temperature.

Creep-exposure of the small grain material (Figs. 56-58) resulted in a considerable decrease in the number of discrete particles and an increase in the size of those remaining. Exposure of the large grain material (Figs. 60-62) caused an increased number of discrete particles to appear within the grains and a dissolution of the continuous boundary phase. For samples of either material exposed at intermediate temperatures (Figs. 56, 60, 61), the structures were fairly similar.

The results of x-ray diffraction analysis of the residues from bromine-alcohol extractions of selected samples are summarized in Table 6. The minor phases in both as-treated conditions were identified as a complex chromium-iron carbide and a complex manganese-iron oxide. (The chemical analysis of this material (page 2) indicated the presence of appreciable amounts of both iron and manganese.) The pattern for the carbide was somewhat weaker in the large grain sample. This further suggests that the increased matrix lattice parameter could have followed solution of the carbide. Calculations of the effects of solutioning on the matrix lattice parameter, however, indicated that the difference between the case where all the carbon was in the form of the complex carbide and the case where the complex carbide was completely in solution could not completely account for the apparent measured difference between the large grain and small grain conditions. This suggests that either there was some other solutioning effect or that the measured difference of 0.004 Å in the lattice parameter was in

error. A further possibility could have been segregation of chromium so that an over-all difference in chromium level could have contributed to the parameter difference. For nickel-chromium solid solutions in this range of chromium content, a difference of 1 atomic percent chromium would account for a parameter change of approximately 0.0015 Å.

After creep-exposure at 1400° and 1600°F, the complex carbide and oxide phases were again identified and a possible identification was also made of Cr_2O_3. Only oxide phases were identified in the specimen exposed 484 hours at 1800°F. Several diffraction lines were observed that did not correspond to any of the listed phases.

In view of the relatively slight effects of aging on the short-time mechanical properties after heating at 1550°F and the indication that distribution of the minor phases, although initially different, tended to become similar for comparable conditions of exposure, it is suggested that the minor phases could have had only slight effects on the response of the alloy to creep-exposure.

Correlation of Results

The outstanding feature of this investigation was the remarkable retention of properties in the 80Ni - 20Cr alloy in the presence of extensive microcracking. The cracking had to develop to an amazing extent before there was a noticeable reduction in properties. This is evident in Figure 54b for the correlation between the amount of cracking and the ductility of the coarse grained material. The microstructures of the fine grained material also showed quite large amounts of cracking before there was a substantial reduction in properties. This carried over to impact testing where the changes were nowhere near as extensive as is commonly encountered, particularly when there are thermally-induced structural changes.

It must be recognized that, in general, large amounts of creep strain were required to develop the extensive cracking. Both this and the amount required to affect properties point to a severe limitation on the generality of these results in any application to the effects of cracking from creep on properties of higher-strength alloys. In all the other investigations of this series (Refs. 1-6), internal cracking has not been a factor in the results. It is reasonably certain, however, that if cracking did occur in high-strength alloys, not as much creep would be required to develop damaging cracking. Furthermore, damage would be noticeable at less cracking. Higher-strength alloys would almost certainly be more susceptible to the stress concentration effects of the cracks.

The 80Ni - 20Cr alloy did fulfill the requirement of a material which underwent changes in mechanical properties due primarily to creep itself. For both the fine and coarse grains, the following effects can be identified:

1) At 1000° and 1200°F, the ultimate strengths were raised and the ductility reduced primarily by strain hardening similar to

the results of strain hardening at room temperature. The yield strengths were unquestionably subject to Bauschinger effects due to residual stresses such that the tensile yield strength was raised and the compressive yield strength would have been reduced if it had been measured.

2) The microstructural examination, however, disclosed that the coarse grained material did exhibit microcracking after creep-exposure as low as 1000°F when the exposure time for a large amount of creep was 50 hours. Thus, the reduction in ductility observed for such conditions was, in part, due to cracking.

3) When the exposure temperature was increased above 1200°F, the mechanisms changed in the following manner:
   (a) The amount of increase in strength from strain hardening and Bauschinger effects decreased. Presumably, this was due to simultaneous recovery during creep.
   (b) The tendency to internally crack increased. This reduced ductility, thus offsetting the presumed increase in ductility from relief of strain hardening.
   (c) The recovery process in the fine grained material was so strong that actual grain growth was visible after exposure at 1600° and 1800°F. This was responsible for the reversal of strengths with amount of creep at relatively low temperatures and the decrease to values below those of the coarse grained. Presumably, the recovery process was effective whether or not grain growth was visible. The lesser effect for the longer exposures and the grain growth noted at 1800°F for exposure without stress suggest that the grain growth effect was completed in a relatively short time.
   (d) Exposure at 1800°F resulted in considerable internal oxidation of the fine grained material with a resultant drastic reduction in properties at room temperature. This apparently is strain-time dependent as well as structure sensitive.

4) One of the difficulties in identifying the effects summarized in Item 3 is the inclusion of the effect of the difference in cracking susceptibility of the materials with fine and coarse grains. Cracking first of all reduced ductility. When this was sufficiently severe, the ultimate tensile strength was also reduced. Very little effect of cracking on yield strength was noted. The low ultimate and yield strengths of the fine grained material relative to the original condition after exposure at the higher temperatures was due to the recovery and grain growth. The greater cracking

tendency of the coarse grained material was not associated with reduced yield strengths. It is otherwise nearly impossible to separate from the effects of cracking the reduced effects of strain hardening and residual stresses with increasing temperature and time of creep. The cracking certainly was a factor in the reduced ultimate strengths for the larger strains and longer time periods of exposure, particularly for the coarse grained material. It was a major factor in ductility under the same conditions.

5) Considerable effort was expended attempting to understand the difference in cracking tendencies between the materials with fine and coarse grains. Present theories could be made to both support and refute explanations based on the influence of grain size and its preparation on dislocation movements (Refs. 11-18). Due to lack of time, this feature of the analysis was dropped. It can be observed that the results were usual in the sense that cracking is generally more severe in reducing properties (particularly ductility) in coarse grained material.

6) It was necessary also to leave the possible effects of impurities and minor phases in a rather unsatisfactory state. It is difficult to attribute all the increase in creep resistance for the large grain material to grain size and its method of production alone. Solution effects as well as substructural characteristics seem to be likely candidates in addition. The higher creep resistance would be expected to contribute to increased cracking due to the reduced relaxation of stress concentrations at the points in the grain boundaries where they develop during creep. There seemed to be little on which to base any appreciable effects from the observed minor phases. This could well be in error, however.

7) It should be noted that the effects of thermally-induced structural changes in the 80Ni - 20Cr alloy were minor compared to all other materials used in this series of investigations. In all other materials, particularly Rene' 41 (Refs. 1, 6), the thermally-induced structural changes were the major factor for the temperatures above which strain hardening was a major factor.

Conclusions from Studies of 80Ni - 20Cr Alloy

The following conclusions were derived from the investigation of the effect of creep-exposure of 80Ni - 20Cr alloy on mechanical properties at room temperature:

1) As in the other materials previously investigated, the 80Ni - 20Cr alloy was subject to increased ultimate tensile strength and reduced ductility from strain hardening after creep at the lower temperatures

of exposure. The tensile yield strengths were increased by Bauschinger effects from residual stresses.

2) In this essentially structurally stable material, the only significant evidence of thermally-induced structural change was that the fine grained condition was subject to reduced strength from recovery and grain growth at the higher temperatures.

3) The major effect of creep on properties other than the strain hardening effect was to cause internal microcracking. The amount of cracking required to significantly reduce properties was amazingly large. Presumably, this was due to the low strength and high ductility of the material. Higher-strength alloys subject to internal cracking from creep would show far more pronounced reductions in properties.

4) Internal cracking started to become a factor both as the temperature of creep increased and the time period for a given amount of creep was increased. For large amounts of creep, it was a factor as low as 1000°F. The amount of creep required to develop noticeable cracking decreased as the exposure temperature and time was increased. The cracking mainly reduced ductility, although when severe enough, it reduced ultimate tensile strength as well. For the large grain material, the extent of cracking was quantitatively related to the subsequent tensile ductility. Yield strengths were hardly changed. The onset of cracking overlapped the reduced effects of strain hardening with increasing temperature so that the two effects could not be separated.

5) The coarse grained condition was damaged more by cracking than the fine grained condition. This is the usual behavior for coarse grained materials. The reasons for this were not satisfactorily explained. The creep strength of the coarse grained material was higher than that of the fine grained material. The data suggest that factors over and above the method of producing the large grain size may have been involved. The small amount of impurities and minor phases in the alloy may have had a larger effect than was readily evident.

STUDIES OF THE Mo-0.55Ti-0.064Zr (TZM) ALLOY

A molybdenum-base alloy was to be included in the investigation as an example of the high melting point or refractory alloys expected to be of future importance to the Air Force. The material was to be evaluated both in vacuum in the uncoated condition and in air with one of the more promising coatings applied.

Procurement of the alloy was delayed until vacuum testing equipment was available. This procedure was adopted in order to eliminate the danger of obtaining an expensive material that might become obsolete before testing could be initiated. The tentative test material was to be the binary Mo-0.5Ti alloy, however, by the time that procurement became possible the higher-strength TZM alloy had become available and so was substituted.

Equipment and Procedures for Vacuum Creep-Exposures

The test unit utilized a vacuum chamber over the standard loading system of the creep units at the University of Michigan. The specimen was heated by radiation from a tantalum wire resistance heating element inside the vacuum chamber. Molybdenum radiation shields were used with one shield movable to assist in obtaining proper temperature distribution. The chamber was sealed to the pull rods by specially designed water cooled O-ring seals.

Vacuum was provided by a system including a mechanical pump and a diffusion pump. Thermocouple and ionization gages were used to measure vacuum. Deformation was measured by the movement of the pull rods outside the vacuum chamber.

The pressure could be reduced to 0.03 micron at room temperature with a leak rate of 0.6 micron per minute. The temperature was brought up gradually so that the pressure could be maintained below 1 micron. After one hour at 1800°F, a pressure of 0.1-0.2 micron was maintained with a leak rate of 0.8 micron per minute. After about 10 hours at 1800°F, the pressure dropped to 0.05 micron.

The general procedure in starting a test was to load the unit in the afternoon and pump it down cold overnight. The heating element was turned on the next morning and the specimen brought up to temperature in several steps which took most of the morning. When the test temperature was reached, the specimen was adjusted to the position giving proper temperature distribution and the load was then applied.

Temperature measurement was by means of three chromel-alumel thermocouples attached to the gage section. Several methods of thermocouple attachment were investigated with the most satisfactory results obtained with 28-gage couples percussion welded directly to the reduced gage section. In the course of another investigation conducted in the vacuum creep unit, several chromel-alumel couples were checked after being used for 135 hours at 1800°F and found to be less than 8°F off calibration. For the time being, it was decided to use the relatively cheap chromel-alumel couples in preference to noble-metal couples in short-time tests. By continued checking, it was intended to establish maximum time and temperature limits for the utilization of chromel-alumel. The temperature difference observed over the gage section at 1800°F was ± 3°F.

The original intention was to use one-inch long, 0.250-inch diameter-gage section threaded-end specimens. The first tensile specimen fractured in the threads and the gage sections of later specimens were machined down to 0.187-inch diameter (Fig. 64). Although the revised specimens proved satisfactory for creep-rupture testing, difficulties were encountered in removing the threaded portions from the holders following the test. Accordingly, the holding system was revised to permit the use of button-head specimens to be gripped at the shoulders. This would also obviate the possibility of premature failure at the threads in tensile tests. Details of the button-head specimen and holders are also shown in Figure 64. They were used successfully for a tensile test.

Tensile, Creep-Rupture and Creep-Exposure Tests of TZM Molybdenum Alloy

Tensile tests at room temperature were run on specimens of the as-received TZM alloy to provide base property information for evaluating the effects of creep-exposure and to confirm that the material had properties representative of the alloy.

Three tensile tests were run, two on threaded-end specimens and one on a button-head specimen. The data are included in Table 7. The first threaded-end specimen failed in the threads rather than in the 0.250-inch diameter gage section. This was the only indication of brittleness encountered until specimens were exposed at 1800°F. The gage section of succeeding specimens were then machined down to 0.187 inch. Comparative tensile test data were available from Reference 19 for a heat of the following nominal composition, 0.46Ti-0.074Zr-0.017C. The 0.2 percent offset yield strength was not reported in Reference 19, consequently, comparisons with the present heat TZ-6001 were made on the basis of the 0.1 percent offset yield strength. Good agreement was obtained between the two sets of data -- particularly with respect to the yield strength and ductilities. This confirmed that the tensile properties of Heat TZ-6001 were apparently representative.

Creep-exposure at 1800°F for 10 hours at 60,000 psi resulted in about 2-percent creep (Table 7), while 65,000 psi resulted in about 4 percent. Rupture occurred in 2.2 hours at 70,000 psi and in 5.4 hours at 67,500 psi. These data, together with a test at 57,500 psi which ruptured in 114.5 hours, were used to establish the stress-rupture curve of Figure 65. The indicated stress for rupture in 100 hours of about 57,500 psi was considerably below the 70,000 psi indicated for a similar alloy in Reference 19.

The creep curves are included in Figure 66 and the stress-time values for 2 and 4 percent of creep have been added to Figure 65.

At room temperature, the yield strength was increased slightly and the ultimate strength was reduced slightly (Table 7 and Fig. 67) by exposure to about 2 and 4 percent of creep in 10 hours at 1800°F. The ductility was reduced

severely in the specimen exposed to 4-percent creep. Extremely brittle behavior was encountered when attempts were made to remove the fractured threaded-end specimen from adapters after rupture tests. A very light torque caused them to shatter.

Further progress was blocked for several reasons. Difficulties were encountered with the creep-unit heating elements. In order to avoid problems in removing threaded-end specimens from holders after creep-exposure, it was deemed necessary to develop the button-head method of attachment. Foremost, however, was the uncertainty of the suitability of the material or the exposure conditions in view of the low apparent ductility after exposure and the low rupture strength. Attempts to resolve these problems were not successful. The uncertainty regarding the material was judged as reason to not undertake the expense of having specimens coated.

Metallographic Examination of TZM Alloy

Optical micrographs at 500x of the microstructure of the TZM alloy before and after creep-exposure are presented in Figures 68 through 71. The as-received stress-relieved material (Figs. 68 and 69) had an essentially single phase structure with a considerable amount of substructure. There are also indications of small amounts of minor phases. Creep-exposure for 10 hours or creep-rupture after 114.5 hours at 1800°F (Figs. 70 and 71) had little, if any, discernable effect on the microstructure in the interior of the specimens.

The fractured areas of specimens M-3 and M-4 which were tensile tested at room temperature after 10 hours prior creep at 1800°F are shown in composite pictures in Figures 72a and 73a. Specimen M-3 had 24-percent elongation, while specimen M-4 had only 2-percent elongation. Of particular interest are transverse cracks occurring at approximately 60°-90° to the tension axis of the specimens. Specimen M-4 fractured at two places -- one of which was parallel to the transverse cracks. A schematic drawing of the cracking (Fig. 72a) was made from the actual sample to supplement the composite (and re-photographed) micrograph of Figure 72a. These cracks apparently represent planes of weakness rather than shear cracks since there appeared to be no translation of the structure along the crack direction. Figure 72a shows a longitudinal crack almost along the center line of specimen M-3. A similar crack was observed in the tensile fracture of specimen M-2, the as-received condition.

Figures 72b and 72c also show that recrystallization occurred in a narrow zone at the surface of specimen M-3. Indications of similar recrystallization in specimen M-4 were slight. The transverse cracks shown in Figure 72b may have originated as intercrystalline cracks propagated inwards from the recrystallized skin. The available data do not indicate if the surface effect was a true recrystallization or a manifestation of surface reactions. Other than the possibility that surface changes were responsible for embrittlement, no other reason was found.

Conclusions from Tests of TZM Molybdenum Alloy

The results were too limited for any definite conclusion as to the effect of creep-exposure in vacuum on mechanical properties at room temperature. The loss in ductility could have been due to the particular lot of material or to exposure conditions not being suitable. Experimental difficulties precluded resolution of the questions.

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Table 1

TENSILE TEST DATA FOR 80 Ni-20Cr ALLOY

Heat Treatment	Test Temp (°F)	Ultimate Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (%)	Reduction of Area (%)	Modulus, E x10^6 (psi)
As Hot Rolled	Room	116,000	68,000	30.4	59.5	29.7
	Room	116,100	70,000	30.0	61.0	32.0
	Avg.	116,050	69,000	30.2	60.2	30.8
Annealed - 1550°F 1 hr.	Room	112,800	63,400	27.8	60.3	29.7
	Room	112,800	63,400	30.2	61.5	30.8
	Avg.	112,800	63,400	29.0	60.9	30.2
Annealed - 1550°F 4hr. -(coded "C1")	Room	112,800	59,000	35.8	60.0	29.4
	Room	111,200	60,200	30.8	60.2	29.6
	Room	111,100	59,600	29.6	60.0	29.4
	Room	113,000	57,600	36.2	61.3	33.4
	Room	111,400	58,400	35.0	61.1	29.6
	Avg.	111,900	58,960	33.5	60.5	30.3
Annealed - 1550°F 24 hrs.	Room	111,000	57,100	30.6	58.9	30.3
	Room	110,900	57,000	30.0	60.9	30.0
	Avg.	110,950	57,050	30.3	59.9	30.2
Annealed - 2100°F 1 hr. -(coded "C2")	Room	96,900	32,400	50.0	67.7	30.6
	Room	97,200	32,200	47.6	67.4	30.2
	Room	97,500	33,000	46.6	67.4	29.7
	Avg.	97,200	32,533	48.1	67.5	30.2

Table 2

RUPTURE TEST DATA FOR 80 Ni-20Cr ALLOY

Spec. No.	Test Temp (°F)	Stress (psi)	Rupture Time (hrs)	Elongation (%)	Reduction of Area(%)	Parameter *
			Annealed 1550°F - 4 hrs.			
C1-6	1000	80,000	0.5	33.6	38.0	28.8
C1-9	1000	55,000	54.3	28.0	36.5	31.7
C1-2	1200	35,000	5.4	46.5	46.0	34.5
C1-10	1200	20,000	63 ± 5	57.5	44.8	36.2
C1-3	1400	15,000	3.1	35.8	34.6	38.2
C1-45	1400	6,000	84.4	35.1	28.4	40.8
C1-7	1400	5,000	150.5	28.5	24.4	41.3
C1-4	1600	5,000	4.9	23.6	21.6	42.7
C1-54	1600	2,250	92.1	49.5	26.0	45.4
C1-8	1600	2,000	216.1	68.0	29.0	46.1
C1-5	1800	3,500	3.6	13.8	14.2	46.6
C1-56	1800	3,200	5.0	10.9	9.5	46.8
C1-60	1800	2,900	3.4	13.2	17.5	46.5
C1-25	1800	2,500	17.5	6.5	5.6	48.0
C1-64	1800	1,800	17 ± 3	18.4	18.5	48.0
			Annealed 2100°F - 1 hr.			
C2-13	1000	80,000	On Load	51.7	62.0	25.±
C2-33	1000	70,000	18.9	36.2	35.4	31.1
C2-37	1000	68,000	37.4	31.5	28.5	31.5
C2-3	1000	60,500	102.8	21.2	25.2	32.1
C2-2	1200	35,000	14.7	24.2	24.9	35.1
C2-6	1200	22,000	109.9	18.5	20.4	36.6
C2-4	1400	15,000	12.5	22.0	19.0	39.2
C2-7	1400	10,000	54.0	12.5	13.8	40.5
C2-5	1600	8,000	4.7	17.7	14.7	42.7
C2-10	1600	5,000	28.6	10.8	10.2	44.3
C2-35	1600	4,100	66.6	11.6	8.5	45.1
C2-12	1800	3,500	10.1	9.5	11.5	47.6
C2-34	1800	2,500	26.3	9.5	10.7	48.4
C2-39	1800	2,500	29.6	12.5	11.1	48.6

* $P=T(20 + \log t)$; where T= temperature, °R ; t= time, hrs.

Table 3

CREEP EXPOSURE TEST DATA FOR ANNEALED 80 Ni-20Cr ALLOY

C1 CONDITION (SMALL GRAIN)

Spec. No.	Exposure Conditions: Temp (°F)	Time* (hrs)	Stress (psi)	% of Rupture Life (est)	Total Load Def. (%)	Plastic Load Def. (%)	Creep Def. (%)	Total Plastic Def. (%)	Total Def. (%)	Room Temperature Properties After Exposure: Ult. Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (%)	Reduction of Area (%)	Modulus, E x 10^6 (psi)	Ratio: Property After Exposure / Property Before Exposure: UTS_f/UTS_o	$0.2\%\ YS_f/0.2\%\ YS_o$	El_f/El_o	RA_f/RA_i
TENSILE TESTS																		
As Treated (avg)	--	--	--	--	--	--	--	--	--	111,900	58,960	33.5	60.5	30.3	1.00	1.00	1.00	1.00
C1-17	Room	.005 in./in./min.	Prestrained in tensile machine	--	--	--	--	9.9	9.9	125,300	106,000	21.5	55.4	31.3	1.12	1.81	0.64	0.91
		.005 in./in./min.	Prestrained in tensile machine					19.3	19.3	144,000	139,000	13.6	51.5	29.3	1.29	2.37	0.40	0.85
C1-11	1000	5.0	64,000	48	4.17	3.95	6.23	10.18	10.40	126,000	99,000	21.8	56.5	29.8	1.13	1.69	0.65	0.93
C1-33	1000	5.0	55,000	9	1.95	1.77	0.75	2.52	2.70	117,300	71,400	30.6	58.8	29.2	1.05	1.22	0.92	0.97
C1-21	1000	50.1	52,000	50	1.55	1.36	8.55	9.91	10.10	126,100	93,800	24.4	58.2	31.3	1.13	1.60	0.73	0.96
C1-34	1000	50.0	47,000	19	1.35	1.15	4.20	5.35	5.55	120,800	83,600	25.4	58.2	31.2	1.08	1.42	0.76	0.96
C1-43	1000	100.0	49,000	59	2.20	2.08	33.40	35.48	35.60	135,000	113,000	14.4	47.5	31.4	1.21	1.92	0.43	0.79
C1-38	1000	100.0	44,000	25	0.25	Nil	4.62	4.62	4.87	120,000	70,600	24.3	58.0	32.8	1.07	1.20	0.73	0.96
C1-12	1200	5.0	31,000	48	0.16	Nil	11.28	11.28	11.44	121,000	81,000	25.2	55.7	30.8	1.08	1.38	0.75	0.92
C1-27	1200	5.0	27,000	25	0.18	0.04	0.46	0.50	0.64	114,000	65,300	32.1	58.8	30.8	1.02	1.11	0.96	0.97
C1-65	1200	5.0	27,000	25	0.13	Nil	3.45	3.45	3.58	117,000	70,000	31.0	59.2	29.6	1.05	1.19	0.93	0.98
C1-26	1200	50.0	19,000	69	0.09	Nil	11.6 (est)	11.6 (est)	11.69(est)	118,000	74,300	26.4	50.1	34.3	1.06	1.27	0.79	0.83
C1-20	1200	50.2	12,000	23	0.06	Nil	1.41	1.41	1.47	115,200	61,800	33.2	59.9	31.0	1.03	1.05	0.99	0.99
C1-44	1200	100.0	16,000	85	0.08	0.01	12.77	12.78	12.85	115,400	68,000	27.7	46.8	31.2	1.03	1.16	0.83	0.77
C1-40	1200	100.0	14,000	62	0.03	Nil	5.20	5.20	5.23	114,600	69,600	31.6	57.4	31.3	1.02	1.19	0.94	0.95
C1-35	1200	100.0	10,000	33	0.06	Nil	1.32	1.32	1.38	113,800	61,800	33.1	58.2	31.5	1.02	1.05	0.99	0.96
C1-61	1400	5.0	13,250	78	0.10	Nil	21.70	21.70	21.80	107,300	67,600	15.4	26.5	29.5	0.96	1.15	0.46	0.44
C1-13	1400	5.0	12,000	34	0.09	0.01	11.41	11.42	11.51	113,200	63,500	30.0	52.0	30.0	1.01	1.08	0.90	0.86
C1-29	1400	5.0	10,000	23	0.07	Nil	3.45	3.45	3.52	114,200	61,500	33.9	57.8	31.1	1.02	1.05	1.01	0.96
C1-30	1400	50.0	6,500	62	0.04	Nil	8.02	8.02	8.06	109,600	58,100	29.8	50.4	30.5	0.98	0.99	0.89	0.83
C1-18	1400	50.0	4,800	35	0.05	Nil	2.91	2.91	2.96	112,800	59,000	34.3	58.8	30.8	1.01	1.00	1.03	0.97
C1-52	1400	100.0	5,500	85	0.03	0.01	14.47	14.48	14.50	106,300	57,000	24.0	30.0	31.5	0.94	0.97	0.72	0.50
C1-41	1400	100.0	5,000	69	0.04	Nil	5.75	5.75	5.79	113,000	58,800	32.9	55.0	29.6	1.01	1.00	0.98	0.91
C1-36	1400	100.0	4,000	48	0.06	Nil	3.04	3.04	3.10	115,000	60,100	32.6	58.7	29.8	1.03	1.02	0.97	0.97
C1-32	1600	5.0	4,700	62	0.05	Nil	9.42	9.42	9.47	109,200	57,000	30.9	43.8	29.8	0.98	0.97	0.92	0.72
C1-14	1600	5.0	4,300	33	0.05	Nil	6.00	6.00	6.05	105,800	51,700	27.4	38.6	32.1	0.95	0.88	0.82	0.64
C1-31	1600	50.1	2,500	48	0.03	0.01	40.9 (est)	40.9 (est)	40.9 (est)	25,800	25,000	0.6	0.8	30.0	0.23	0.42	0.02	0.01
C1-53	1600	50.0	2,000	25	0.02	Nil	11.20(est)	11.20(est)	11.22(est)	106,500	52,300	28.9	34.0	30.4	0.94	0.89	0.86	0.56
C1-19	1600	50.2	1,500	17	0.03	0.01	0.28	0.29	0.31	112,200	57,600	37.1	61.1	30.5	1.01	0.98	1.11	1.01
C1-46	1600	100.0	2,000	50	0.02	Nil	11.23	11.23	11.25	105,600	51,300	28.0	33.9	30.2	0.94	0.87	0.84	0.56
C1-37	1600	100.0	1,500	33	0.04	Nil	1.08	1.08	1.12	112,900	58,200	34.0	58.1	30.3	1.01	0.99	0.96	0.96
C1-57	1600	100.0	None	Nil	--	--	--	--	--	113,200	60,200	33.8	58.6	29.2	1.01	1.02	0.95	0.97
C1-42	1800	5.0	3,200	85	0.02	Nil	2.00	2.00	2.02	79,200	35,500	16.5	18.9	33.5	0.71	0.60	0.49	0.31
C1-22	1800	5.0	2,900	50	0.04	Nil	2.46	2.46	2.50	85,600	35,200	22.5	24.7	30.6	0.77	0.60	0.67	0.41
C1-28	1800	50.0	1,900	16	0.04	Nil	9.94	9.94	9.98	9,850	--	Nil	Nil	30.0(est)	0.09	--	Nil	Nil
C1-66	1800	50.0	1,700	5	0.01	Nil	6.5 (est)	6.5 (est)	6.51(est)	10,900	10,900	0.8	1.3	30.0	0.10	0.19	0.02	0.02
C1-55	1800	100.0	1,750	11	0.01	Nil	8.0 (est)	8.0 (est)	8.0 (est)	13,300	--	Nil	Nil	30.0(est)	0.12	--	Nil	Nil
C1-58	1800	100.0	None	Nil	--	--	--	--	--	101,200	37,400	42.9	63.8	30.4	0.91	0.64	1.28	1.06
C1-15	1800	483.8	1,500	19	0.02	0.01	9.82	9.83	9.84	13,000	--	Nil	Nil	30.0(est)	0.12	--	Nil	Nil
IMPACT TESTS										Impact Strength (ft. lb.)	Lateral Expansion (%)	Lateral Contraction (%)						
As Treated (avg. of 2)		--	--	--	--	--	--	--	--	11.5	11.7	15.7						
C1-48	1000	5.0	67,000	85	6.63	6.18	12.82	19.00	19.45	6.0	8.9	5.7						
C1-23	1000	5.0	64,000	48	5.45	5.31	4.55	9.86	10.00	9.5	9.0	8.6						
C1-47	1000	5.0	44,000	25	0.18	Nil	0.05	0.05	0.23	11.0	13.1	12.7						
C1-50	1400	5.0	13,250	78	0.08	0.01	24.62	24.63	24.70	2.0	4.5	3.1						
C1-24	1400	5.0	12,000	34	0.08	0.02	9.32	9.34	9.40	7.0	8.5	7.6						
C1-49	1400	5.0	8,000	11	0.06	Nil	1.96	1.96	2.02	11.0	15.2	11.2						
C1-63	1600	5.0	4,700	62	0.05	0.01	8.80	8.81	8.85	7.0	9.0	6.1						
C1-62	1600	5.0	4,300	33	0.03	Nil	7.87	7.87	7.90	4.0	8.6	3.5						
EXPOSED TO CREEP AND SECTIONED FOR METALLOGRAPHIC EXAMINATION																		
C1-59	1600	5.0	4,700	62	0.07	0.03	12.93	12.96	13.00	--	--	--						

* Plus 4 hr pre-heat before load application.

Table 4

CREEP EXPOSURE TEST DATA FOR ANNEALED 80 Ni-20Cr ALLOY

C2 CONDITION (LARGE GRAIN)

Spec. No.	Exposure Conditions: Temp (°F)	Time* (hrs)	Stress (psi)	% of Rupture Life (est)	Total Load Def. (%)	Plastic Load Def. (%)	Creep Def. (%)	Total Plastic Def. (%)	Total Def. (%)	Room Temperature Properties After Exposure: Ult. Tensile Strength (psi)	0.2% Offset Yield Strength (psi)	Elongation (%)	Reduction of Area (%)	Modulus, E x 10^6 (psi)	Ratio: Property After Exposure / Property Before Exposure: UTS_f/UTS_o	$0.2\%\ YS_f/0.2\%\ YS_o$	El_f/El_o	RA_f/RA_o
TENSILE TESTS																		
As Treated (avg)	--	--	--	--	--	--	--	--	--	97,200	32,533	48.1	67.5	30.2	1.00	1.00	1.00	1.00
C2-24	Room	.005 in./in./min.	Prestrained in tensile machine	--			--	29.8	--	134,300	130,000	13.2	54.7	30.1	1.38	4.00	0.27	0.81
C2-23	Room	.005 in./in./min.	Prestrained in tensile machine	--			--	9.9	--	112,000	76,500	34.1	64.1	31.4	1.15	2.35	0.72	0.95
C2-25	Room	.005 in./in./min.	Prestrained in tensile machine	--			--	4.8	--	103,500	56,300	40.8	66.0	29.9	1.07	1.73	0.85	0.98
C2-42	1000	5.0	65,000	10	29.80	28.00	1.02	29.02	30.82	124,000	101,000	22.0	57.1	32.1	1.28	3.11	0.46	0.85
C2-46	1000	5.0	53,000	3	16.10	13.50	0.53	14.13	16.63	118,000	87,000	28.7	61.5	32.0	1.22	2.68	0.60	0.91
C2-47	1000	50.0	64,000	82	20.00	17.80	4.90	22.70	24.90	124,600	106,200	15.9	29.6	31.0	1.28	3.26	0.33	0.44
C2-41	1000	100.0	58,000	66	18.75	15.50	2.93	18.43	21.68	121,000	92,800	22.6	43.6	32.5	1.25	2.86	0.41	0.65
C2-64	1000	116.0	45,000	12	7.03	6.88	0.35	7.23	7.38	109,300	65,500	35.6	62.0	32.4	1.13	2.01	0.74	0.92
C2-52	1200	5.0	38,000	53	5.96	5.76	2.14	7.90	8.10	107,800	63,800	35.4	59.5	29.8	1.11	1.96	0.73	0.88
C2-9	1200	5.0	35,000	34	3.53	3.30	5.30	8.60	8.83	110,600	68,000	31.0	55.4	32.5	1.14	2.09	0.64	0.82
C2-21	1200	5.0	28,000	12	1.18	1.72	0.85	2.57	2.74	102,500	51,000	43.1	62.5	30.6	1.05	1.57	0.90	0.93
C2-17	1200	10.0	35,000	69	3.75	3.70	6.36	10.06	10.11	107,500	62,600	30.0	54.6	30.1	1.11	1.92	0.62	0.81
C2-22	1200	50.0	25,000	74	0.80	0.70	3.85	4.55	4.65	104,900	51,600	40.5	58.3	30.7	1.08	1.59	0.84	0.86
C2-14	1200	50.0	22,000	47	0.51	0.43	1.90	2.33	2.41	100,000	48,000	41.7	59.7	33.1	1.03	1.48	0.87	0.88
C2-36	1200	100.1	21,000	81	0.10	0.02	4.85	4.87	4.95	104,000	51,300	33.7	54.5	30.5	1.07	1.58	0.70	0.81
C2-30	1400	5.0	17,000	74	0.10	Nil	11.90	11.90	12.00	102,500	46,600	31.2	45.6	30.3	1.05	1.43	0.65	0.68
C2-8	1400	5.0	15,000	40	0.07	Nil	3.85	3.85	3.92	101,700	46,500	40.2	52.8	30.0	1.05	1.43	0.83	0.78
C2-18	1400	10.0	15,000	80	0.06	Nil	14.14	14.14	14.20	93,000	45,900	22.3	28.2	31.2	0.96	1.41	0.46	0.42
C2-31	1400	10.0	15,000	80	0.11	Nil	14.60	14.60	14.71	88,100	44,300	18.1	23.6	31.1 NR	0.91	1.36	0.38	0.35
C2-38	1400	50.1	9,300	72	0.04	Nil	6.22	6.22	6.26	94,300	40,500	30.0	33.5	30.0	0.97	1.24	0.62	0.50
C2-15	1400	50.0	8,000	50	0.03	Nil	2.69	2.69	2.72	105,200	41,700	35.8	48.9	31.1	1.08	1.28	0.74	0.72
C2-43	1400	100.0	7,500	87	0.04	Nil	3.85	3.85	3.89	97,200	37,600	32.2	41.1	30.6	1.00	1.16	0.67	0.61
C2-32	1400	100.0	6,500	66	0.05	Nil	2.26	2.26	2.31	99,200	34,900	37.9	49.9	30.0	1.02	1.07	0.79	0.74
C2-44	1600	5.0	7,500	78	0.03	Nil	7.53	7.53	7.56	83,000	43,500	19.5	19.8	27.0	0.85	1.34	0.40	0.29
C2-29	1600	5.0	6,500	42	0.06	Nil	4.80	4.80	4.86	99,000	41,000	37.9	43.3	29.6	1.02	1.26	0.79	0.64
C2-19	1600	33.0	3,500	30	0.03	Nil	0.85	0.88	0.91	97,000	31,000	38.0	51.5	28.4	1.00	0.95	0.79	0.76
C2-40	1600	50.2	4,100	76	0.03	0.01	2.74	2.75	2.77	81,500	34,500	18.7	21.7	29.8	0.84	1.06	0.39	0.32
C2-51	1600	100.0	3,400	74	0.02	Nil	1.54	1.54	1.56	96,400	33,100	36.8	44.2	30.8	0.99	0.99	0.76	0.66
C2-45	1600	117.0	3,100	79	<0.01	Nil	1.52	1.52	1.52	98,600	37,600	36.9	41.6	30.6	1.02	1.16	0.76	0.62
C2-57	1600	100.0	None	Nil	--	--	--	--	--	97,500	31,300	42.7	55.0	30.0	1.00	0.96	0.89	0.82
C2-50	1800	5.0	3,900	72	0.04	0.01	4.77	4.78	4.81	94,400	35,800	33.4	29.8	29.8	0.97	1.10	0.69	0.44
C2-20	1800	5.0	3,500	50	0.04	0.01	3.87	3.88	3.91	95,300	34,400	37.9	45.2	29.6	0.98	1.06	0.79	0.67
C2-59	1800	50.0	2,000	?	0.02	Nil	2.02	2.02	2.04	83,400	37,400	40.4	56.4	30.0	0.86	1.15	0.84	0.83
C2-48	1800	50.0	1,500	?	<0.01	Nil	0.20	0.20	0.21	97,500	32,000	43.4	61.9	30.2	1.00	0.98	0.90	0.91
C2-60	1800	100.0	1,500	?	<0.01	Nil	0.63	0.63	0.64	97,000	27,500	45.5	59.8	29.4	1.00	0.84	0.95	0.89
C2-49	1800	117.5	1,000	?	<0.01	Nil	0.13	0.13	0.13	97,200	33,400	46.8	63.5	29.2	1.00	1.03	0.97	0.94
C2-58	1800	100.0	None	Nil	--	--	-0.06	-0.06	-0.06	97,500	31,600	45.6	61.8	30.5	1.00	0.97	0.95	0.92
IMPACT TESTS										Impact Strength ft. lb.	Lateral Expansion (%)	Lateral Contraction (%)						
As Treated (avg. of 2)	--	--	--	--	--	--	--	--	--	10	12.2	13.9						
C2-54	1200	5.0	40,000	74	5.92	5.76	5.13	10.89	11.05	11	14.5	14.2						
C2-26	1200	5.0	35,000	34	3.51	3.38	1.29	4.67	4.80	12	9.5	15.7						
C2-65	1400	5.0	17,500	86	0.09	0.01	12.71	12.72	12.80	9	13.1	12.2						
C2-53	1400	5.0	17,000	74	0.09	Nil	8.95	8.95	9.04	10	11.5	13.7						
C2-27	1400	5.0	15,000	40	0.06	Nil	4.46	4.46	4.52	11	9.0	13.1						
C2-67	1600	5.0	7,500	78	0.05	0.01	7.86	7.87	7.91	3	6.5	3.6						
C2-66	1600	5.0	6,500	42	0.03	Nil	2.74	2.74	2.77	10	12.1	11.7						
EXPOSED TO CREEP AND SECTIONED FOR METALLOGRAPHIC EXAMINATION																		
C2-61	1600	5.0	7,500	78	0.03	0.02	9.24	9.26	9.27	--	--	--						
C2-62	1600	50.0	4,100	76	0.03	0.01	3.80	3.81	3.83	--	--	--						
C2-63	1800	5.0	3,900	72	0.03	Nil	7.63	7.63	7.66	--	--	--						

* plus 4 hour pre-heat prior to load application

< less than

NR - not remachined

Table 5

LATTICE PARAMETER DETERMINATION OF 80 Ni-20 Cr ALLOY

Radiation: Cobalt (unfiltered) (4 hour exposure)

Spec. No.	Line	hkl	$S=h^2+k^2+l^2$	Radiation	θ (Measured)	$\sin^2\theta$	"a" (A°)	"d" (calc)
C1-1	1	111	3	K_β	23.425	0.1580	3.53120	2.248
(As Treated)	2	111	3	K_α	26.00	0.1922	3.53640	2.04
	3	200	4	K_β	27.25	0.2097	3.53930	1.94
	4	200	4	K_α	30.35	0.2553	3.54306	1.768
	5	220	8	K_β	40.313	0.4185	3.54308	1.38
	6	220	8	K_α	45.563	0.5098	3.54583	1.252
	7	311	11	K_β	49.200	0.5730	3.55062	1.181
	8	311	11	K_α	56.887	0.7017	3.54405	1.067
	9	222	12	K_α	61.012	0.7652	3.54467	1.022
							$a_o \cong 3.549$	
C2-1	1	111	3	K_β	23.3250	0.1568	3.544676	2.258
(As Treated)	2	111	3	K_α	25.9125	0.1908	3.549308	2.045
	3	200	4	K_β	27.1625	0.2089	3.546065	1.95
	4	200	4	K_α	30.2875	0.2542	3.550718	1.776
	5	311	11	K_α	56.8125	0.7002	3.54781	1.067
	6	222	12	K_α	60.8875	0.7635	3.54862	1.023
							$a_o \cong 3.545$	

Table 6

X-RAY DIFFRACTION DATA FROM EXTRACTION RESIDUES OF 80 Ni-20 Cr ALLOY
EXTRACTION: BROMINE-METHYL ALCOHOL
DIFFRACTION: COPPER Kα RADIATION (4 HOUR EXPOSURE)

(Data given are "d" values and relative intensity of the line)

Spec. No.	C2-1	C2-18	C2-15	C2-29	C1-1	C1-18	C1-14	C1-15	Standard Patterns of Possible Phases (ASTM Cards)				
Exposure Conditions	As Treated	1400°-10 hr	1400°-50 hr	1600°-5 hr	As Treated	1400°-50 hr	1600°-5 hr	1800°-484 hr	$(MnFe)_2O_3$	Cr_2O_3	$(CrFe)_7C_3$	γ-MnO_2	?*
"d" Range									3.83 60				
3.50-3.99								3.62 ms		3.63 74			
3.20-3.49									3.35 10			3.20 60	
3.00-3.19													
2.80-2.99									2.99 30				
2.60-2.79									2.72 100				
	2.71 vs	2.70 ms	2.70 ms	2.71 s	2.71 s	2.70 s	2.71 s			2.67 100			
	2.63 mw	2.63 mw	2.63 w	2.63 w		2.63 mw+		2.65 ms					x
2.50-2.59									2.51 20				
2.40-2.49								2.47 ms		2.48 96			
		2.425 m		2.42 m-				2.405 m-				2.40 100	x
2.30-2.39	2.34 vs	2.34 ms	2.34 ms	2.34 s	2.345 s	2.34 s+	2.34 s	2.34 m	2.35 40		2.30 40		
2.20-2.29	2.28 m-	2.28 mw	2.29 mw	2.295 mw	2.295 mw	2.28 m-	2.295 m-			2.26 12			
		2.22 mw	2.22 mw	2.22 m	2.22 mw			2.22 m	2.21 20				
2.10-2.19						2.17 mw		2.163 m		2.17 38			
	2.135 mw	2.128 vs	2.13 m	2.13 s+	2.13 ms	2.12 mw	2.12 m	2.115 s	2.11 10		2.12 60	2.11 70	
2.00-2.09		2.045 m	2.045 m-	2.045 m+	2.045 m-	2.045 m	2.045 ms		2.01 40	2.05 9	2.04 100		
1.90-1.99				1.995 vw	1.995 mw				1.92 10				x
1.80-1.89				{1.81 / 1.85} mw-		1.88 vw	1.84 w 1.82 m-		1.873 40				x
		1.81 mw	1.815 w+			1.815 vw		1.81 m		1.82 39	1.81 60		
1.70-1.79				1.755 mw-	1.785 mw	1.75 vw					1.74 60		x
					1.71 w		1.71 mw		1.72 25				
1.60-1.69	1.655 s	1.657 m	1.655 m	1.655 ms 1.64 w+	1.655 ms	1.657 ms	1.66 ms	1.667 ms 1.637 m	1.66 90	1.67 90			
	1.613 mw	1.607 vw	1.613 vw	1.612 w		1.615 mw			1.62 20			1.63 80	
1.50-1.59									1.57 20 1.53 30	1.58 13			
1.40-1.49								1.46 m-	1.48 20				
			1.43 w+				1.437 ms	1.43 m	1.45 30	1.46 25	1.46 30		
	1.413 ms		1.413 m	1.414 ms	1.413 m	1.413 m			1.42 80	1.43 40			
1.30-1.39	1.377 mw	1.396 m		1.376 vw		1.376 mw			1.39 40				x
	1.352 m+	1.352 mw	1.352 mw	1.352 m-	1.352 m	1.352 m	1.352 m		1.36 30		1.35 20		
	1.322 w								1.33 10				
									1.31 20	1.30 20			
1.20-1.29						1.294 w		1.288 m					
		1.264 m	1.264 w	1.263 m	1.264 mw				1.28 30 1.26 20				
						1.253 w							
						1.226 w		1.241 mw		1.24 17			x
		1.21 w		1.21 w	1.21 mw	1.208 w	1.212 mw	1.208 mw		1.21 7	1.205 60		
1.10-1.19						1.188 w	1.187 w		1.19 20				
		1.185 w	1.185 mw	1.185 mw	1.185 mw			1.18 m	1.18 40		1.178 60		
	1.173 m	1.171 mw	1.173 mw	1.171 mw	1.173 m	1.172 mw	1.17 mw						
								1.164 m-	1.16 40	1.15 10	1.16 60		
									1.14 30				
									1.12 30 1.11 30	1.12 10			
1.00-1.09									1.09 30	1.09 17			
								1.084 m 1.058 mw	1.08 70				x
			1.044 mw	1.045 m	1.044 m	1.044 m-		1.038 mw		1.04 16			
<1.00	3 lines		2 lines	5 lines	4 lines	3 lines	2 lines	4 lines	?	?	?	?	
Phases Tentatively Identified	$(MnFe)_2O_3$ $(CrFe)_7C_3$?	$(MnFe)_2O_3$ $(CrFe)_7C_3$ Cr_2O_3 ?	$(MnFe)_2O_3$ $(CrFe)_7C_3$ Cr_2O_3 ?	$(MnFe)_2O_3$ $(CrFe)_7C_3$ Cr_2O_3 ?	$(MnFe)_2O_3$ $(CrFe)_7C_3$	$(MnFe)_2O_3$ $(CrFe)_7C_3$ Cr_2O_3 ?	$(MnFe)_2O_3$ $(CrFe)_7C_3$	$(MnFe)_2O_3$ Cr_2O_3 γ-MnO_2					

* lines not showing reasonable agreement with any of the possible listed phases

Table 7

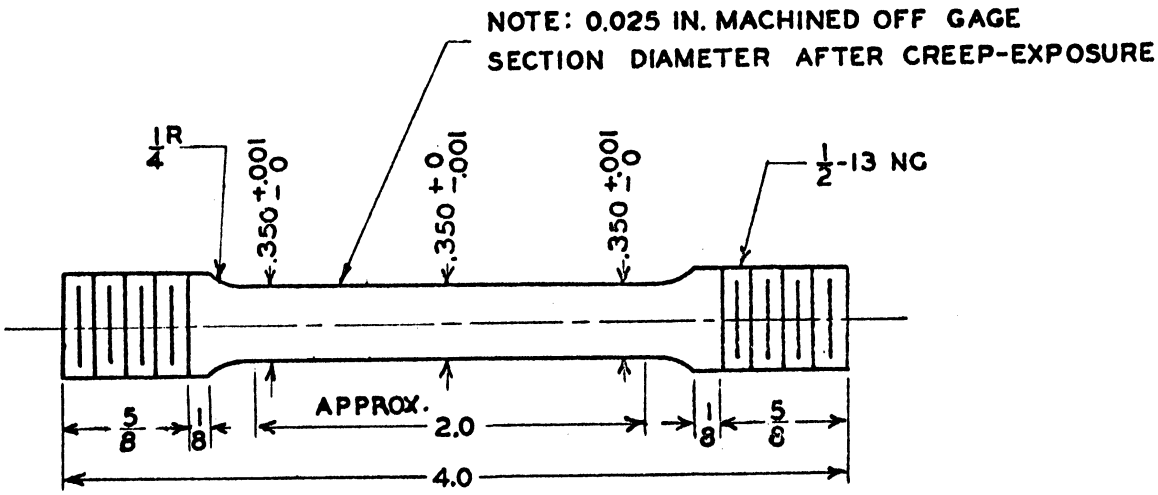
TEST DATA FOR Mo-.55Ti-.064Zr-.017C (TZM) ALLOY

Spec. No.	Exposure Conditions: Temp (°F)	Stress (psi)	Time (hrs)	Est. Load Def. (%)*	Creep Def. (%)	Elongation (%)	Red. of Area (%)	Room Temperature Tensile Properties After Exposure: Ult. Tensile Strength(psi)	.1% Offset Yield Strength(psi)	.2% Offset Yield Strength(psi)	Elongation (%)	Red. of Area (%)	Remarks
M-1	As Received (Stress Relieved)			-	-	-	-	-	99,800	106,000	-	-	Broke in threads
M-2				-	-	-	-	125,000	100,200	105,800	28.0	61.0	
Button-1				-	-	-	-	120,800	98,400	101,300	27.0	69.8	
Data of Ref. 19 - Avg. of 2 tests				-	-	-	-	128,400	100,300	No Report	27.5	60.8	
M-3	1800	60,000	10.0 I	0.24	2.25	-	-	121,800	98,000	112,000	24.0	55.0	
M-4	1800	65,000	10.0 I	0.30	4.26	-	-	112,000	110,500	112,000	2.0	1.1	
M-5	1800	70,000	2.2 R	0.42	-	19	85	-	-	-	-	-	
M-6	1800	67,500	5.4 R	0.35	-	25	90	-	-	-	-	-	
M-7	1800	65,000	8.5 R	0.30	-	15	>90	-	-	-	-	-	
M-8	1800	65,000	10.4 R	0.30	-	24	>90	-	-	-	-	-	
M-9	1800	57,500	114.5 R	0.16	-	19	>90	-	-	-	-	-	

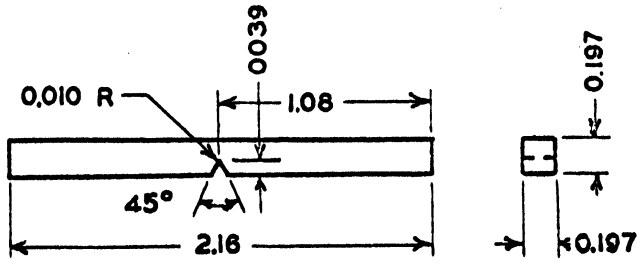
I = interrupted
R = ruptured

M1-9 = threaded end specimens

* Estimated from data of Ref. 20 , page 183-184



TENSILE AND CREEP SPECIMEN



TYPE-W IMPACT SPECIMEN

Note: Center of the Impact Specimen to Coincide with the Center of the Creep Specimen.

Figure 1 Details of Specimens For Tests of 80Ni-20Cr Alloy

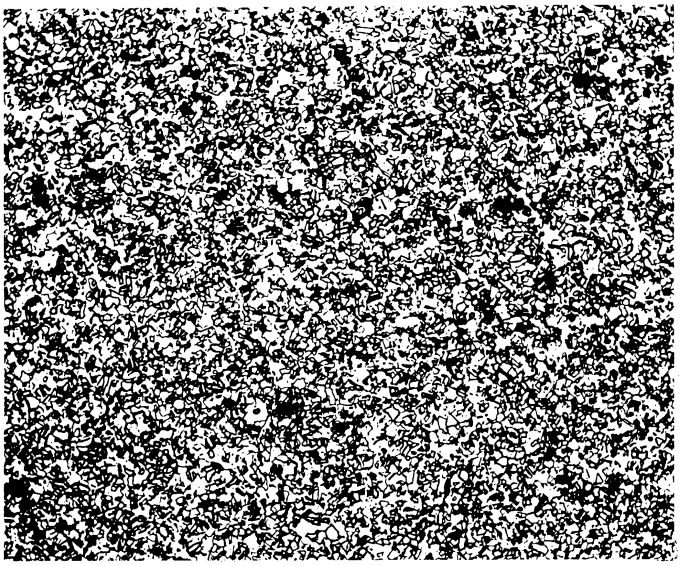


Figure 2 80Ni-20Cr Alloy
As Hot Rolled

100x

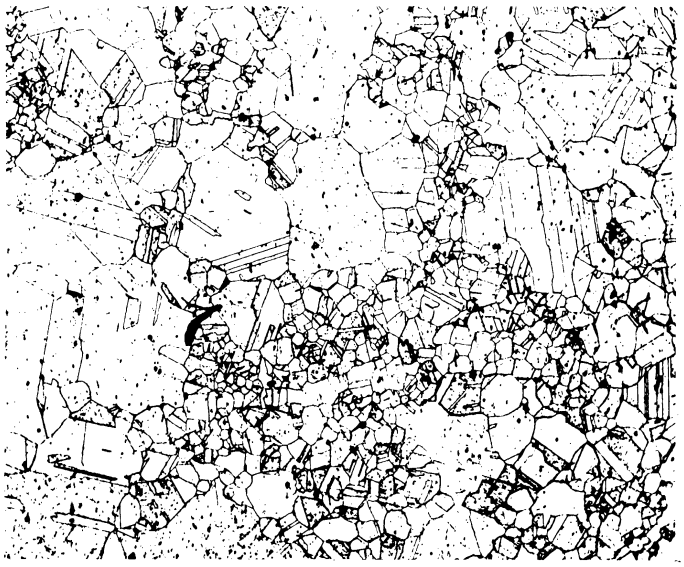


Figure 3 80Ni-20Cr Alloy
2000°F - 1/2 hr + Air Cool

100x

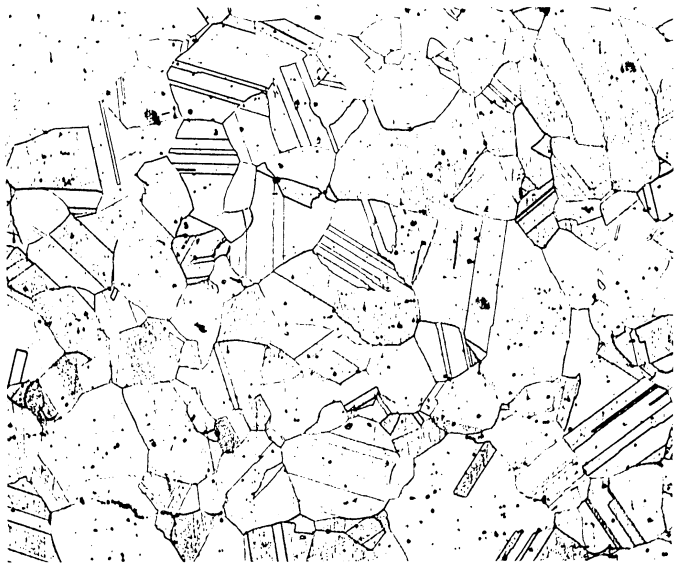


Figure 4 80Ni-20Cr Alloy
2100°F - 1 hr + Air Cool

100x

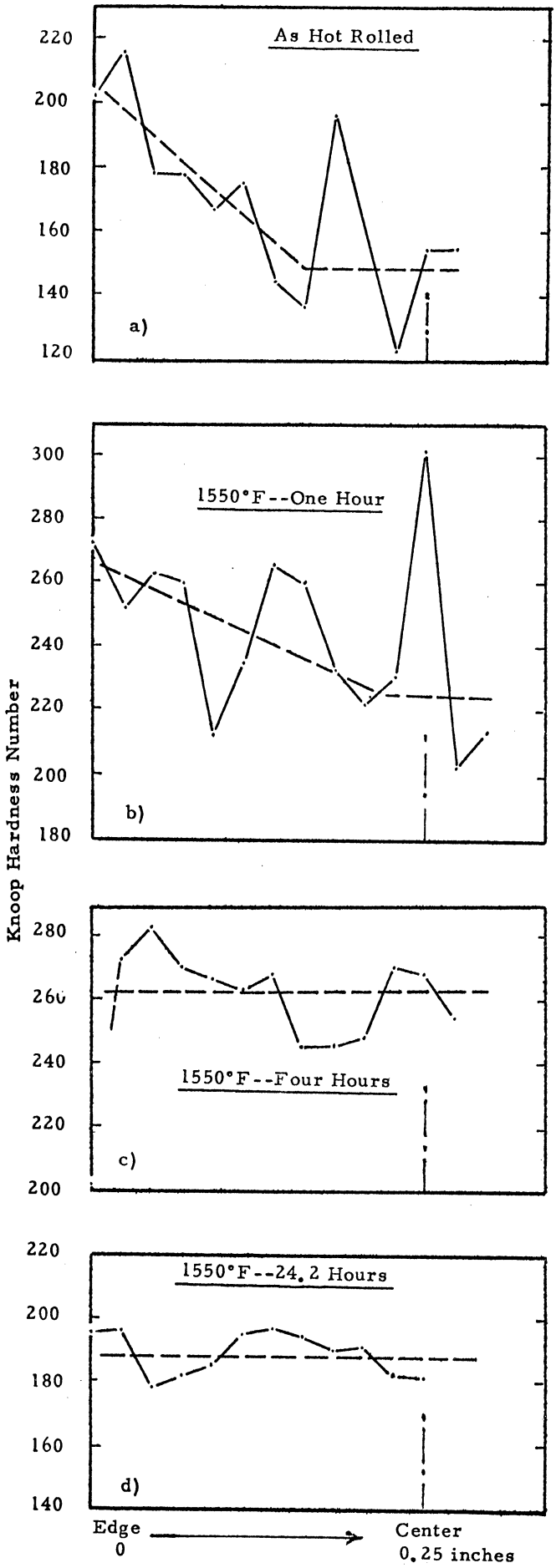


Figure 5 Knoop Micro-Hardness Surveys of Annealed 80Ni-20Cr Alloy

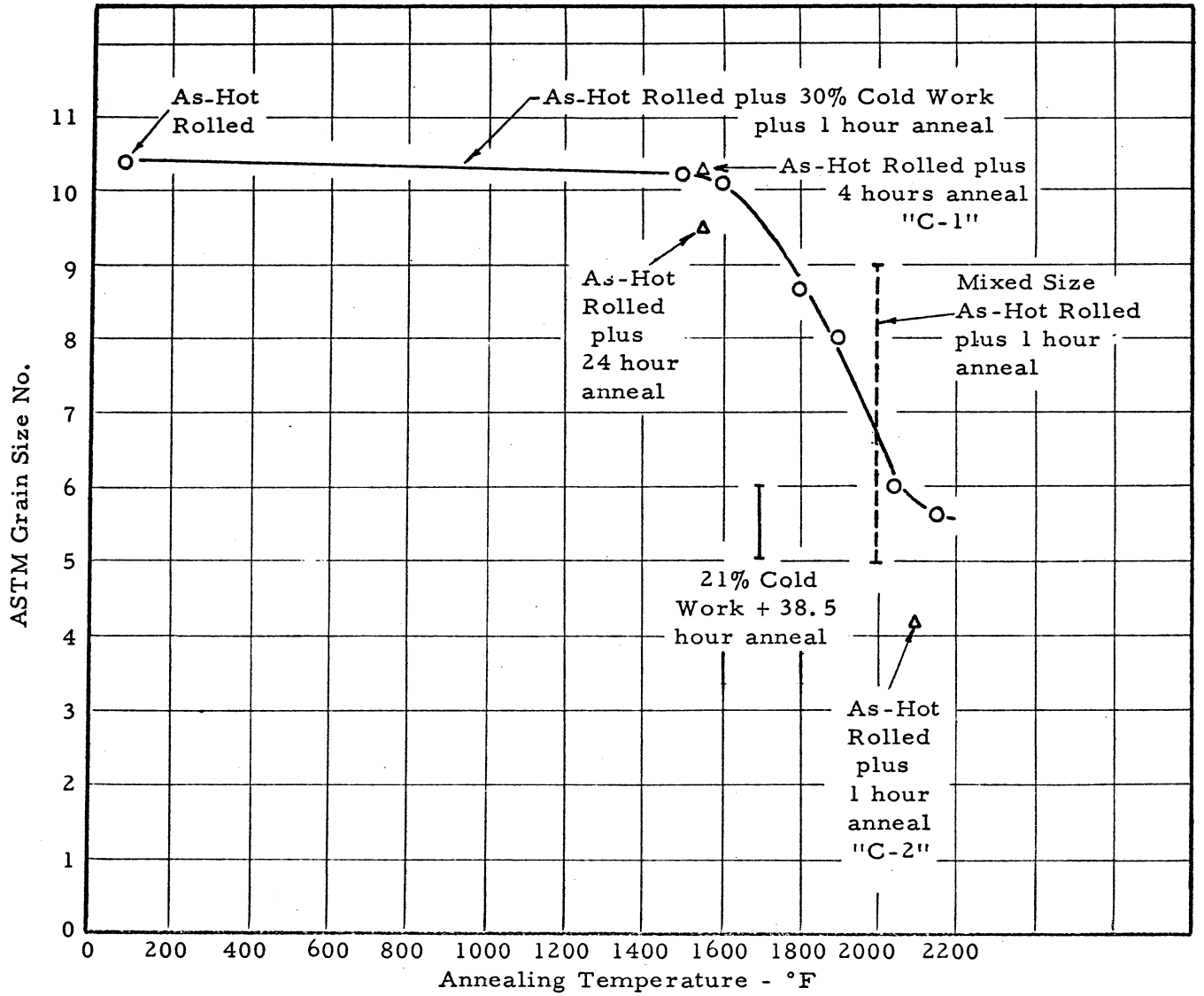


Figure 6 Effect of Annealing Temperature and Prior Cold Work on Grain Size of 80 Ni - 20 Cr Alloy.

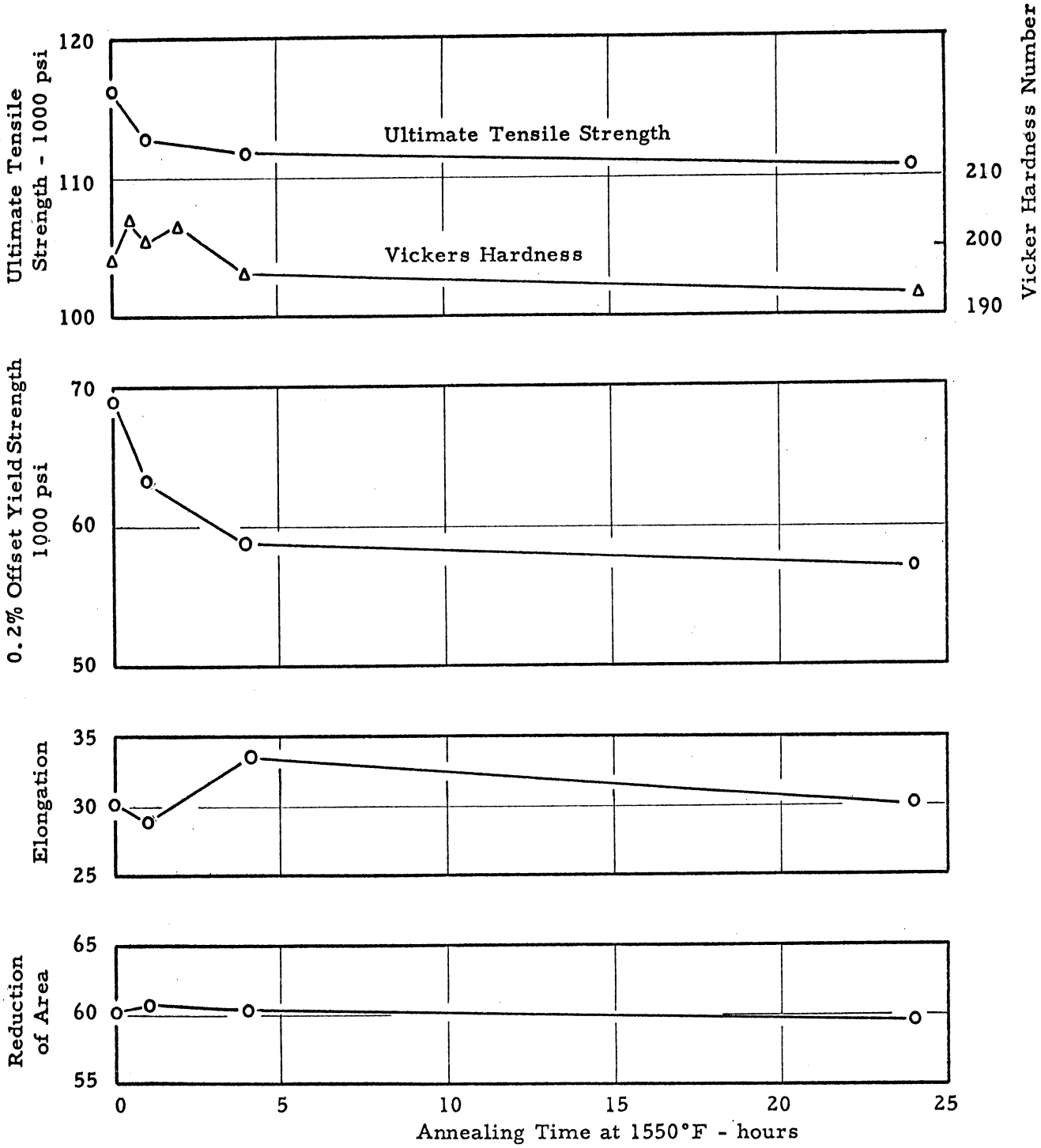


Figure 7 Effect of Annealing Time at 1550°F on Room Temperature Tensile Properties and Hardness of 80Ni-20Cr Alloy

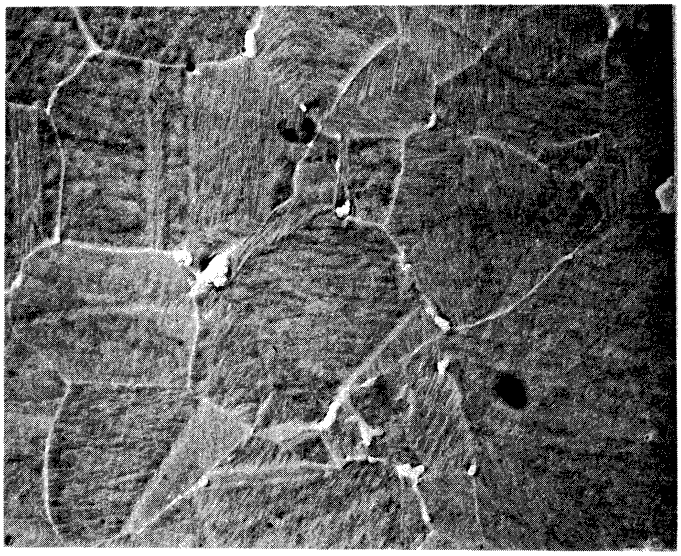


Figure 8 80Ni-20Cr Alloy
As Hot Rolled

3500x

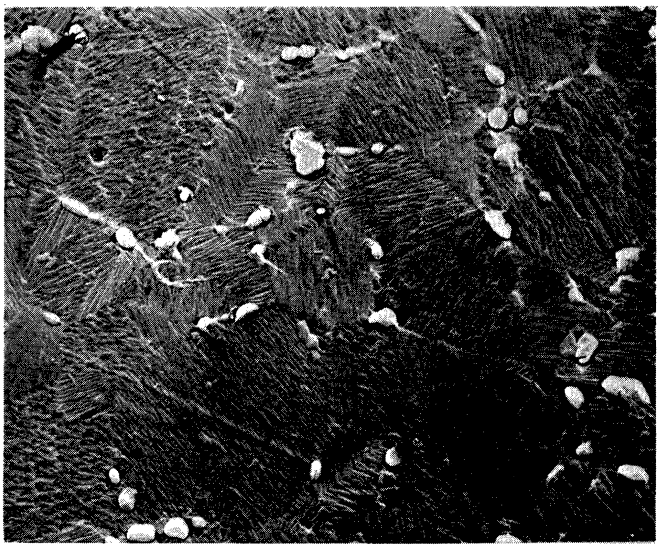


Figure 9 80Ni-20Cr Alloy
1550°F - 4 hrs + Air Cool

3500x

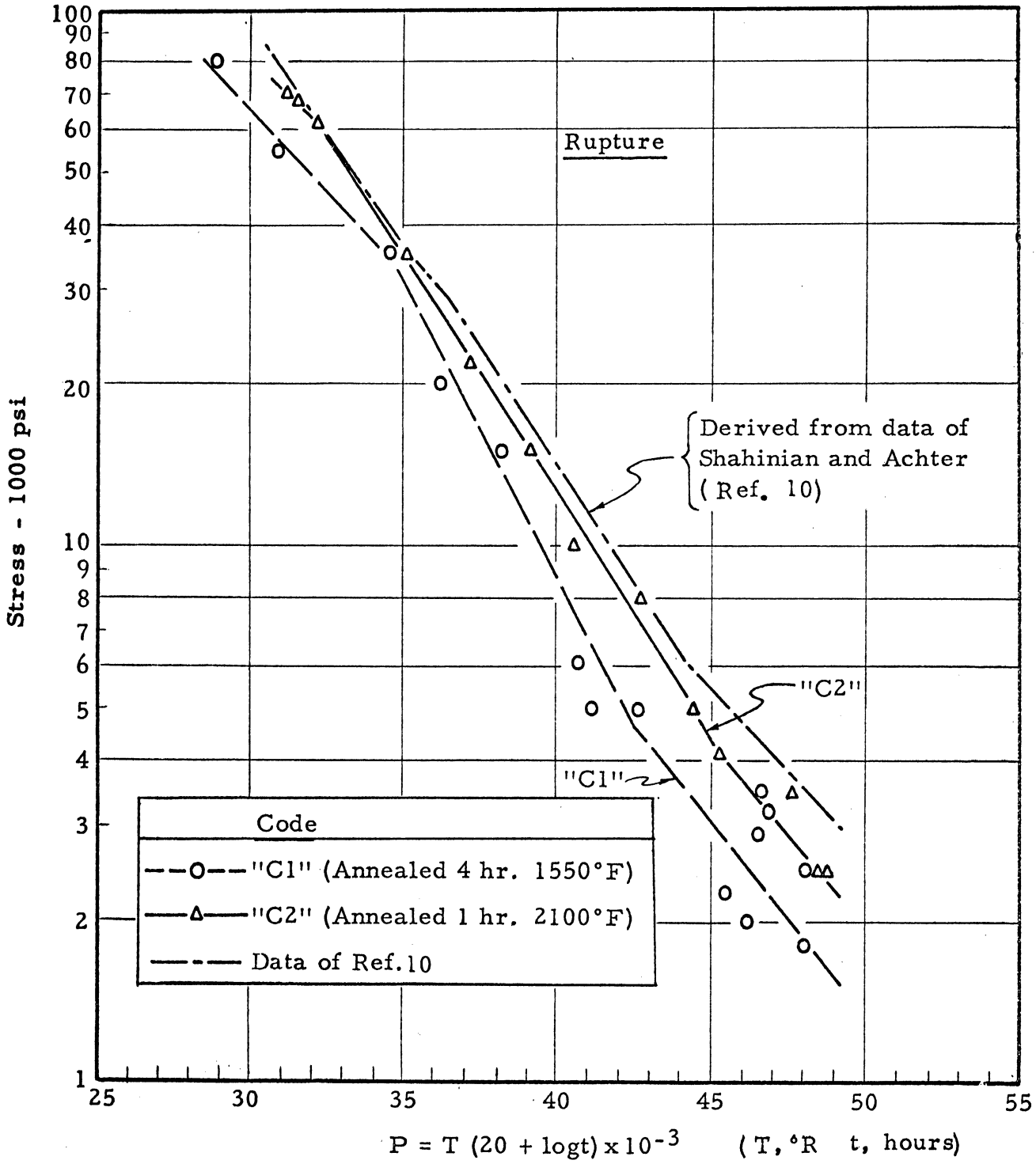


Figure 10 Master Rupture Curve for Annealed 80 Ni-20Cr Alloy

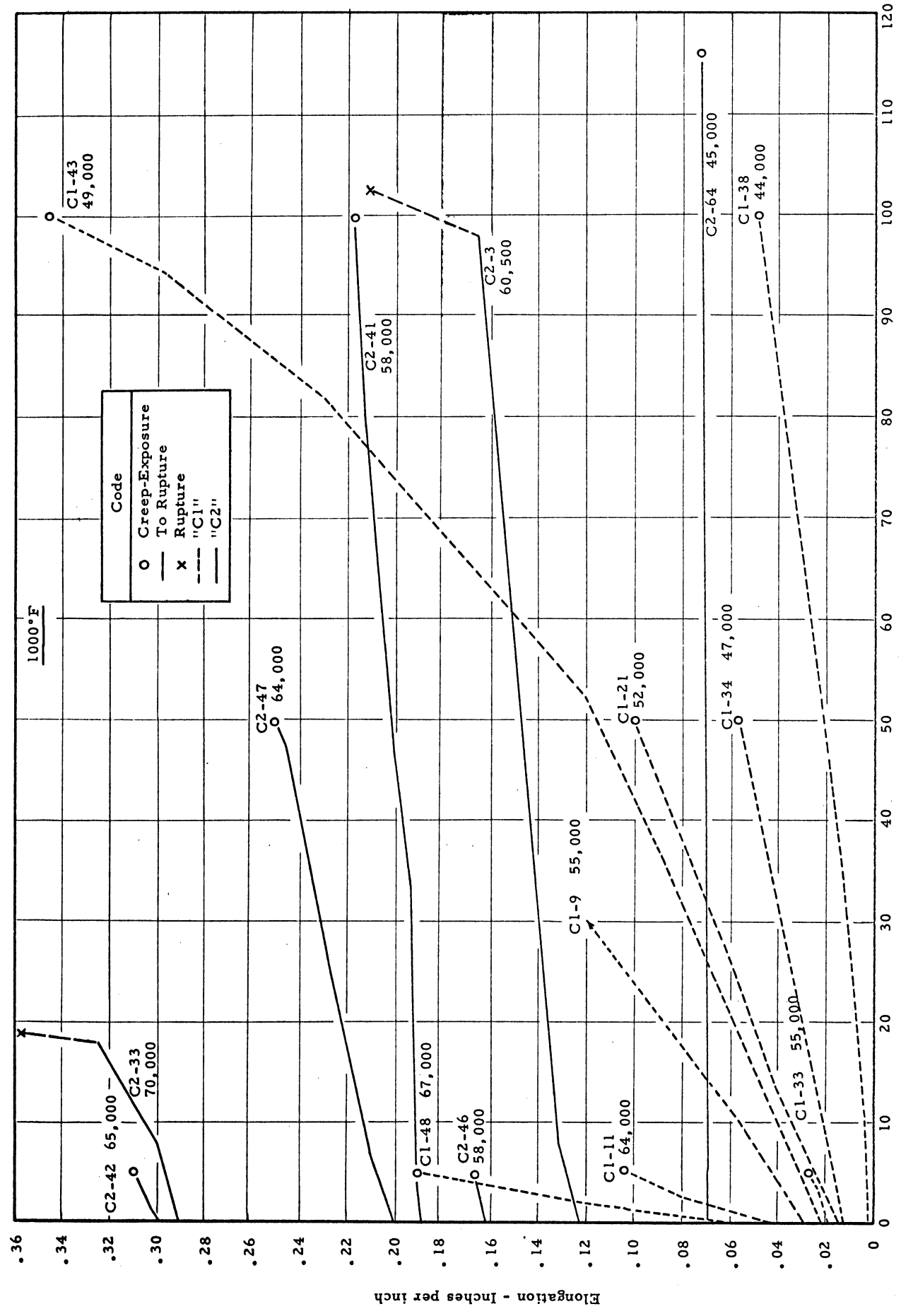


Figure 11 Time-Elongation Curves for 80Ni-20Cr at 1000°F

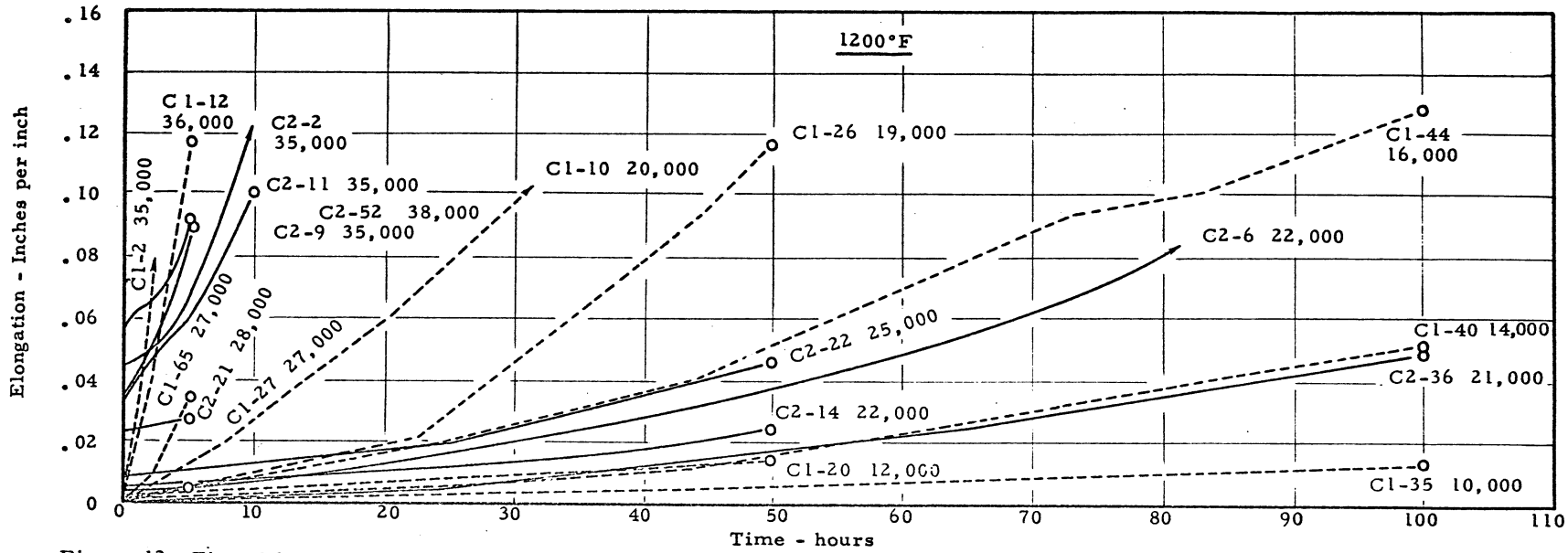


Figure 12 Time-Elongation Curves for 80Ni-20Cr at 1200°F

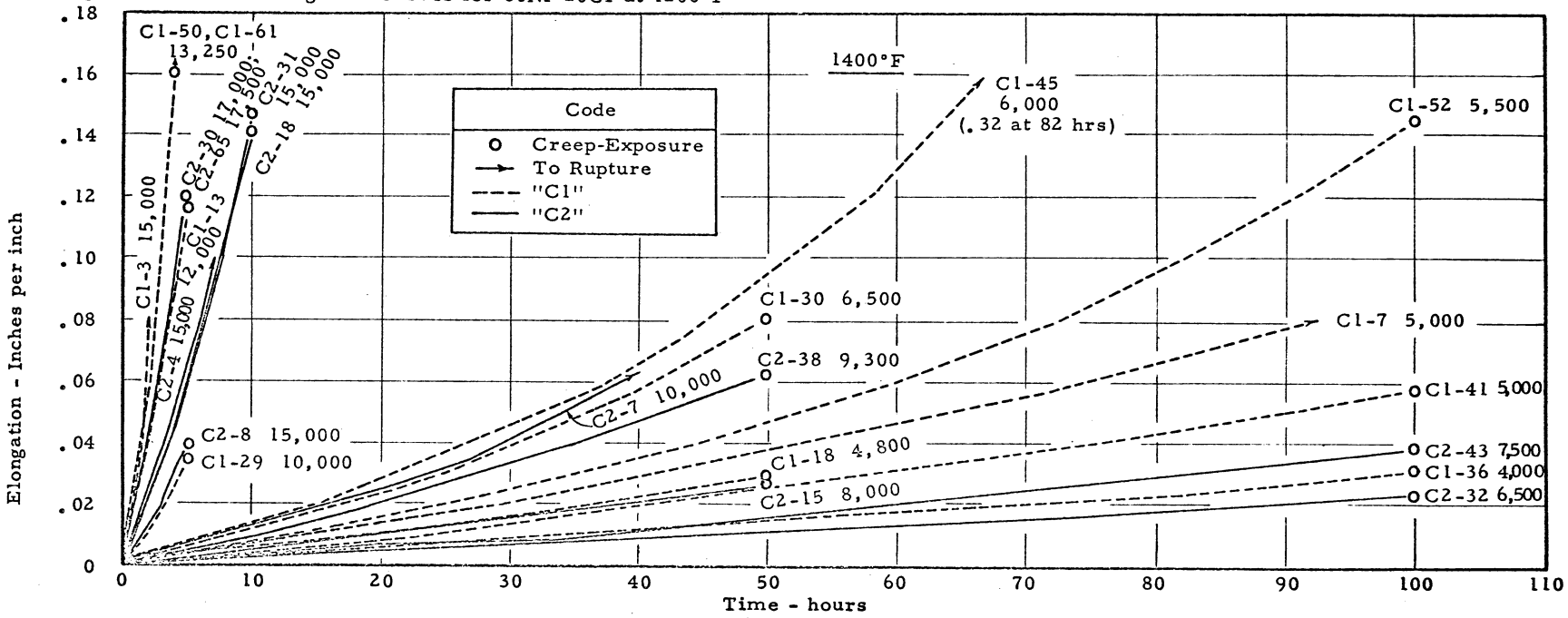


Figure 13 Time-Elongation Curves for 80Ni-20Cr at 14

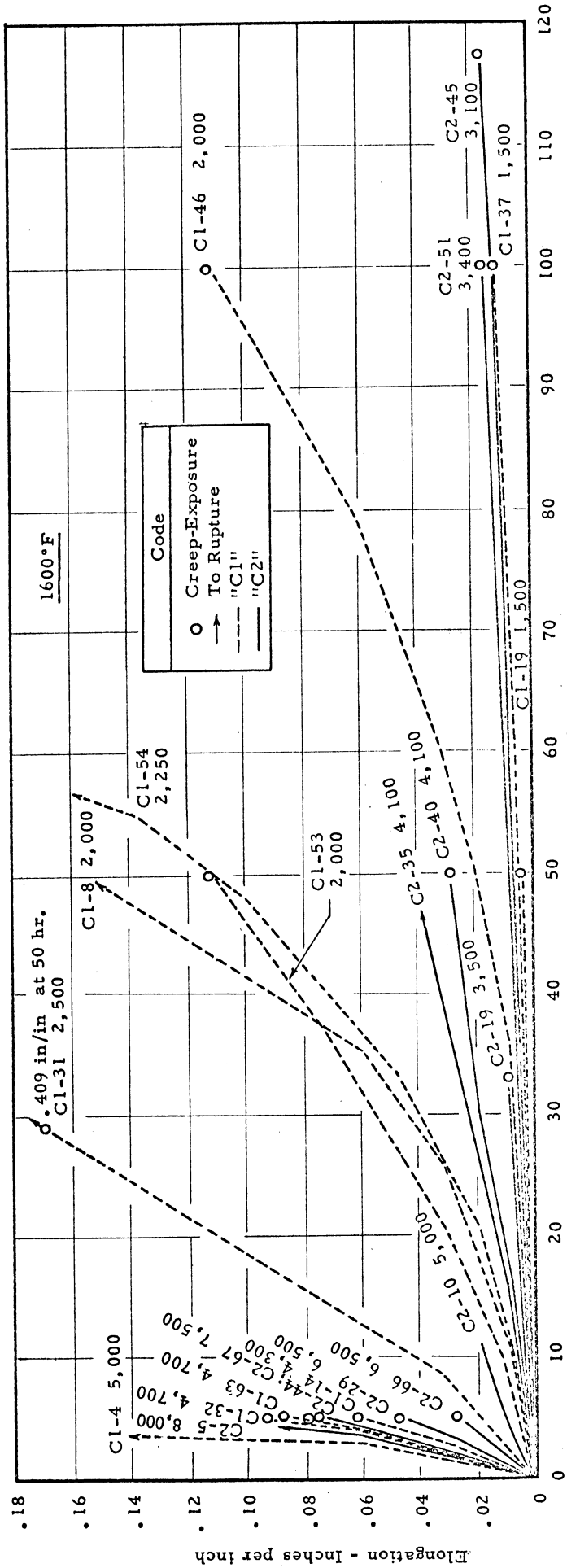


Figure 14 Time-Elongation Curves for 80Ni-20Cr at 1600°F

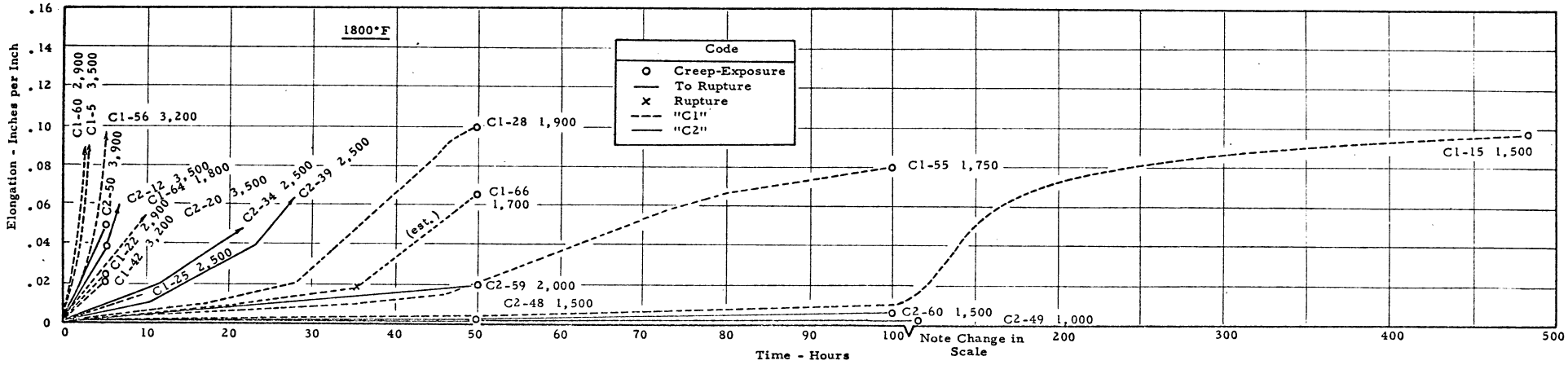


Figure 15 Time-Elongation Curves for 80Ni-20Cr at 1800°F

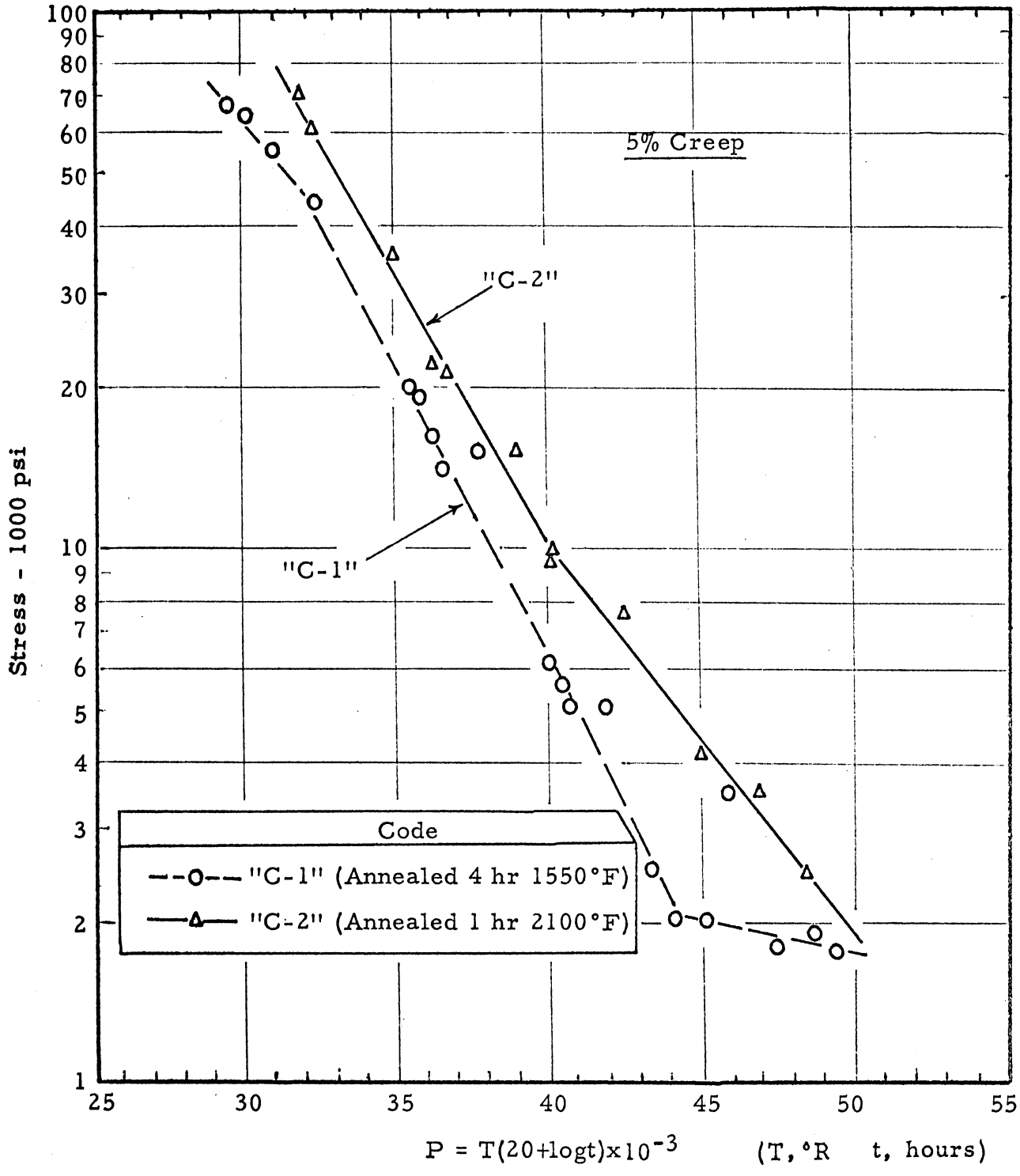


Figure 16 Master Curve for 5 Percent Creep for Annealed 80Ni-20Cr Alloy

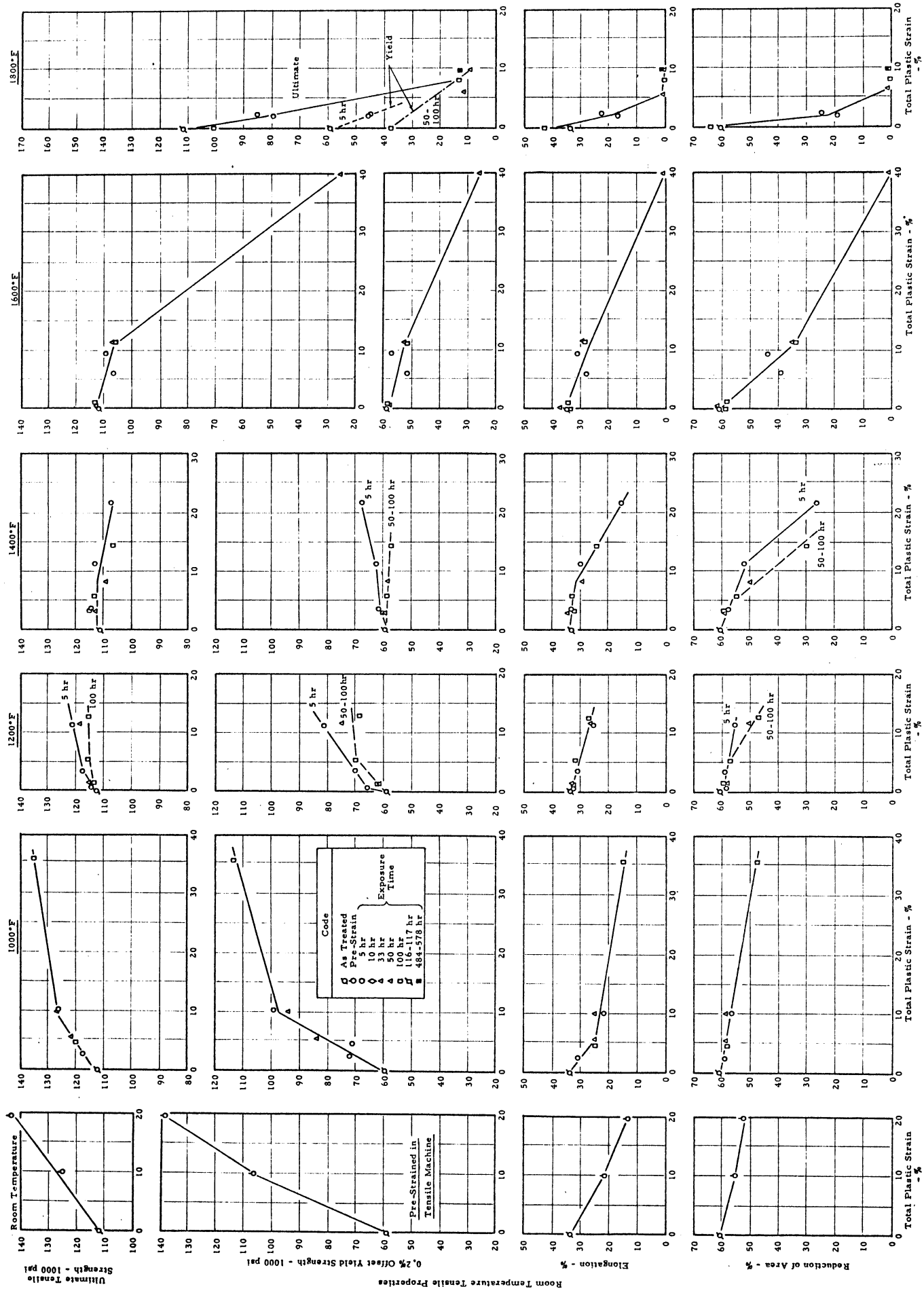


Figure 17 Effect of Prior Creep-Exposure on Room Temperature Tensile Properties of 80 Ni-20 Cr Alloy (Condition "C-1" (small grains))

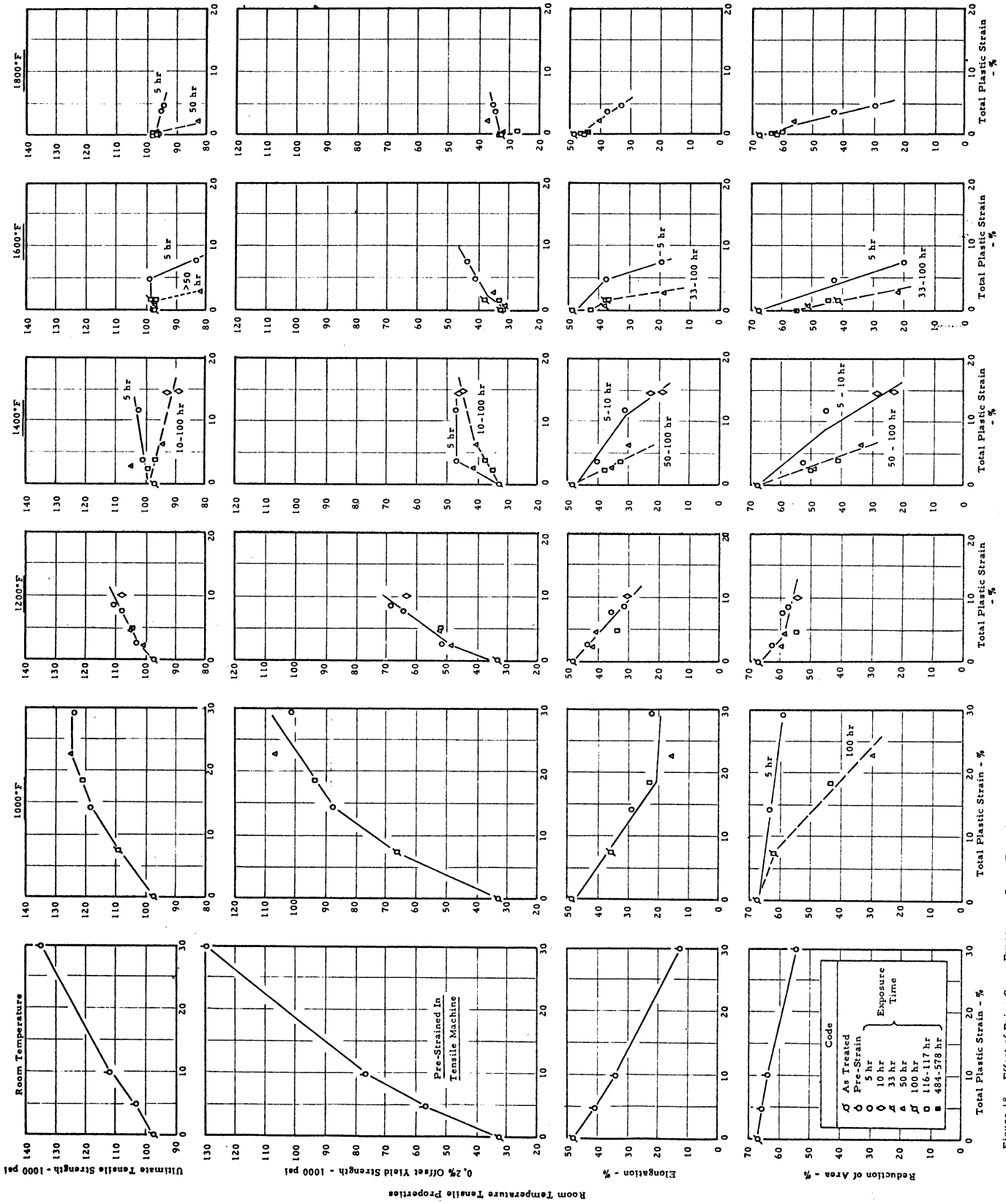


Figure 18 Effect of Prior Creep Exposure on Room Temperature Tensile Properties of 80 Ni-20 Cr Alloy (Condition "C-2" (large grains))

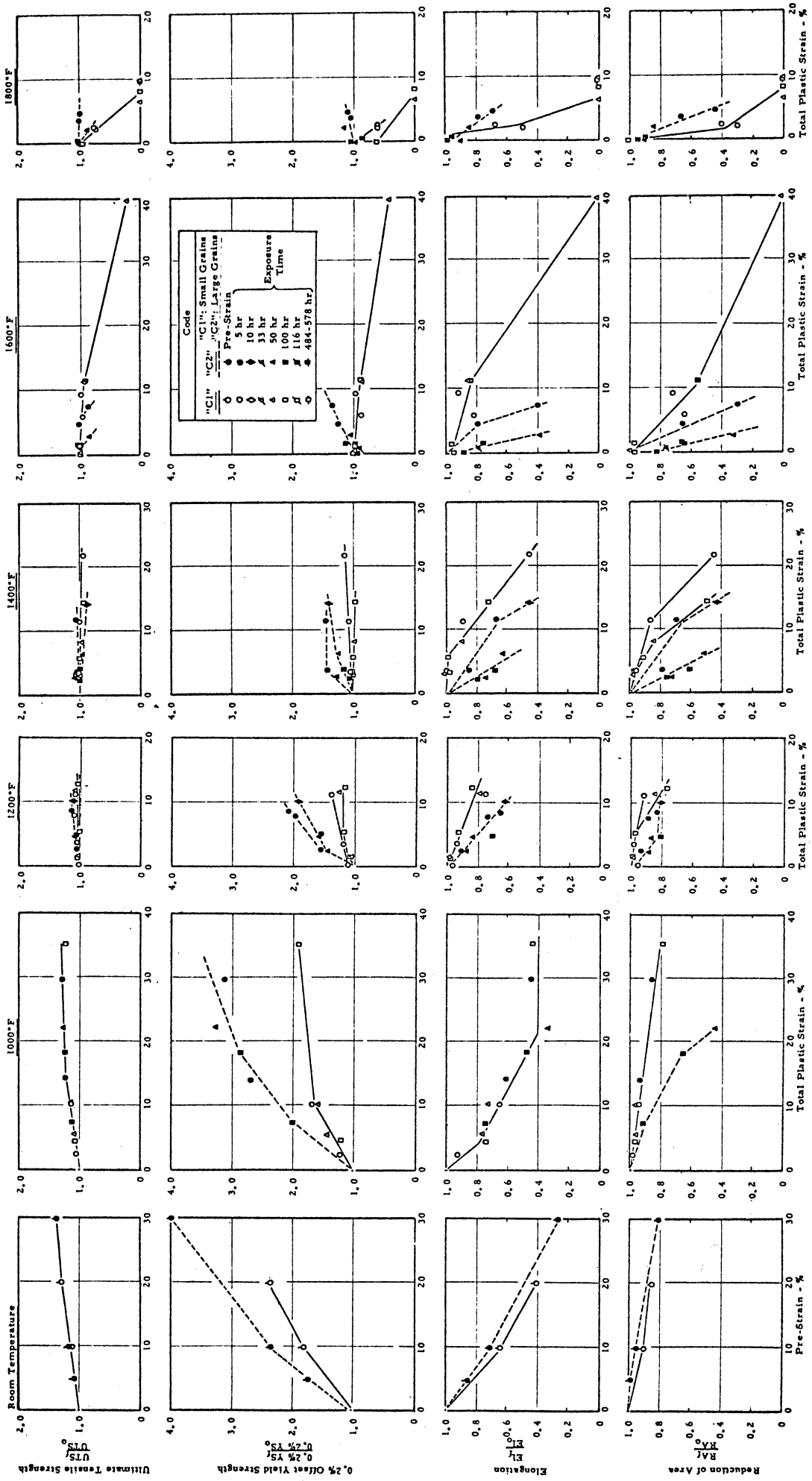


Figure 19 Relative Effect of Prior Creep on Room Temperature Tensile Properties of Two Conditions of 80 NI-20 Cr Alloy

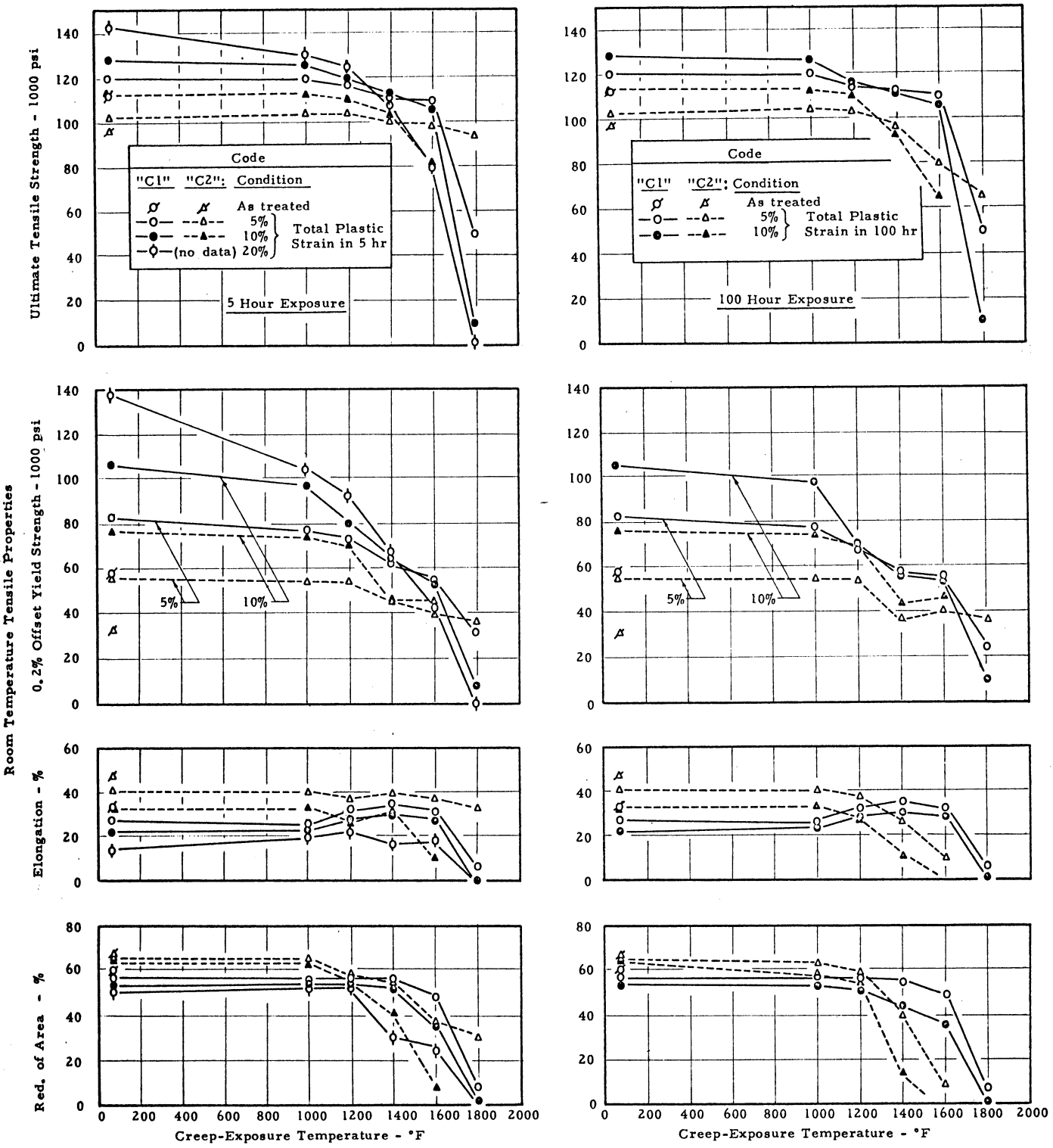
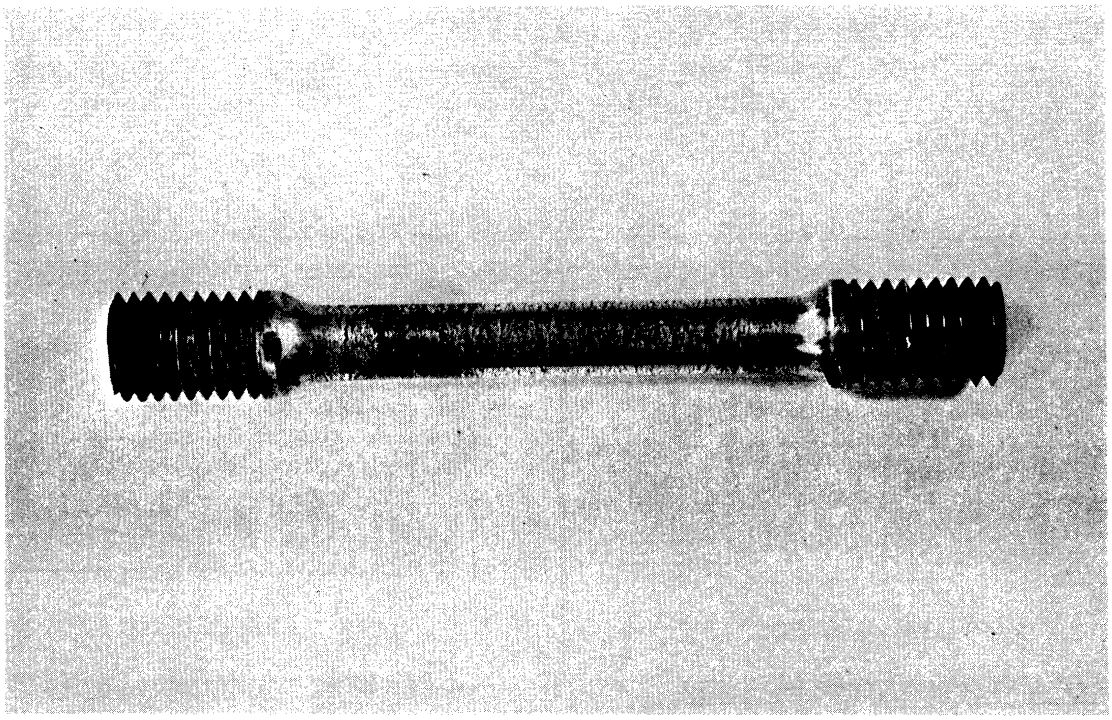
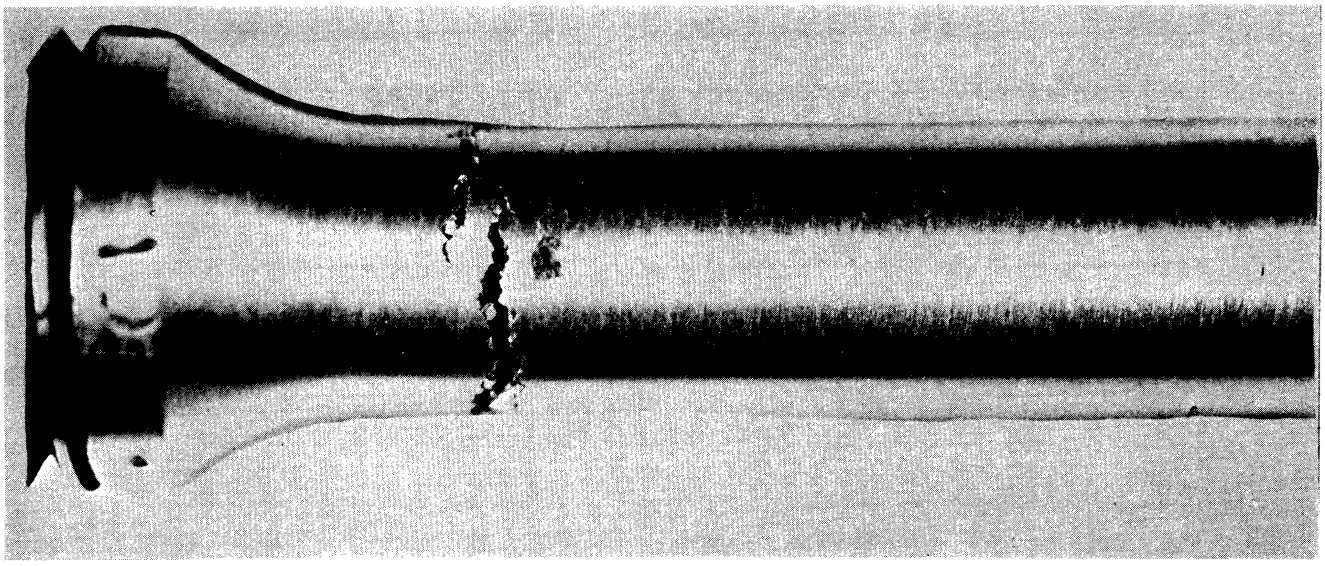


Figure 20 Effect of Creep-Exposure Temperature on Room Temperature Tensile Properties of 80 Ni-20 Cr Alloy Subjected to Prior Creep of 5 to 20 Percent in 5 Hours or 100 Hours



1.15x

Figure 21 Specimen No. C1-28 after 50 hours Creep-Exposure at 1800°F and 1900 psi. Note the extensive cracking. Specimen elongation was 9.3 percent and reduction of area was 4.0 percent after the creep-exposure. The ductility was negligible in the subsequent tensile test. (Surface cleaned before photographing)



4.8x

Figure 22 Specimen No. C1-55 after 100 hours Creep-Exposure at 1800°F and 1750 psi. Specimen elongation was approximately 8 percent and reduction of area 4 percent after the creep-exposure. The ductility in the room temperature tensile test was negligible. (Surface cleaned before photographing)

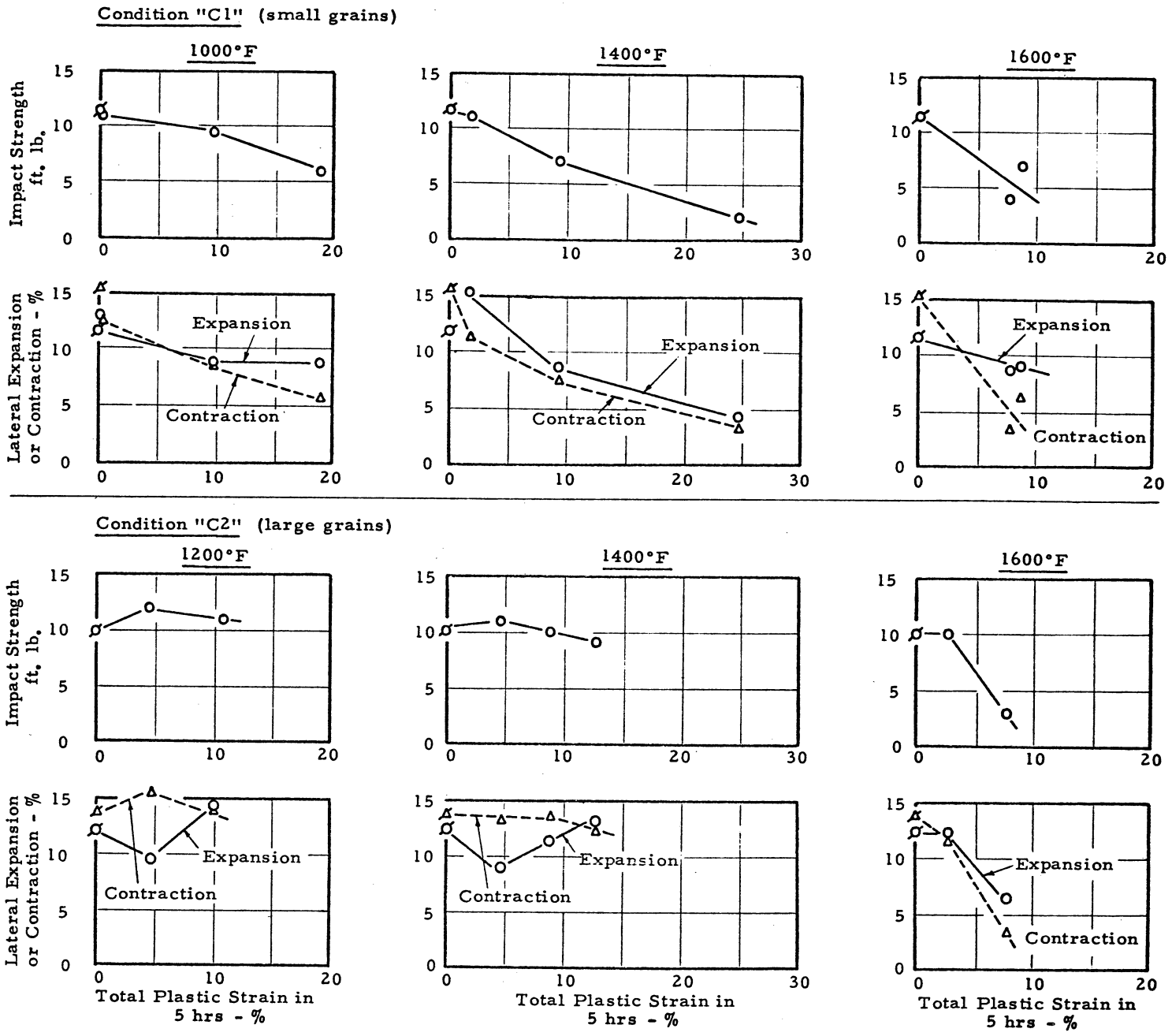


Figure 23 Effect of 5 Hours Prior Creep-Exposure on Izod Impact Properties of 80 Ni-20 Cr Alloy

Annealed 1550°F-4 hr.

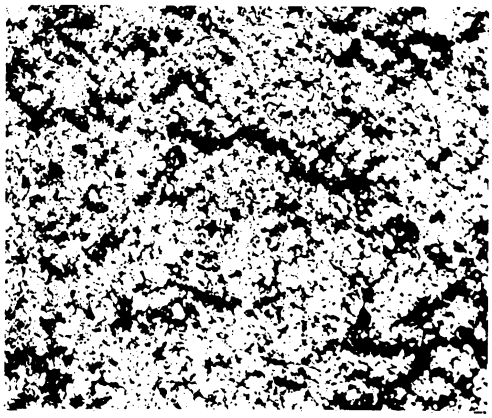


Figure 24 100x
Spec. No. C1-10: 1200°F - 20,000 psi. Failed at 63±5 hrs.

Annealed 2100°F-1 hr.

Figure 25 100x
Spec. No. C2-6: 1200°F - 22,000 psi. Failed at 109.9 hrs.

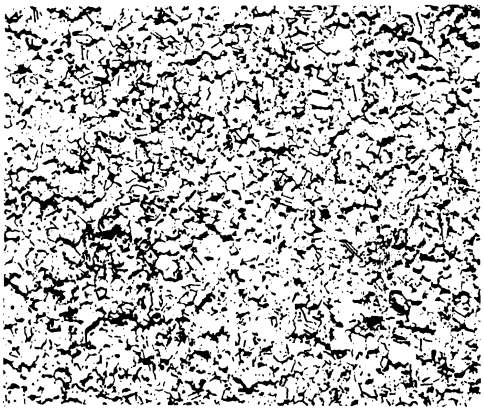


Figure 26 100x
Spec. No. C1-4: 1600°F - 5000 psi. Failed at 4.9 hrs.

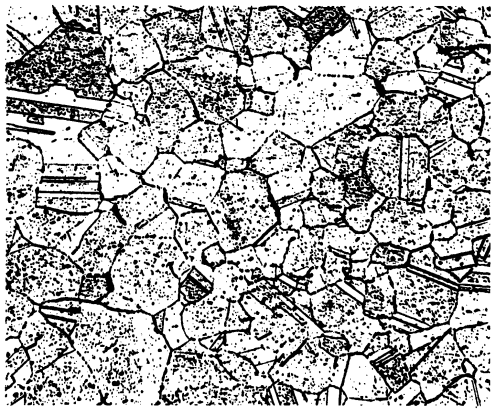


Figure 27 100x
Spec. No. C2-5: 1600°F - 8000 psi. Failed at 4.7 hrs.

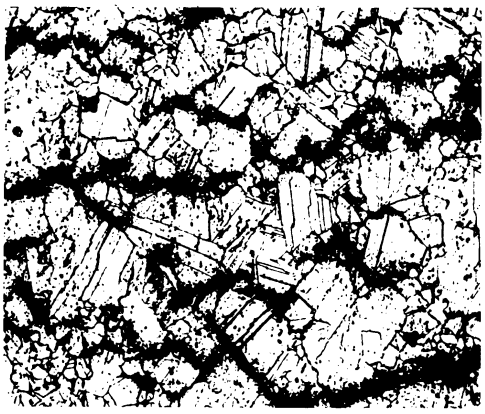


Figure 28 100x
Spec. No. C1-5: 1800°F - 3500 psi. Failed at 3.6 hrs.

Figure 29 100x
Spec. No. C2-12: 1800°F - 3500 psi. Failed at 10.1 hrs.

Etchant: Marble's Reagent

Optical Micrographs of 80Ni-20Cr Alloy After Rupture.

Figure 30 Spec. No. C2-31
Creep-Exposure 10 hours at 1400°F to 14.6% total plastic strain. NOT remachined before tensile test.

Tensile Properties

UTS	=	88,100 psi
.2% YS	=	44,300 psi
El	=	18.1%
RA	=	23.6%

100x

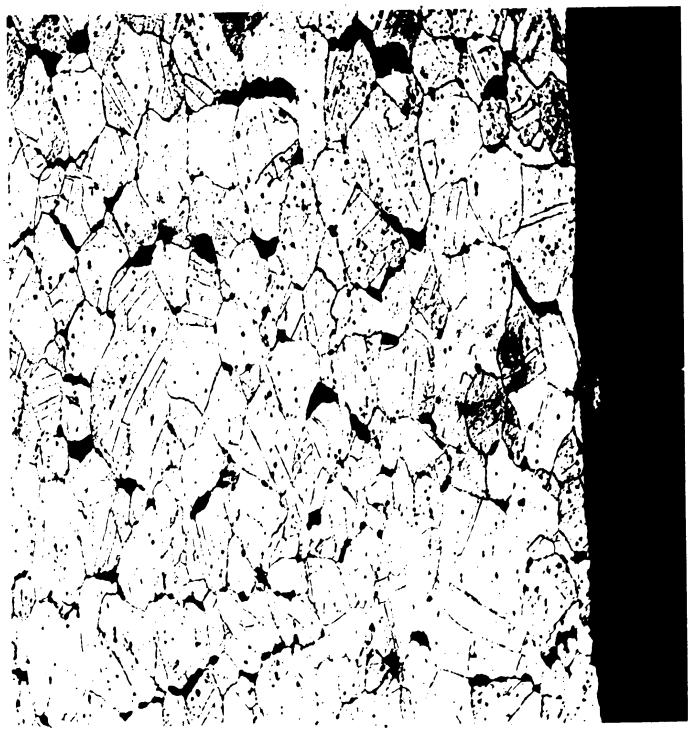


Figure 31 Spec. No. C2-18
Creep-Exposure 10 hours at 1400°F to 14.1% total plastic strain. 0.023-inches machined from diameter before tensile test.

Tensile Properties

UTS	=	93,000 psi
.2% YS	=	45,900 psi
El	=	22.3%
RA	=	28.2%

100x

Effect of Remachining Gage Section of 80Ni-20Cr Creep-Exposure Specimen Prior to Room Temperature Tensile Test

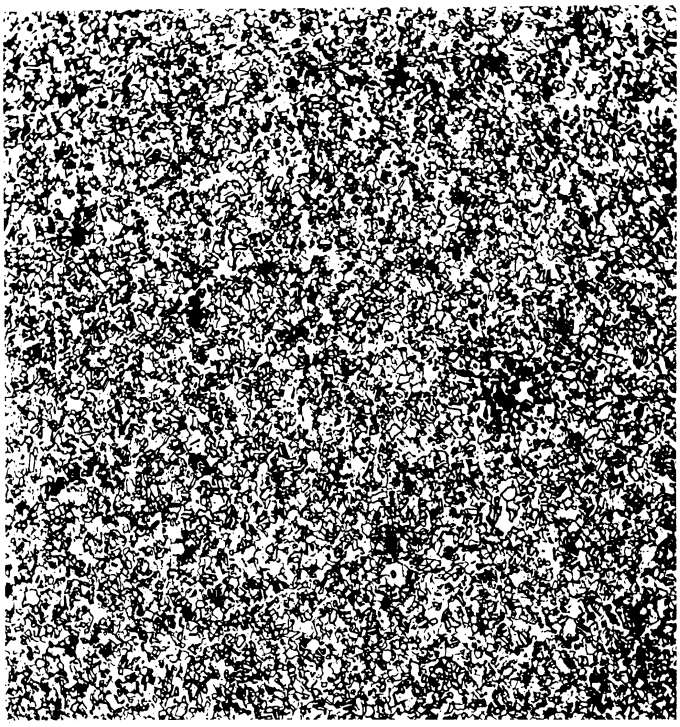


Figure 32 100x
As-Heat Treated C-1 Condition

Figure 33 250x
As-Heat Treated C-1 Condition

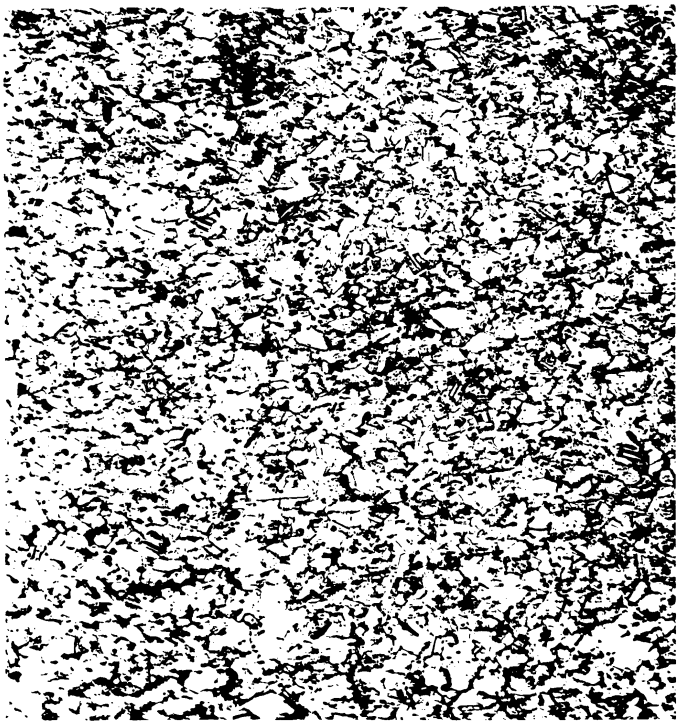


Figure 34 250x
Spec. C1-61 Creep-Exposure
5 hours at 1400°F to 21.7%
Deformation, Then Tensile
Tested at Room Temperature

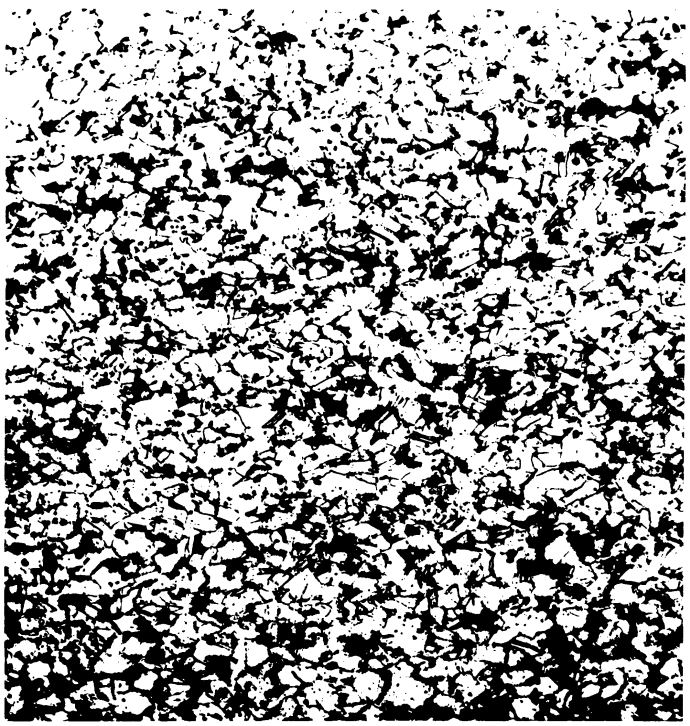


Figure 35 250x
Spec. C1-18 Creep-Exposure
50 hours at 1400°F to 2.9%
Deformation, Then Tensile
Tested at Room Temperature

Figure 36 100x

Spec. C1-53 Creep-Exposure
50 hours at 1600°F to 11.2%
Deformation, Then Tensile
Tested at Room Temperature

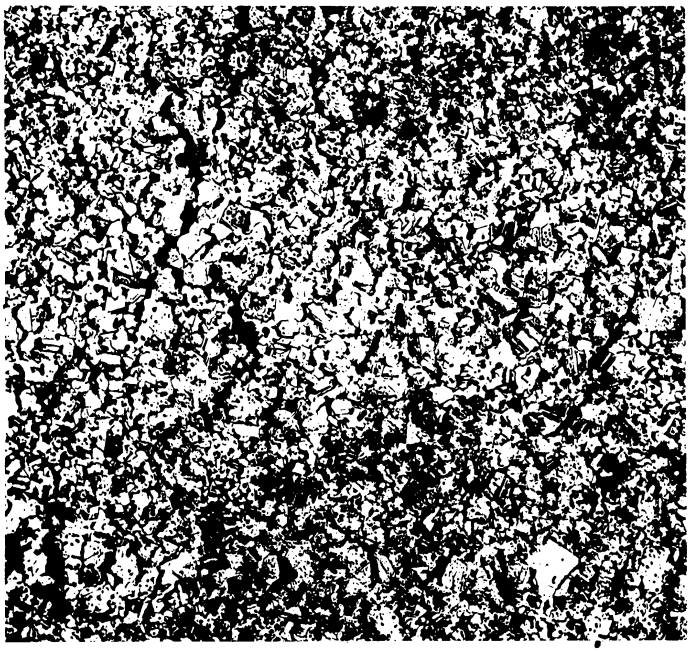


Figure 37 100x

Spec. C1-31 Creep-Exposure
50 hours at 1600°F to 40.9%
Deformation, Then Tensile
Tested at Room Temperature

Figure 38 Approx. 120x

Spec. C1-46 Creep-Exposure 100 hours at 1600°F to 10.5% Deformation,
Then Tensile Tested at Room Temperature

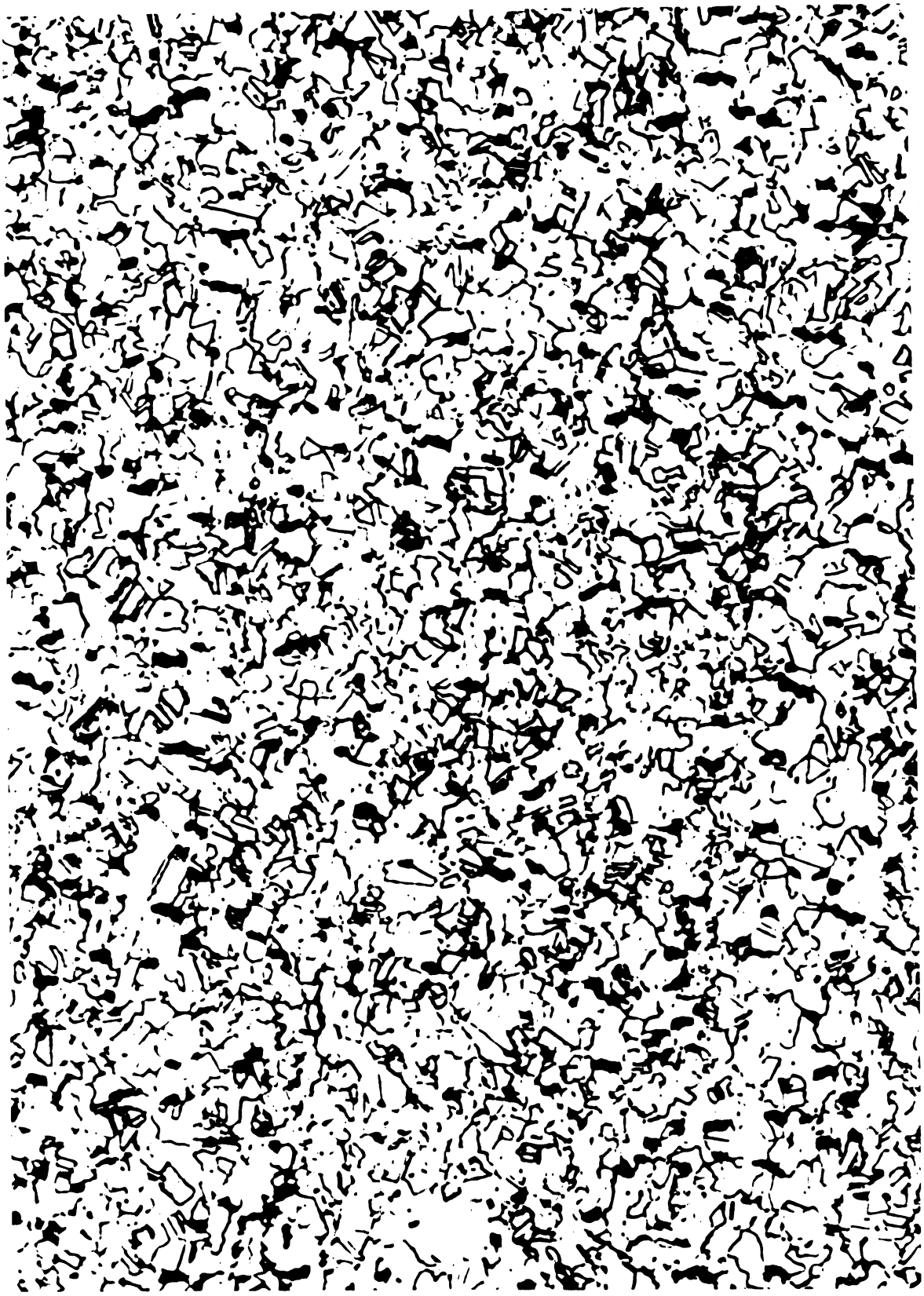


Figure 39 taken at 100x, enlarged to 360x
Spec. C1-59 Creep-Exposure 5 hours at 1600°F to 13% Deformation, NOT Tensile Tested

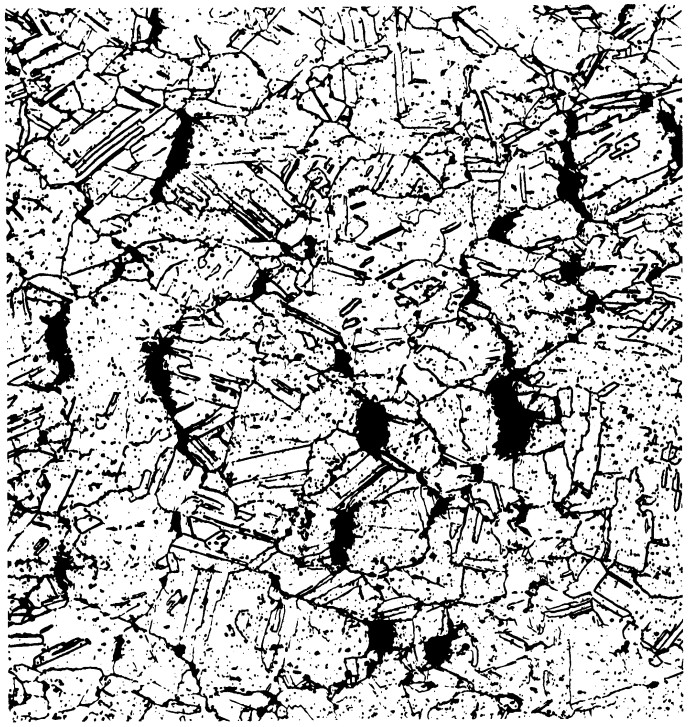


Figure 40 100x

Spec. C1-22 Creep-Exposure
5 hours at 1800°F to 2.5%
Deformation, Then Tensile
Tested at Room Temperature

Figure 41 100x

Spec. C1-66 Creep-Exposure
50 hours at 1800°F to 6.5%
Deformation, Then Tensile
Tested at Room Temperature

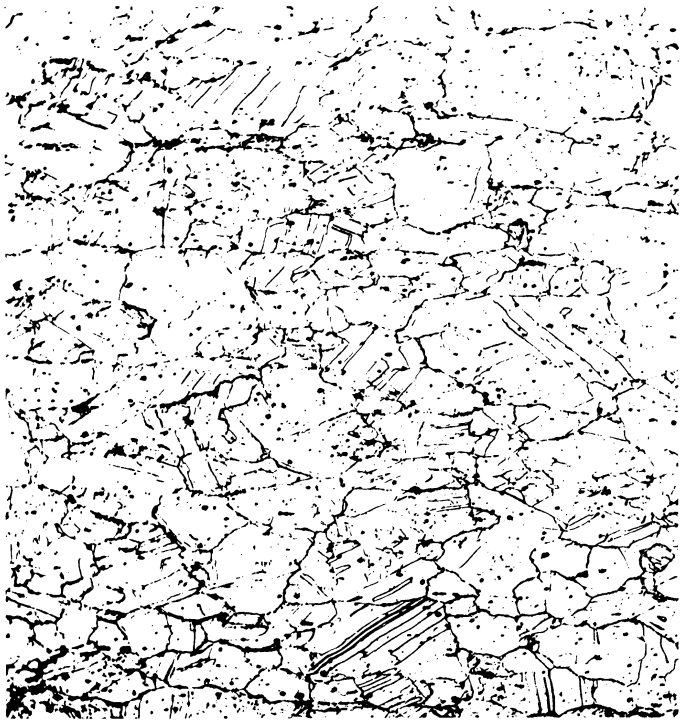


Figure 42 100x

Spec. C1-58 Unstressed Exposure
100 hours at 1800°F, Then Tensile
Tested at Room Temperature

Figure 43 100x

Spec. C1-15 Creep-Exposure
484 hours at 1800°F to 9.8%
Deformation, Then Tensile
Tested at Room Temperature

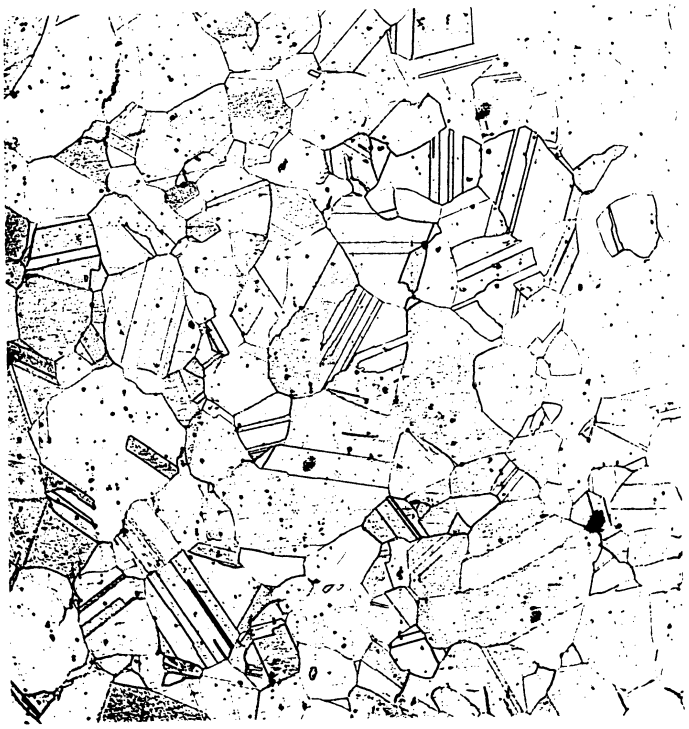


Figure 44 100x
C-2 Condition As-Heat Treated

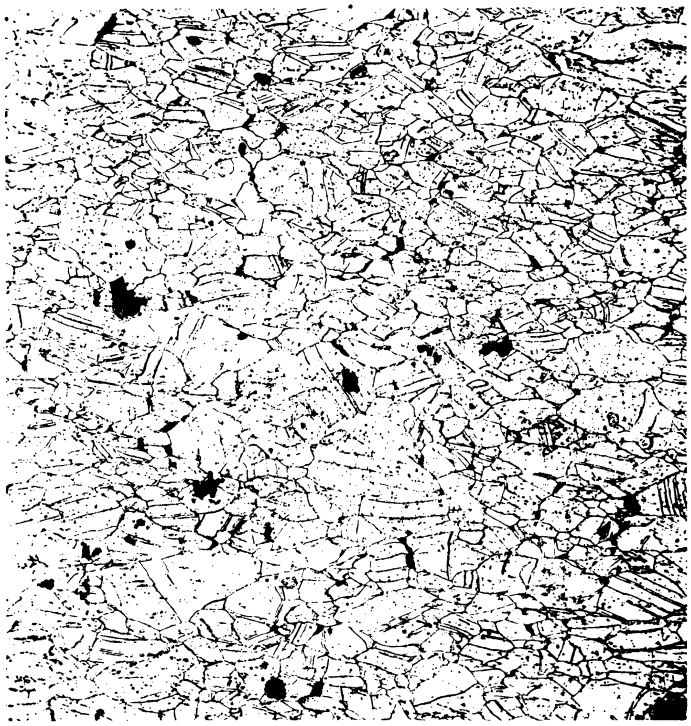


Figure 45 50x
Spec. C2-47 Creep-Exposure
50 hours at 1000°F to 22.7%
Deformation, Then Tensile
Tested at Room Temperature

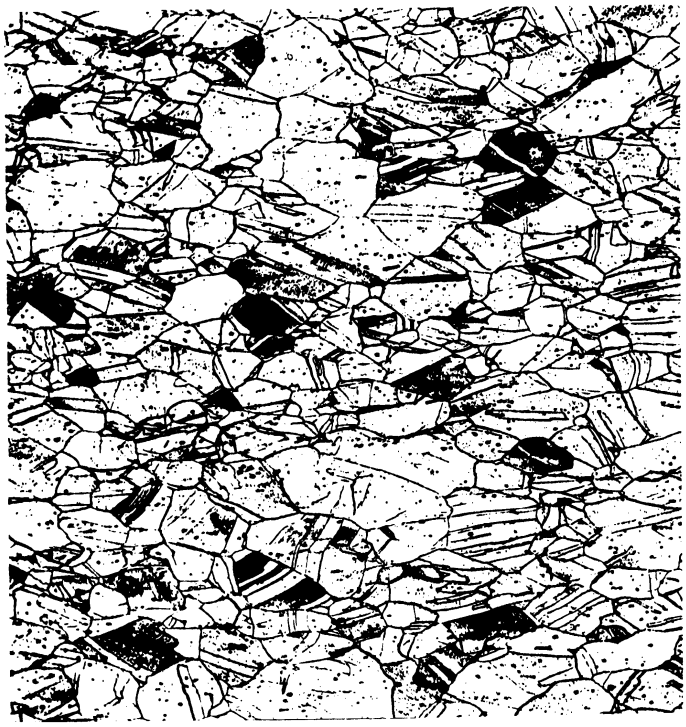


Figure 46 100x
Spec. C2-9 Creep-Exposure
5 hours at 1200°F to 8.6%
Deformation, Then Tensile
Tested at Room Temperature

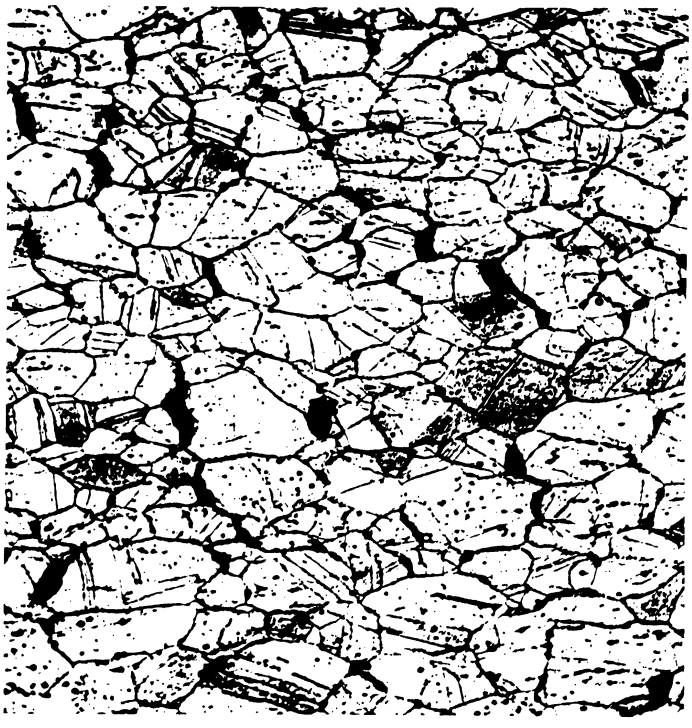


Figure 47 100x
Spec. C2-38 Creep-Exposure
50 hours at 1400°F to 6.2%
Deformation, Then Tensile
Tested at Room Temperature

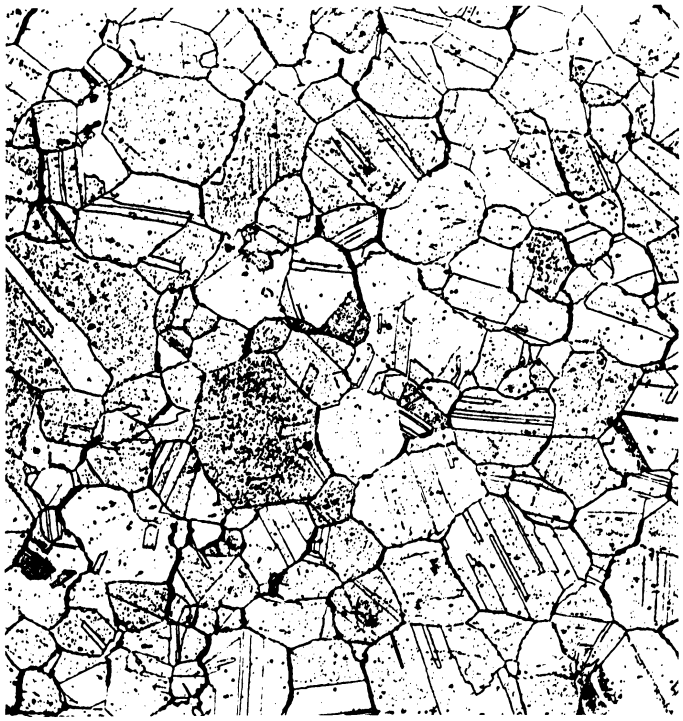


Figure 48 100x

Spec. C2-61 Creep-Exposure
5 hours at 1600°F to 9.2%
Deformation, NOT Tensile
Tested

Figure 49 100x

Spec. C2-62 Creep-Exposure
50 hours at 1600°F to 3.8%
Deformation, NOT Tensile
Tested

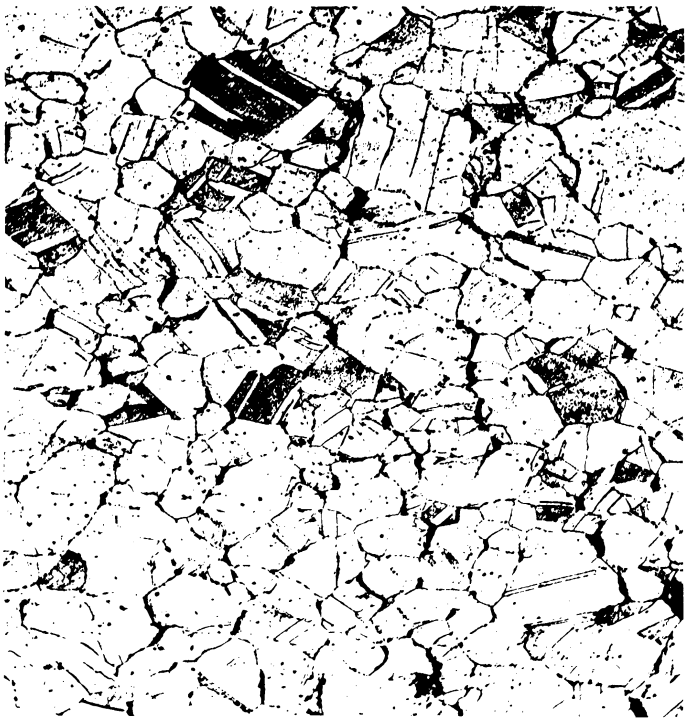


Figure 50 100x

Spec. C2-44 Creep-Exposure
5 hours at 1600°F to 7.5%
Deformation, Then Tensile
Tested at Room Temperature

Figure 51 100x

Spec. C2-63 Creep-Exposure
5 hours at 1800°F to 7.6%
Deformation, NOT Tensile
Tested

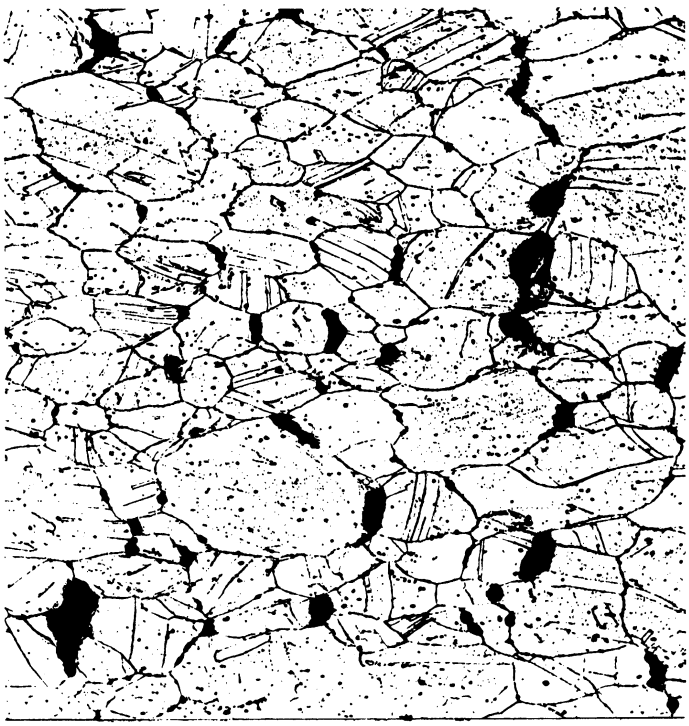


Figure 52 100x

Spec. C2-20 Creep-Exposure
5 hours at 1800°F to 3.9%
Deformation, Then Tensile
Tested at Room Temperature

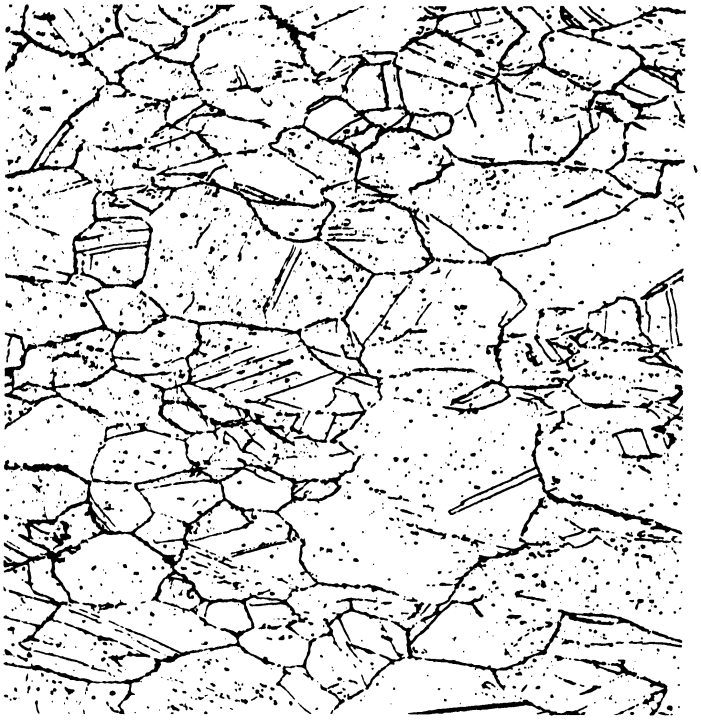
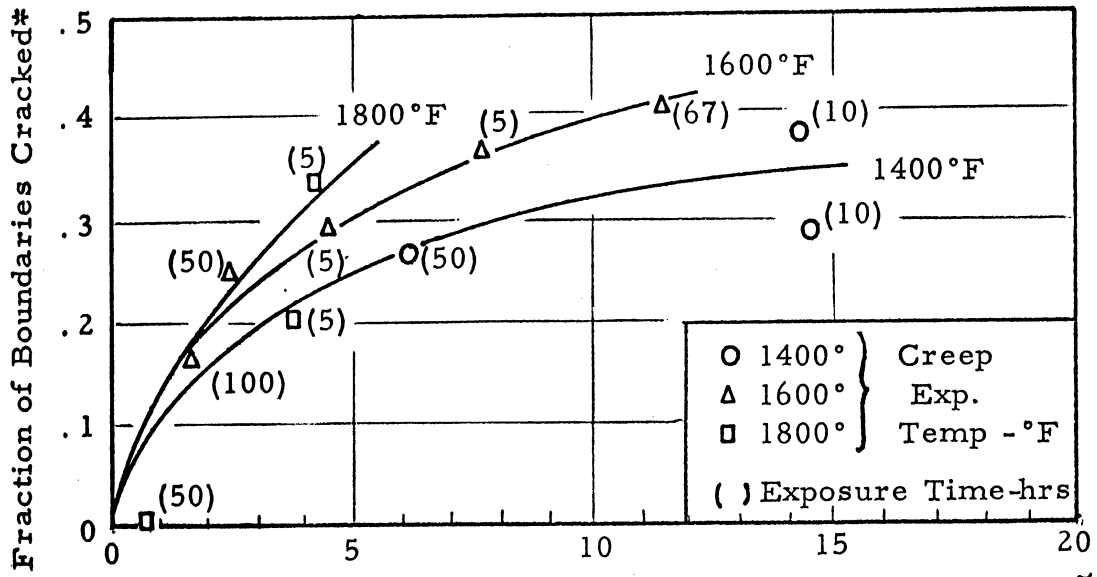
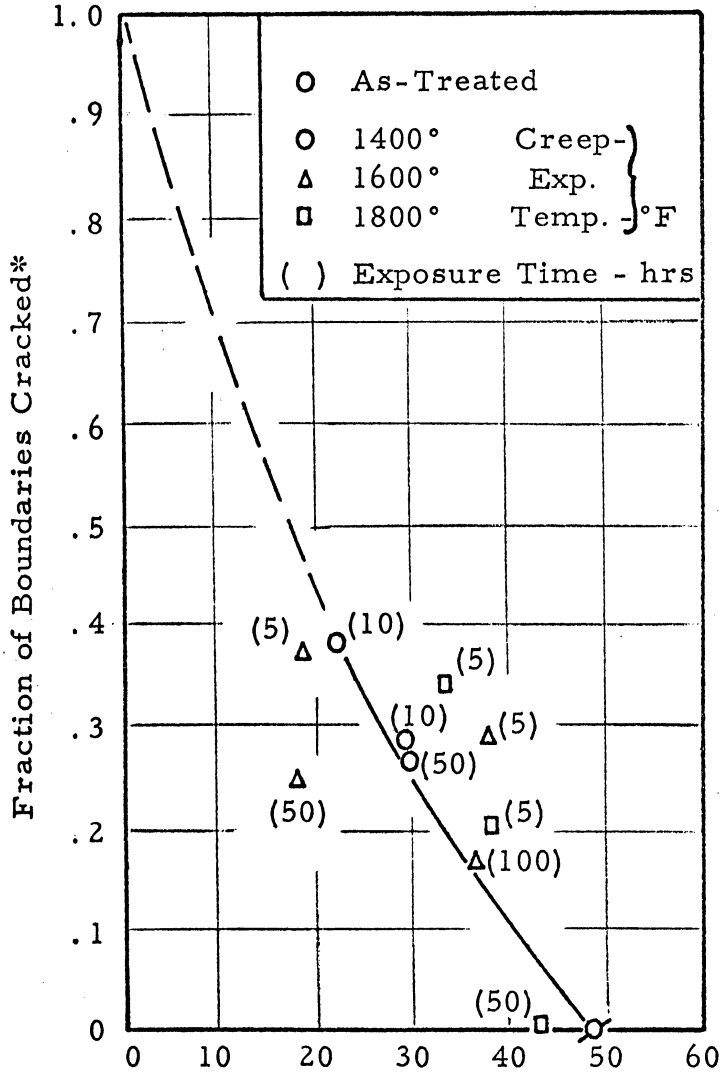


Figure 53 100x

Spec. C2-60 Creep-Exposure
100 hours at 1800°F to 0.6%
Deformation, Then Tensile
Tested at Room Temperature



54a) Deformation in 5-100 Hours at Indicated Temperature - %



54b) Room Temp. Tensile Test Elongation - %

* Fraction of Boundaries Cracked is the ratio:

$$\frac{\text{No. of Cracked Boundaries}}{\text{Total Boundaries Intersected}}$$

for a 4.5-inch line in the tension direction of an approx. 100x micrograph.

Figure 54 Relation of Cracking to Creep-Exposure Conditions and Subsequent Room Temp. Tensile Elongation of "C-2" Material

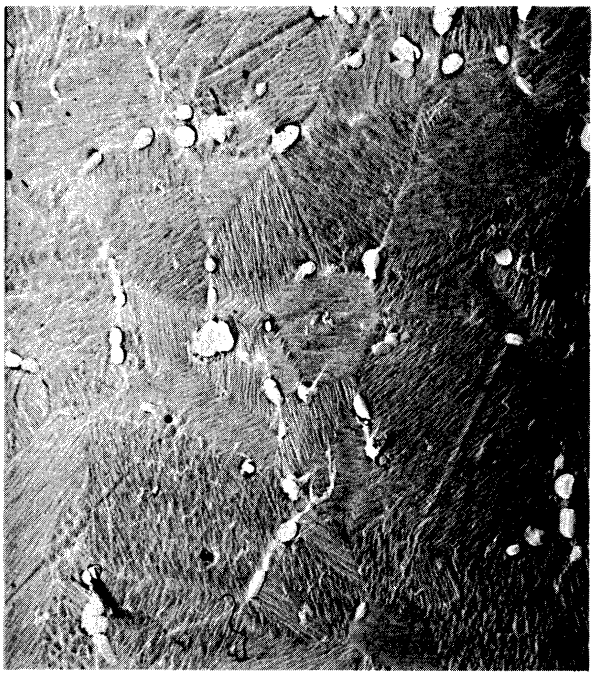


Figure 55 3500x
C1-1 Condition As-Heat Treated

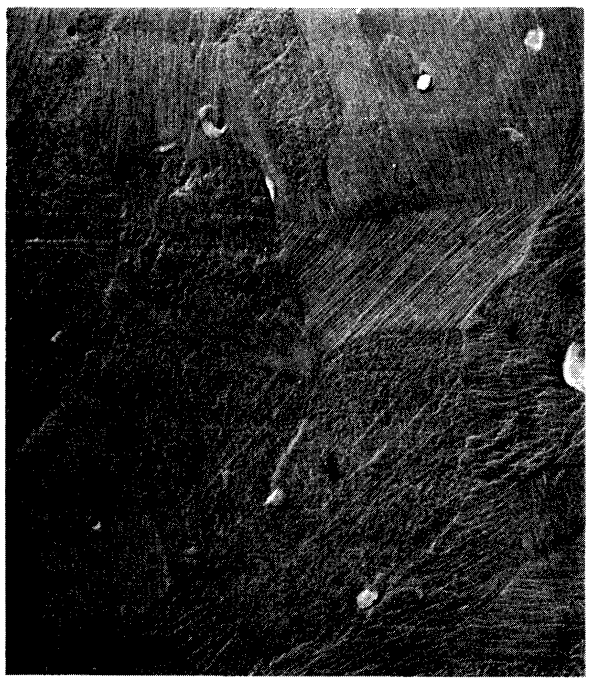


Figure 56 3500x
Spec. C1-14 Creep-Exposure
5 hours at 1600°F to 6%
Deformation

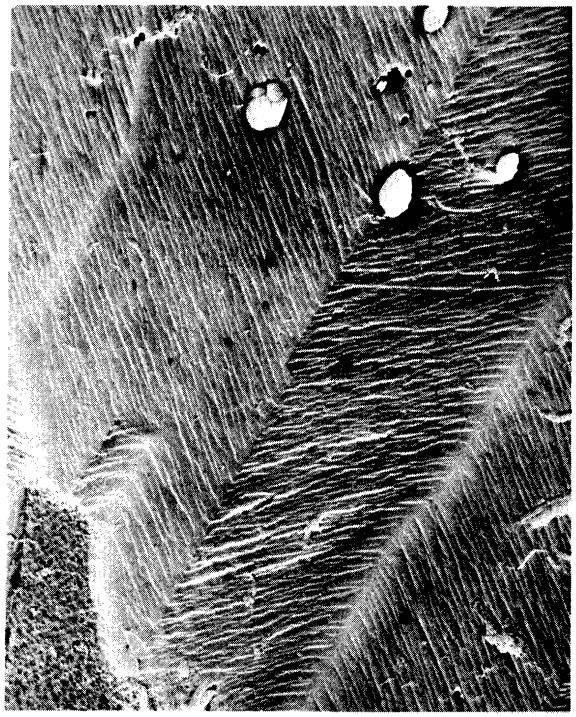


Figure 57 3500x
Spec. C1-22 Creep-Exposure
5 hours at 1800°F to 2.5%
Deformation

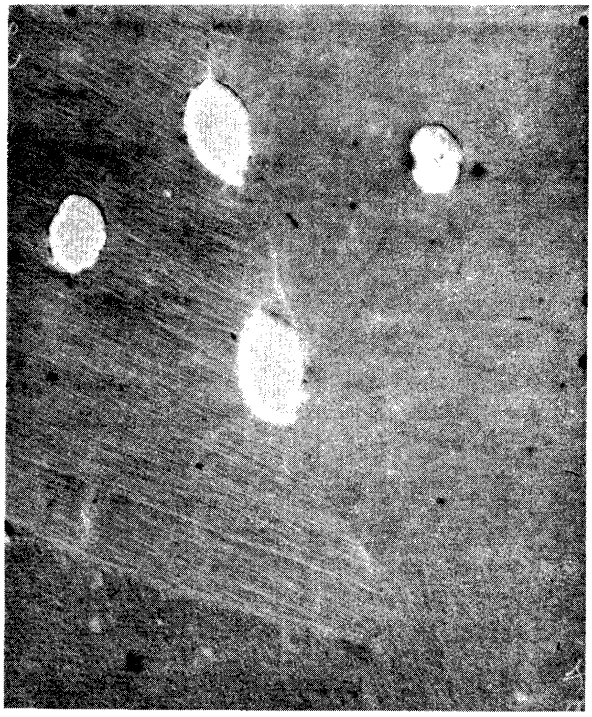


Figure 58 3500x
Spec. C1-15 Creep-Exposure
484 hours at 1800°F to 9.8%
Deformation

Electron Micrographs

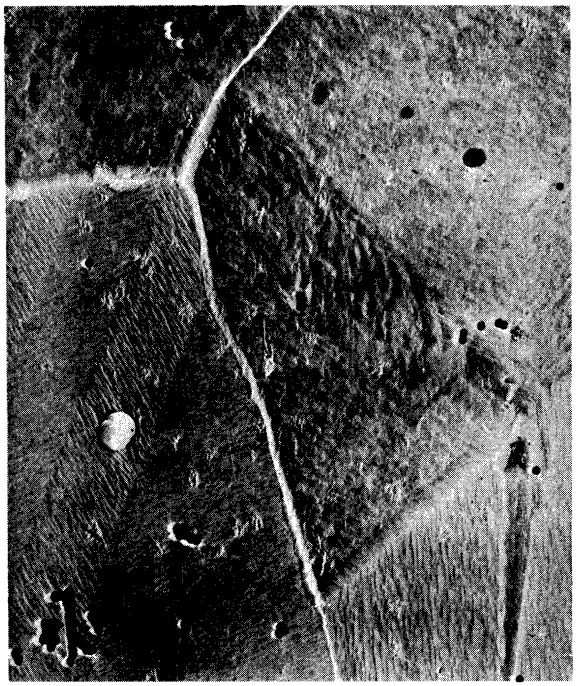


Figure 59 3500x
C2 Condition As-Heat Treated

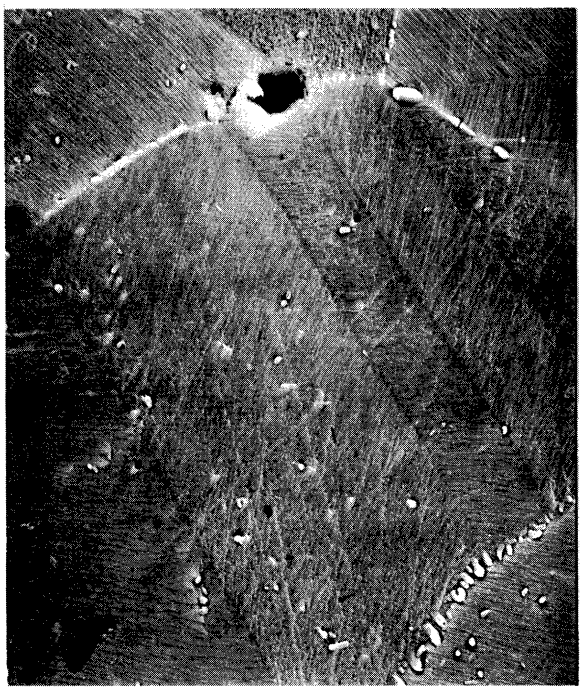


Figure 60 3500x
Spec. C2-18 Creep-Exposure
10 hours at 1400°F to 14.1%
Deformation

Figure 61 3500x
Spec. C2-62 Creep-Exposure
50 hours at 1600°F to 3.8%
Deformation

Figure 62 3500x
Spec. C2-50 Creep-Exposure
5 hours at 1800°F to 4.8%
Deformation

Electron Micrographs

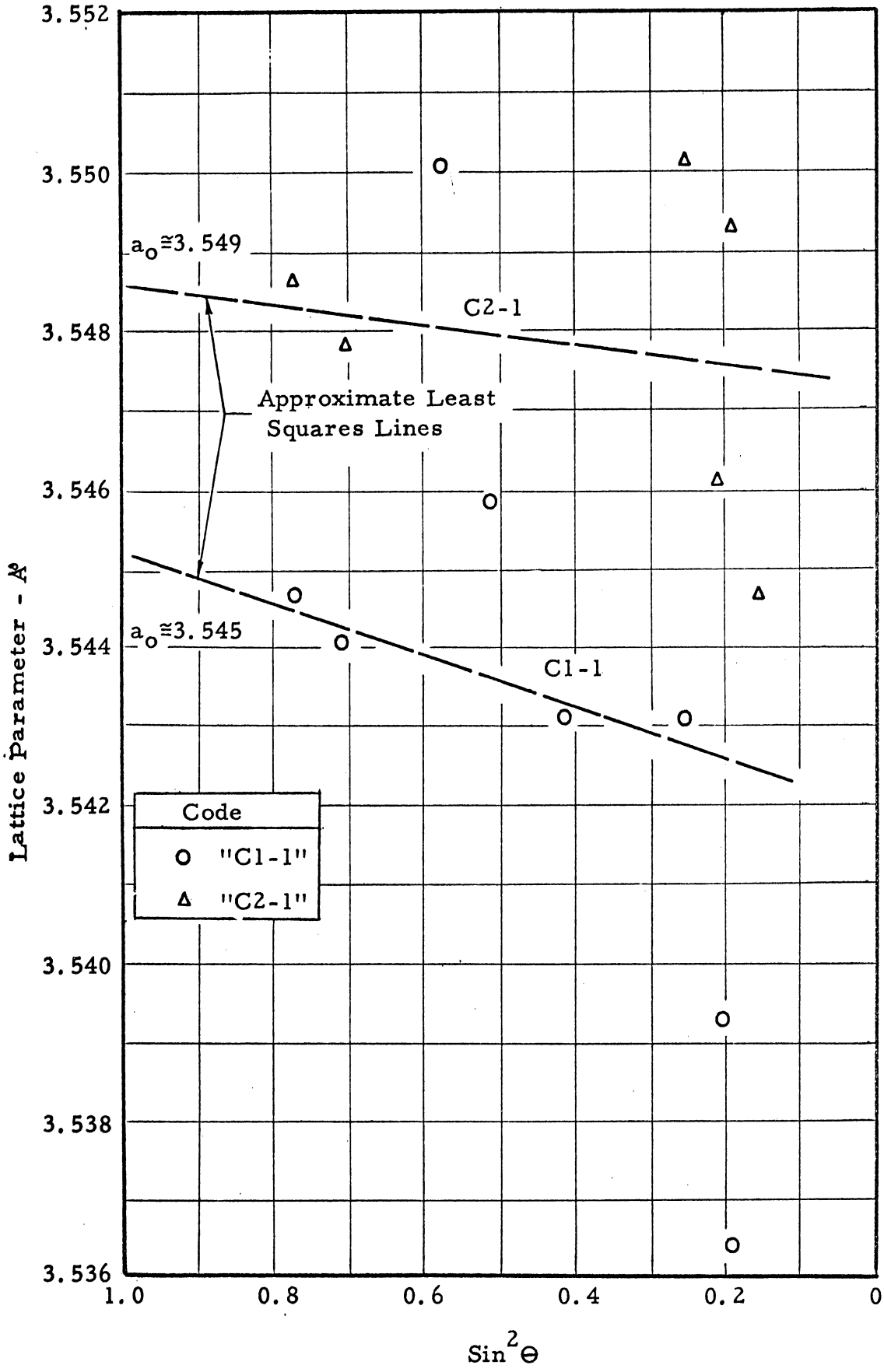


Figure 63 Lattice Parameter Determination for Annealed Samples of 80Ni-20Cr Alloy

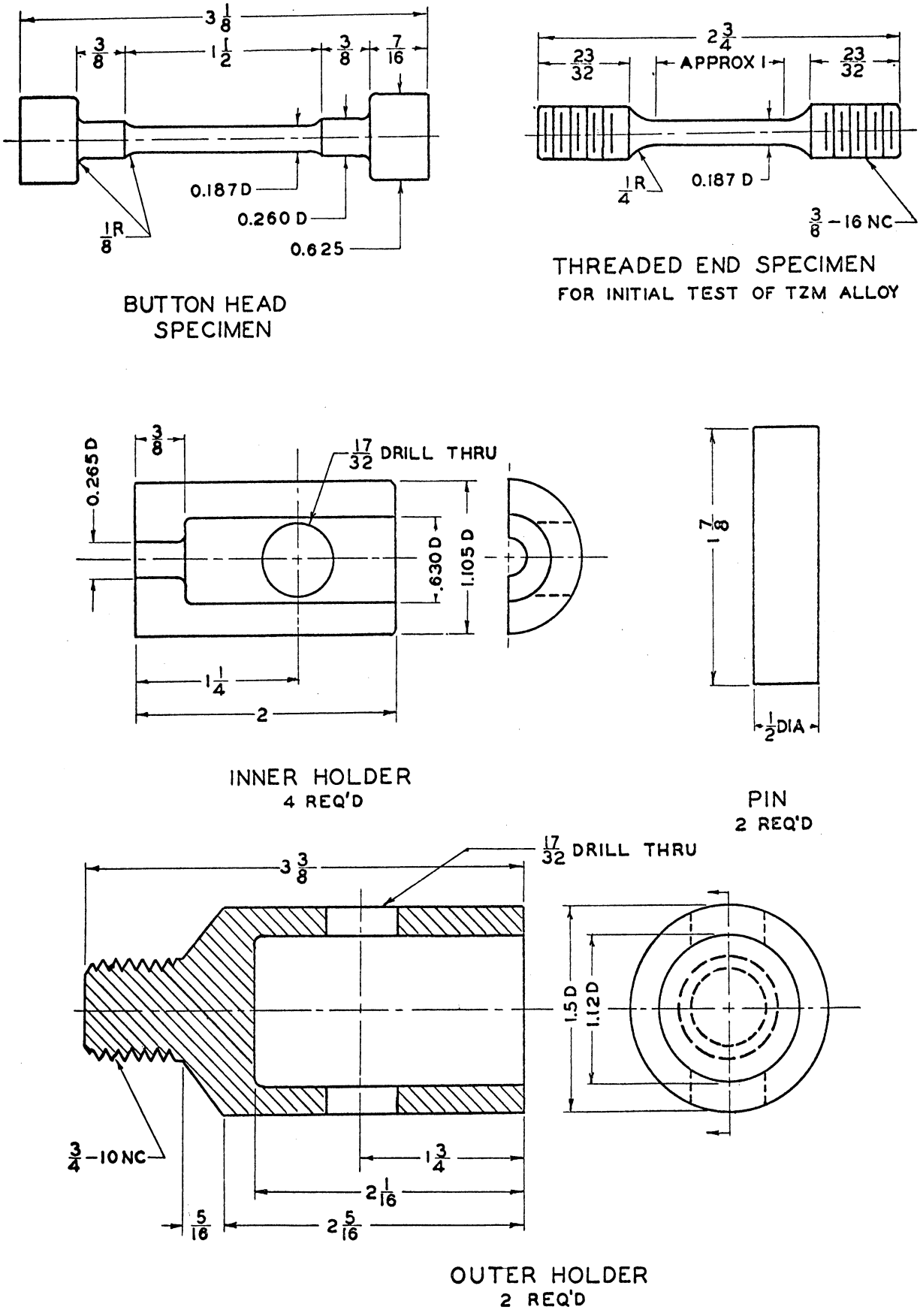


Figure 64 Details of Specimens and Holders for Tests of Molybdenum Alloy

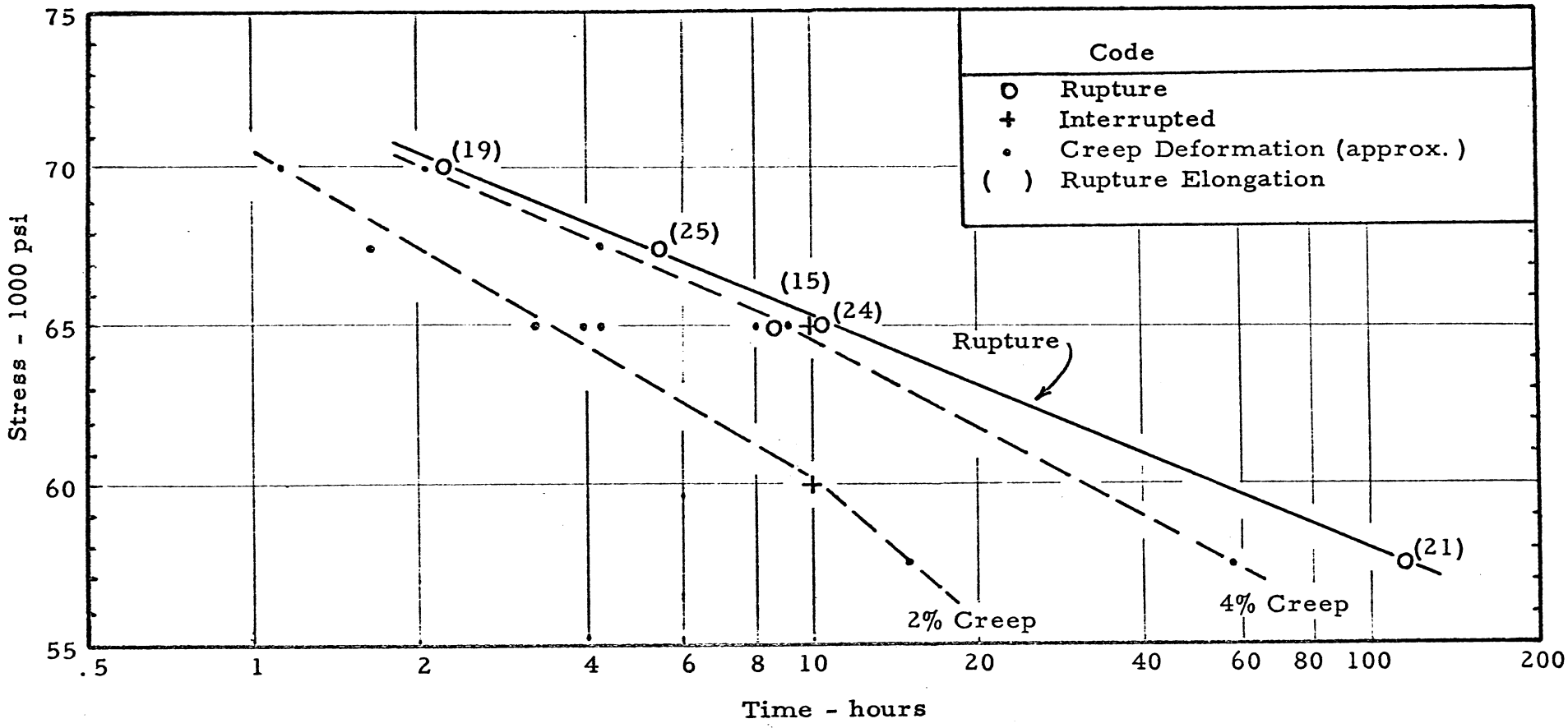


Figure 65 Effect of Stress on Creep-Rupture Life of TZM Alloy at 1800°F

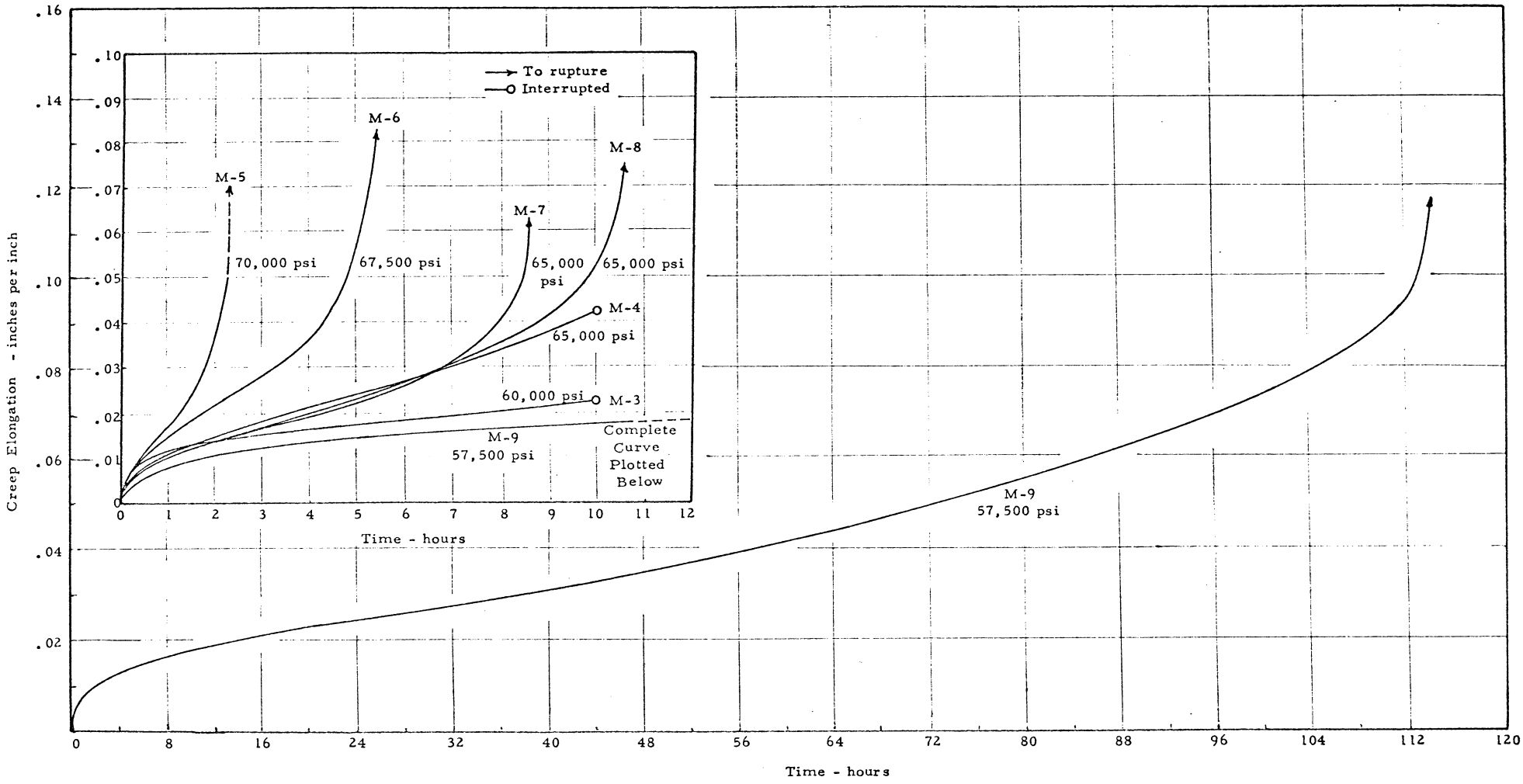


Figure 66 Time-Elongation Curves for TZM Alloy at 1800°F

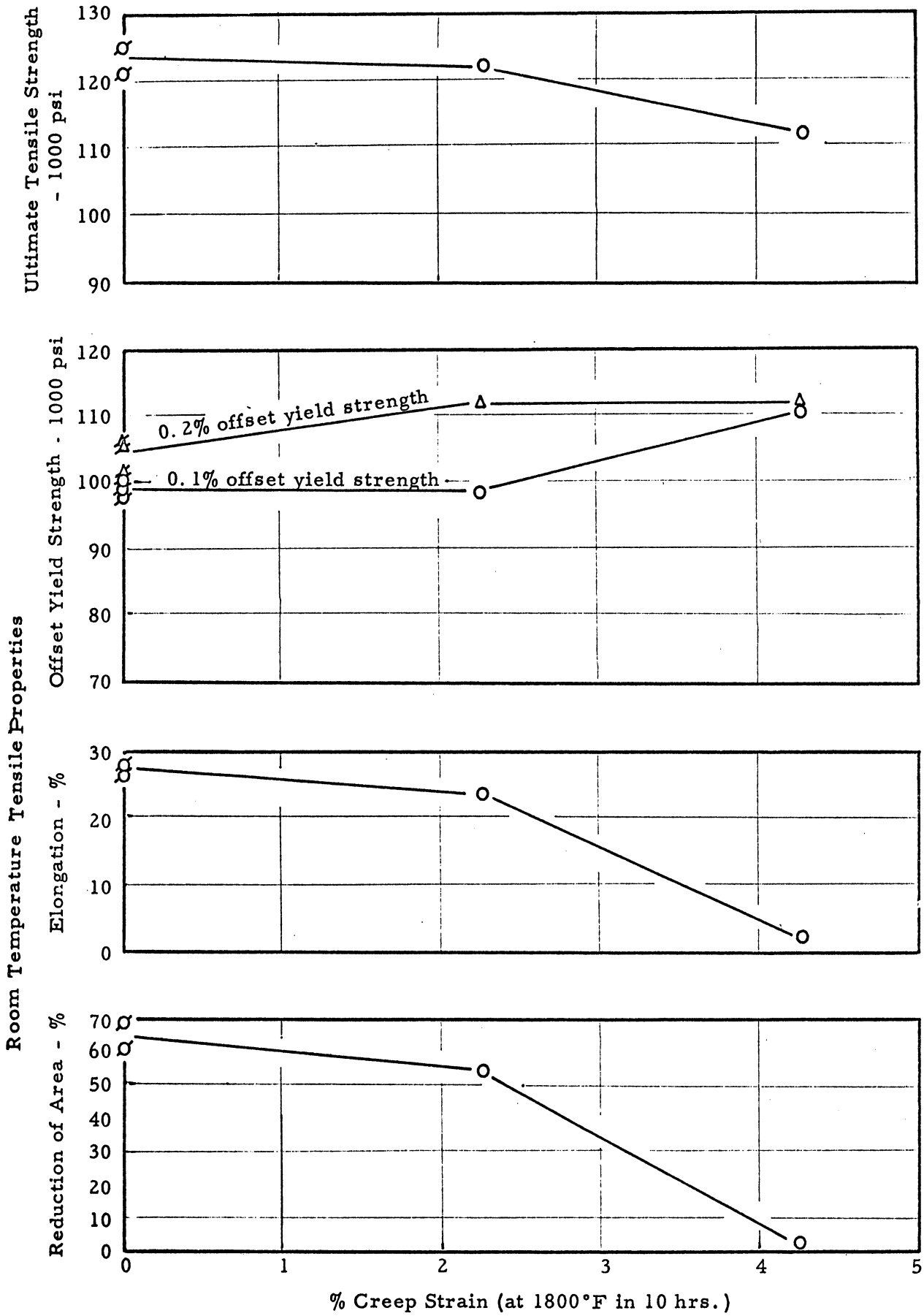


Figure 67 Effect of Creep-Exposure at 1800°F on Room Temperature Tensile Properties of TZM Alloy

Figure 68 500x

TZM As-Received (Transverse Section) (Rolled and Stress-Relieved)

Figure 69 500x

TZM As-Received (Longitudinal Section) (Rolled and Stress-Relieved)

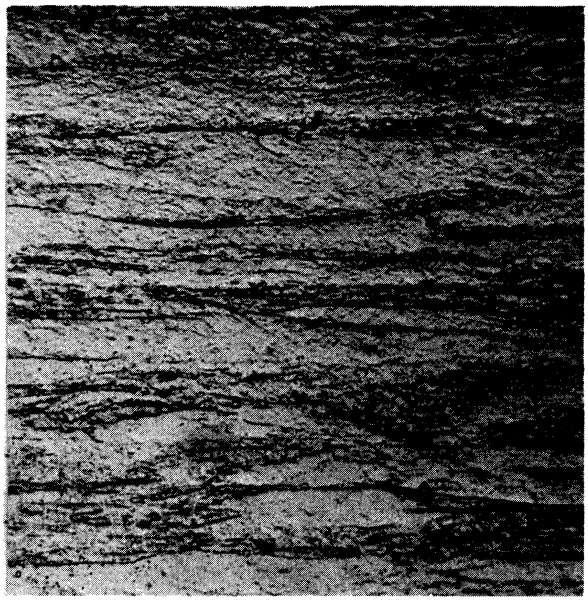


Figure 70 500x

TZM Spec. M-4
Creep-Exposure 10 hours at 1800°F to 4.26 Percent Deformation, Then Tensile Tested at Room Temperature

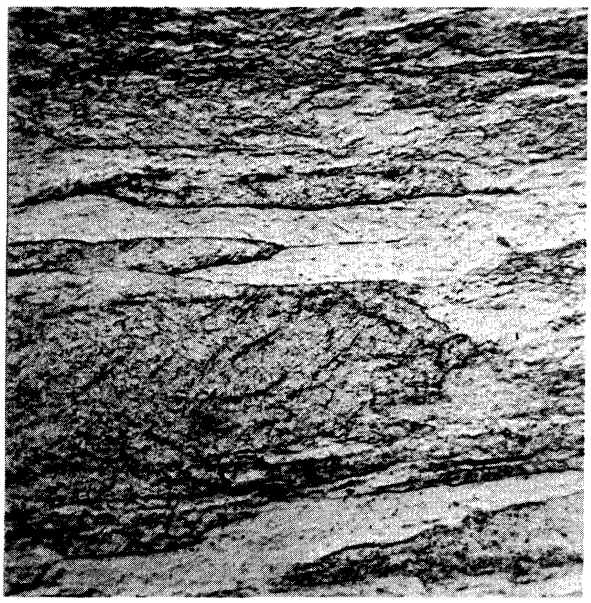


Figure 71 500x

TZM Spec. M-9
Creep-Rupture after 114.5 hours at 1800°F

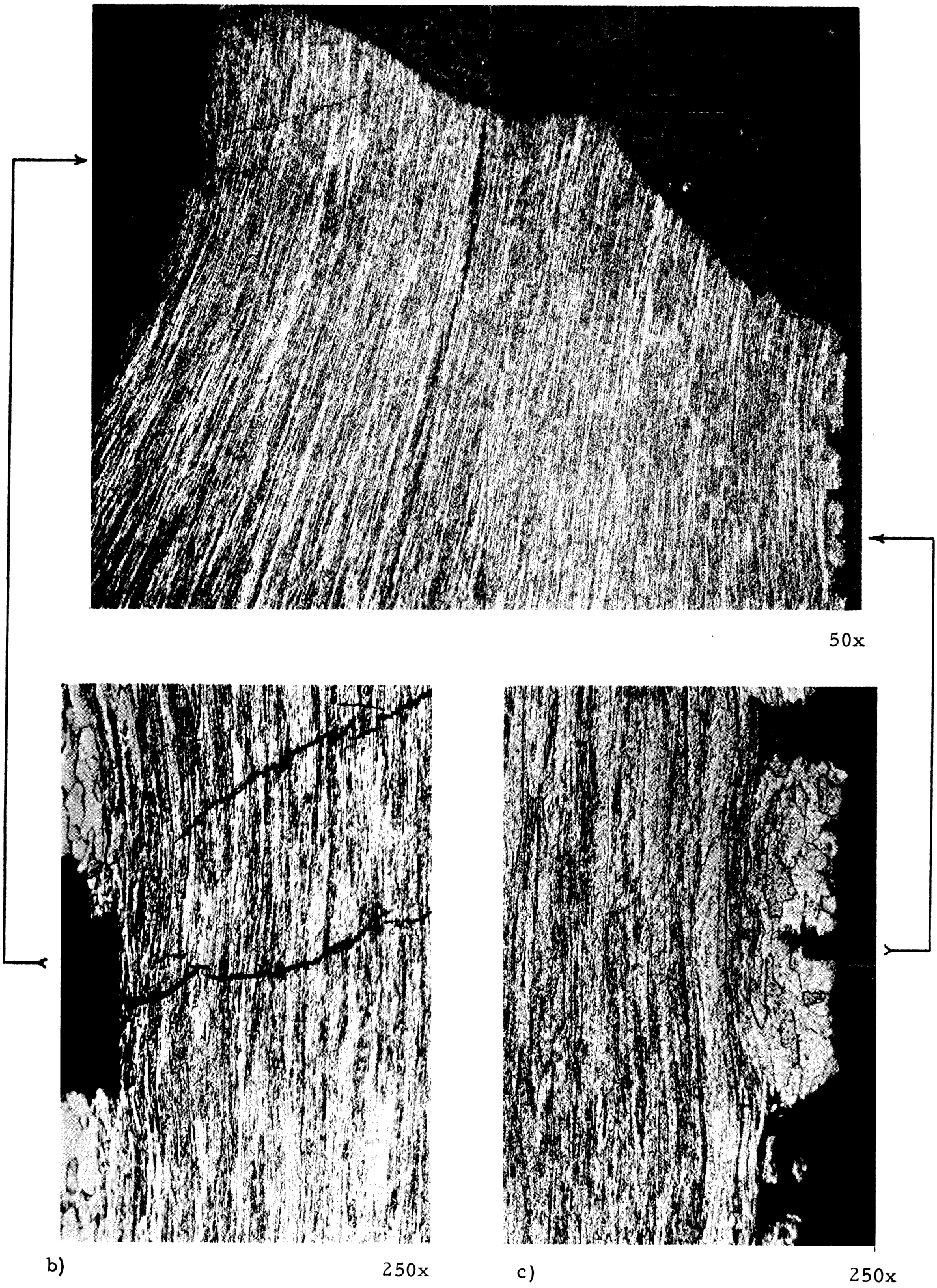
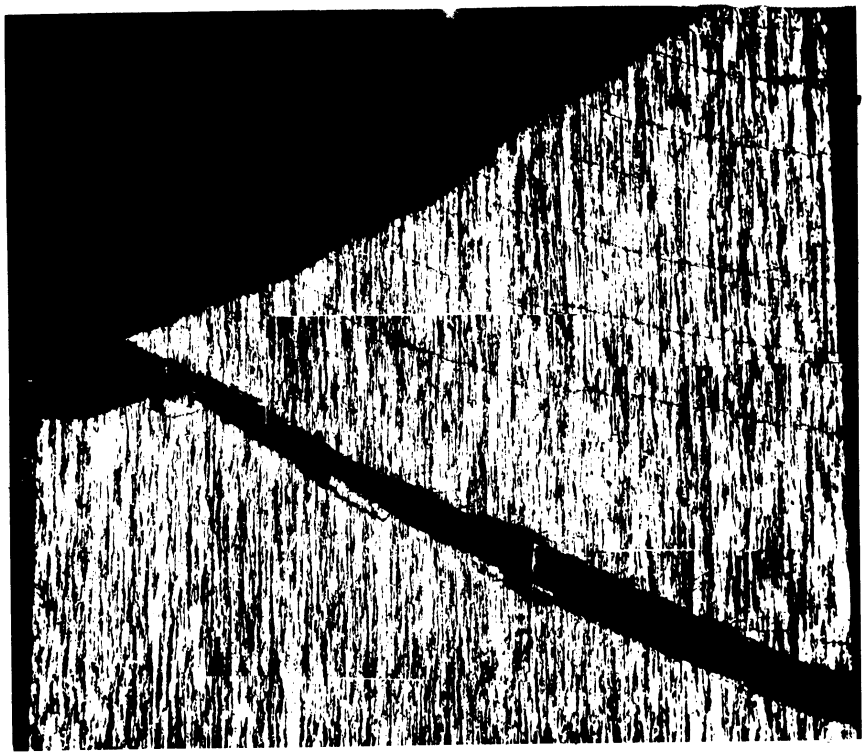
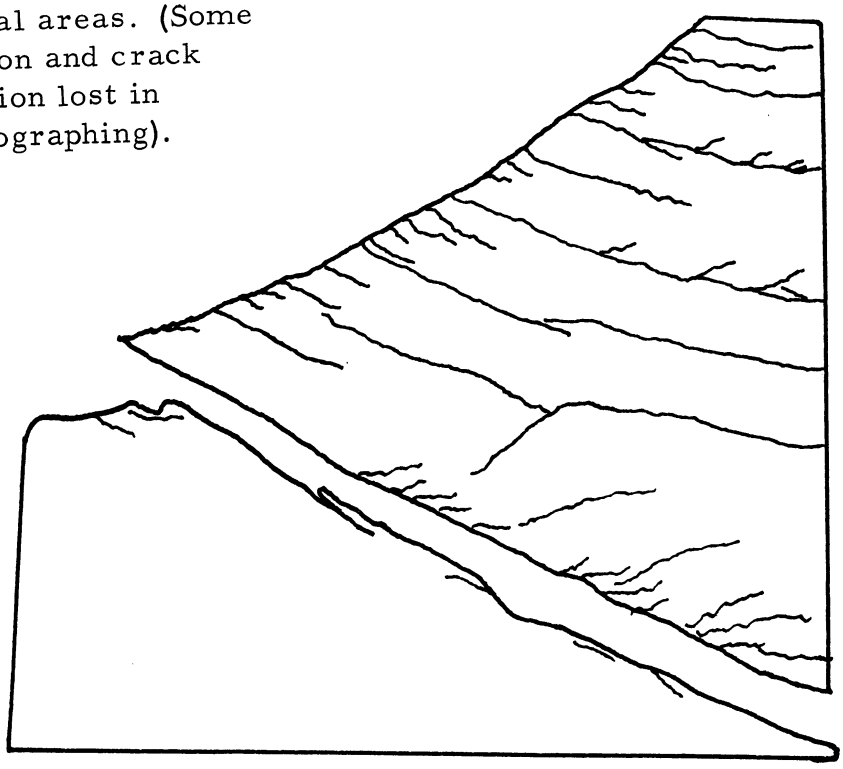


Figure 72 Fracture Area of Specimen M-3 After Creep-Exposure 10 Hours at 1800°F in Vacuum to 2.25-Percent Deformation And Then Tensile Tested at Room Temperature



approx 25x

Re-photographed from composite of 8 50x photomicrographs of individual areas. (Some resolution and crack delineation lost in re-photographing).



Schematic Drawing Showing Location of Cracks In Fracture Area of Specimen M-4

Figure 73

Fracture Area of TZM Specimen M-4 After Creep-Exposure 10 hours at 1800°F in Vacuum to 4.26 Percent Deformation and Then Tensile Tested at Room Temperature