THE UNIVERSITY OF MICHIGAN RESEARCH INSTITUTE
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FIRST PROGRESS REPORT
TO
MATERIALS LABORATORY
WRIGHT AIR DEVELOPMENT CENTER
ON
EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES

by

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Project 02902

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SUMMARY

This report covers progress under Contract AF 33(616)-6462 for the peroid from April 1, 1959 to June 30, 1959 on a study of creep damage to short-time mechanical properties of aircraft structural materials. The materials to be studied include Rene 41 alloy as an example of a complex heat-resistant material of current importance, an 80 Ni-20 Cr alloy as an example of a material of relative structural stability, and (tentatively) a molybdenum plus 0.5 percent titanium alloy as an example of a type of material gaining in importance for future use. The experimental precedure is to define exposure conditions producing creep damage to mechanical properties and then determine the mechanism producing damage.

Work accomplished included the selection of test materials and preparation of test equipment. Limited studies were made of both Rene 41 and Udimet 500 alloys to aid in material selection. Damage to these materials was produced by a creep-exposure conducted at 1700°F. Studies were conducted on the 80 Ni-20 Cr alloy to establish the annealing conditions necessary to produce a fine grain and coarse grain condition for the creep damage studies.

INTRODUCTION

This report, the first progress report to be issued under Air Force Contract No. AF 33(616)-6462, covers the period from April 1, 1959 to June 30, 1959. The research involves the study of the effect of prior creep on the short-time mechanical properties of aircraft strucutral materials with the objectives of the development of methods for the prediction of creep damage to mechanical properties.

Between February 10, 1956 and March 31, 1959, research was conducted at the University under Contract AF 33(616)-3368 to study the effect of creep-exposure on the mechanical properties of typical structural metals in sheet form. The materials studied included 2024-T86 aluminum alloy, C110M and Ti-16V-2.5Al titanium alloys, and 17-7PH stainless steel in the TH1050 and RH 950 conditions. Systematic data were obtained on the effects of up to two percent creep in 100 hours at temperatures in the normal service range of each material, that is 350° to 500°F in the case of the aluminum alloy, and from 600° to 900°F for the other materials. Short-time testing was then carried out at room temperature or the temperatures of creep-exposure. Although a number of examples of damage were found, the research was limited by the necessity of studying established sheet materials. Such materials are relatively immune to damage to short-time mechanical properties by creep. Research to explain the observed effects was very limited because the major objective was to determine the influence of creep-exposure conditions rather than the mechanism of damage itself.

Exposure of a material under creep conditions can result in the alteration of mechanical properties by several means. Creep itself could improve or damage these properties or the exposure to temperature alone can alter properties through thermally induced structural changes. The creep process may also modify the form, rate or degree of a thermally-induced change. Exposure to temperature can also alter mechanical properties through chemical reaction at the surface or by the introduction of contaminants into the material. Furthermore these surface changes may be affected by the creep process.

In the present investigation it is planned to study the damage process by deliberately attempting to produce creep under conditions causing significant alterations of mechanical properties. This may include variations from long-duration, low-creep rate tests under an invariant load to rapid and extensive creep resulting from high temperatures or stresses. The aim will be to induce damage by creep under known conditions that will produce an effect sufficiently large enough to make possible the establishment of the basic mechanisms by metallurgical studies. For the present, it is intended to focus the study on the inter-action of creep and thermally-induced structural changes and to exclude surface damage effects as far as practicable. The evaluation of damage will be made primarily by tensile, compression and impact tests. Material will be tested principally in the form of wrought bar stock. Later tests may include precision investment cast specimens or sheet material.

The primary use of bar stock will permit the introduction of the notched bar impact test as a measure of mechanical property damage. In addition, there is somewhat more freedom in controlling structure in wrought bar stock than in sheet.

The experimental procedure will consist of the following steps:

1. Determination of creep conditions damaging to short-time properties.

2. Determination of the mechanism of damage.

3. Development of a general theory on which creep damage to mechanical properties can be based.

The research is to be conducted in three types of material. The first is a presently used complex heat-resistant alloy subject to marked alteration of mechanical properties during creep. Second, is a material with a simple structure suitable for the study of creep damage itself, relatively free from thermally-induced structural changes. Third, an alloy based on one of the high melting point metals in order to provide an example of a type of material rapidly gaining in importance for future uses.

After preliminary evaluation and consultation with representatives of WADC, the following materials were selected to meet the objectives of the investigation.

1. Complex Heat Resistant Alloy. Rene 41 alloy was selected for this material. This material is a complex nickel base alloy, hardened with titanium and aluminum. It exhibits good properties to temperatures in the range up to 1700°F in wrought, cast, or sheet forms.

2. Simple Structural Alloy. The alloy of 80% Nickel and 20% Chromium was selected to meet the requirements of relative structural stability and simplicity. In addition, this composition is the basis for the family of complex nickel-base alloys.

3. High-Melting Point Alloy. Tentatively selected to be the binary alloy of molybdenum plus 0.5 percent titanium. Specimens would be tested uncoated in vacuum and in air coated with Chrom-alloy W-2 coating.

TEST MATERIALS

Following the selection for study of the complex heat resistant alloy, Rene 41, and the simple structural alloy, 80 Ni-20 Cr, steps were undertaken to procure sufficient stock of these materials for the research program. Stock of the molybdemum-base alloy has not yet been ordered.

Rene 41

Approximately 100 pounds of 1/2-inch diameter wrought bar stock of Rene 41 alloy, all from one heat, have been ordered from the Metallurgical Products Department, General Electric Company. Delivery is expected in the early part of July. The nominal analysis of this alloy is as follows:

Element	Percentage (wt)
Nickel	52.5
Chromium	19.0
Cobalt	11.0
Molybdenum	9.75
Titanium	3.15
Aluminum	1.65
Carbon	0.09
Boron	0.005

The material is shipped in the solutioned condition (1975°F---plus water-quench). Heat treatment is then to be conducted at the University. It is anticipated that the following additional treatment will be used at least in the initial studies of the material: Re-solution treatment one half hour at 1950°F; air cooled; aged 16 hours at 1400°F; air cooled.

80 Ni-20Cr

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. After consultation with representatives of the Hoskins Manufacturing Company, arrangements are being made to obtain approximately 100 pounds of Chromel-AS alloy.

The nominal composition of this alloy follows:

Element	Percentage (wt)
Chromium	20.0
Nickel	78.0
Carbon	0.10
Silicon	1.4
Manganese	0.2

In this composition the carbon content approximates that of the nickel-base super-alloys while the manganese content is low. The high silicon content enhances oxidation-resistance but is said not to cause any precipitation reaction or result in any minor phases.

The material is to be furnished as 1/2-inch diameter hot rolled bar stock. Final annealing will be carried out at the University with the intention of producing both a fine grain and a coarse grain condition. Experiments to aid in establishing these conditions are described in a later section of the report.

SPECIMEN PREPARATION

The test specimens to be used in the major portion of the investigation are designed to be machined from 1/2-inch diameter bar stock. For the creep-exposures and tensile tests a nominal 0.350-inch diameter gauge section specimen will be employed. For tensile tests following creep-exposure, the surface of the specimen will be machined off in order to eliminate surface damage effects. The subsequent compression or impact tests will be conducted on specimens to be machined from the gauge section of the creep specimen. The compression specimen was designed to maintain a 5:1 length to diameter ratio while the impact specimen is the ASTM Type-W sub-sized specimen. Drawings of the various test specimens are included in Figure 1.

TEST EQUIPMENT AND PROCEDURES

Wherever applicable, ASTM Recommended Practices are adhered to in test procedures. Other testing details follow practices developed at the University of Michigan.

In order to test bar stock it was necessary to modify the compression test fixture to accomodate cylindrical specimens. In addition, a special grip was constructed for holding sub-sized impact specimens in the impact testing machine.

Creep tests are conducted in individual creep testing units fitted with wire-wound resistance furnace for heating. The load is applied through a third class lever system. Extension is measured with a modified Martens extension system having a sensitivity of ten millionths of an inch. Since the extension is measured between collars threaded to the specimen shoulders, a correction factor to account for the specimen fillets and shoulders is used in the extension calculations.

All creep specimens are placed in the hot creep furnaces at approximately 50°F below the desired test temperature. The specimens are brought up to the proper test temperature and distribution in a standard four hour holding period prior to application of the load.

Tensile and compression tests are conducted in a Baldwin-Southwork hydraulic tensile machine. Pacing equipment is used to provide a constant strain rate. Extension is measured by a microformer type strain gauge that enables automatic recording of stress-strain curves to be made. The strain gauge is used with a special extensometer bar system built at the University. Compression tests are conducted in a sub-press employing a loading ram actuated by the tensile machine cross-head.

A standard Olsen impact testing machine is available for impact tests.

RESULTS AND DISCUSSION

In order to aid in the selection of a complex heat resistant alloy for extensive testing, preliminary experiments were run on samples of both Rene 41 and Udimet 500 alloys. In addition, some experimentation was carried out to aid in establishing annealing temperatures for the 80 Ni-20Cr alloy. Other experimental work was confined to modification and proving of the test fixtures.

Complex-Heat Resistant Alloys

Prior to the final selection of Rene 41 as the complex heat resistant alloy for study in this investigation, limited preliminary studies were carried out on both Rene 41 and Udimet 500 alloys. The test material employed was from a small supply on hand at the University. The purpose of these studies was to gain a familiarity with this type of material and to ascertain if there were any unforseen experimental difficulties that might cause one material to be favored over the other. It was also desired to see if the type of creep damage desired could actually be produced.

Enough Rene 41 stock in the form of a 3/4-inch thick by 1 1/4-inch wide plate, was available to produce five creep specimens and several impact specimens. The material had been water quenched from 1950°F. Prior to testing it was solution treated one hour at 2150°F, oil quenched, and then aged for four hours at 1650°F and air cooled in accordance with the treatment which was recommended at that time.

This heat treatment produces a high level of rupture strength at temperatures above 1400°F although with some sacrifice of lower temperature yield strength (Ref. 1).

The Udimet 500 was obtained as 5/8-inch diameter bar stock in the solution heated and double aged condition. This treatment, the standard one for this alloy, was as follows:

1. Anneal 2150°F---two hours, air cooled
2. Solution 1975°F---four hours, air cooled
3. Age-----1550°F---24 hours, air cooled
4. Age-----1400°F---16 hours, air cooled

Again, five creep specimens and several impact specimens were prepared for testing.

It was decided to conduct creep-exposures for 100 hours at two extremes of temperature---1200°F and 1700°F. Two specimens were exposed at each temperature at stresses approximating 75 percent of the 100 hour rupture strength of Rene 41. The stresses selected were 75,000 psi at 1200°F and 12,000 psi at 1700°F. Of the two exposed specimens, one was then tensile tested at room temperature and the other machined into an impact specimen. The as-heat treated properties of each material were also determined.

The results of these tests are summarized in Table 1. The time-elongation curves from the creep-exposures are presented in Figure 2. As Figure 2 shows, the amount of creep obtained for each alloy in 100 hours at 1200°F was negligible. A slight shrinkage due to a structural reaction was observed over most of the test period. In the 1700°F tests the creep appeared to be accelerating slightly towards the end of the exposure period. The Rene 41 had somewhat greater creep resistance then did Udimet 500 at 1700°F.

The tensile properties of both materials exhibited changes as a result of the exposure. Generally, the 1200°F exposure resulted in an increase in the yield strength and proportional limit, while the 1700°F exposure caused a decrease in the ultimate tensile strength and ductility. However, the Rene 41 also showed a slight decrease in ultimate tensile strength and elongation from the 1200°F exposure while these properties of the Udimet 500 alloy were apparently unaffected.

The 1200°F behavior probably resulted from a combination of the Bauschinger effect which caused the increase in yield strength and aging from the exposure to temperature. The 1700°F exposure apparently caused a drastic overaging effect. The data are too sparse to indicate if there is any interaction between the creep and thermally-induced changes.

Electron micrographs of the two alloys before and after creep-exposure are presented in Figures 3 through 8. The creep-exposures had a similar effect on the structures of both materials. In the as-treated condition a fine dispersion of gamma prime phase was present in the matrix. (Figures 3 and 6), Exposure at 1200°F caused little, if any, visible change in the structure. (Figures 4 and 7), while the exposure at 1700°F caused considerable growth and agglomeration of the gamma prime phase in the matrix. (Figures 5 and 8) In addition, there was a tendency for agglomeration of gamma prime at the grain boundaries at the expense of the adjacent matrix material. The grain boundary carbides appear to be unchanged.

The impact test results (Table 1) showed extremely low values of strength for all conditions tested. All fractures were of the brittle, crystalline type. The slightly lower impact strengths of the samples exposed at 1700°F are hardly conclusive inasmuch as the materials were notch brittle even in the as-treated condition.

Study of these test results and consultation with representatives of WADC led to the selection of Rene 41 for extensive testing in this investigation.

80 Ni-20Cr

After consultation with the Hoskins Manufacturing Company, it was decided to obtain the test stock of the 80Ni-20Cr alloy in the as-hot rolled form with a recommendation for the final annealing conditions. In this way it would be possible to avoid the cold straightening of the material that would be necessitated by the need to anneal the material in coils in the Hoskins furnaces. In addition it was desired to test material in either two grain sizes and and by annealing the material at the University more flexability would be gained in the preparation of test stock.

The desirability of testing the material in either of two grain sizes was evident from the work of Kozyrskii, et al (Ref. 2) which indicated an appreciable difference in the creep characteristics of an alloy of 80.2% Nickel and 19.8% Chromium when the average grain diameter was changed from 0.02-0.03 mm to 0.5-0.6mm. The coarse grained material exhibited considerably greater creep resistance at 1290°F under a load of 14,200 psi.

For the present investigation it was decided that a variation from coarse grains of a size range from ASTM 1 to 2 to fine grains of a size range from ASTM 7 to 8 would cover the anticipated effect. Accordingly, Hoskins annealed a series of samples for one half hour each at temperatures between 1600°F and 2000°F. The as-hot rolled condition consisted of fairly uniform fine grains (Fig. 9). Difficulty was encountered in producing uniform large grains of the desired size. Particularly noticeable in the sample annealed at 2000°F was a mixture of two widely different grain sizes with the coarse zone existing part way between the surface and the center of the material. (Fig. 10) A similar effect was noted in a sample annealed for one half hour at 1600°F prior to annealing for one half hour at 2000°F. This was probably due to germination caused by a critical amount of residual strain existing as a part of the strain gradient from the surface to the center of the bar. A preliminary sample of the hot rolled stock was then obtained from Hoskins and several samples were annealed at the University. One sample was annealed for an hour at 2100°F (air cooled) and another was annealed for 24.2 hours at 1550°F prior to a one hour anneal at 2100°F. In both samples large grains were observed throughout the cross-section without an excessive gradient in grain size. (Figure 11) Apparently, 2100°F was high enough to overcome the tendency for germination.

The average grain sizes of the various conditions were as follows:

Heat Treatment	Average ASTM Grain Size No.	Average Grain Diameter (mm)
As Hot Rolled	10.4	0.0109
1550°F--24.2 hours	9.5	------
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

The presence of an apparent residual stress gradient in the as-hot rolled bar stock was confirmed by a Knoop microhardness survey. This showed a decrease in hardness from the surface to the center (Figure 12a). Several samples were then annealed for various times at 1550°F. Vickers hardness readings were taken at a point midway between the surface and 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

Knoop hardness surveys were also run on the samples annealed for one, four, and 24.2 hours at 1550°F and plotted in Figure 12 b, c, and d. These data indicate that the annealing treatment for four hours was sufficient to remove the hardness gradient existing between the surface and center of the material although the overall hardness level was first increased slightly by the one hour and four hour anneals and then decreased by the 24.2 hour anneal. This observation agrees with the trend of the Vicker hardnesses and indicates that the material was aging slightly during annealing at 1550°F.

Replicas were made of several samples for examination under the electron microscope. These samples showed a distinct increase in the number of discrete precipitate particles (probably carbides) as the annealing time was increased. A rough estimate of the particle density was made by counting the number of particles in a unit area of replica at a magnification of 2200X. The unit area employed was one grid of the nickel screen in which the replica rested.

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

Electron micographs of these samples are presented in Figures 13 and 14. Figure 14 is a micrograph of an area selected to show a high particle density. These pictures indicate that the particles form at the grain boundaries. The increased hardness appears to be related to the increased particle density.

Based on these observations it would appear desirable to anneal the material for at least four hours at 1550°F prior to the preparation of specimens for the creep-exposures. This will not cause much if any grain growth over the as-hot rolled condition but should remove the existing strain gradient. Inasmuch as the fine grain condition of the material will have an ASTM grain size of about 10, it is felt that the coarse grains of size four produced by the 2100°F anneal will provide a sufficient variation in size for the purposes of this investigation. This would represent a spread of six ASTM numbers, the same desired under the original specifications of ASTM 1-2 and ASTM 7-8.

FUTURE WORK

Work planned for the next three month periods includes the following:

1. Receipt of the Rene 41 and 80 Ni-20 Cr test stock.
2. Heat treatment of the test stock.
3. Initiation of tests to define damaging creep-exposures for these materials.
4. Final selection and procurement of the molybdenum--base alloy.
5. Structural studies of completed exposure specimens.

REFERENCES

1. Jahnke, L. P. and Frank, R. G., "High Temperature Metallurgy Today," Metal Progress, V. 74, No. 6 (Dec. 1958), p. 88.
2. Kozyrskii, G. Ya, Kononenko, V. A., and Okrainets, P. N., "Study of Structural Changes in Nickel-Chromium Alloy During Creep", Izvestia Akad Nauk SSSR, Otd. Tekh.,Nauk, August 1958, No. 8, pp. 90-92 (Henry Brutcher Translation No. 4402).

TABLE 1. EFFECT OF CREEP-EXPOSURE ON MECHANICAL PROPERTIES OF RENE 41 AND UDIMET 500.

	Exposure Conditions								Room Temperature Properties After Exposure					
Material and Specimen No.	Temp. (°F)	Stress (psi)	Time (hours)	Total Load Def. (%)	Plastic Load Def. (%)	Creep Def. (%)	Total Plastic Def. (%)	Total Def. (%)	Ult. Tensile Str. (psi)	0.2% Offset Yield Str. (psi)	Proportional Limit (psi)	Elongation (%)	Reduction of Area(%)	Modulus E (10^6psi)
Rene 41-1	As Heat Treated (a)								>163000 (c)	98400	73000	>15.9	>16.4	29.9
Rene 41-2	1200	75000	100.0	0.34	nil	-0.01	-0.01	0.33	150000	114000	104000	9.5	12.7	28.0
Rene 41-4	1700	12000	100.0	0.06	nil	0.11	0.11	0.17	114400	102800	62500	1.6	1.6	24.4
Udimet 500-1	As Heat Treated (b)								168000	121000	90000	6.8	7.9	29.7
Udimet 500-2	1200	75000	100.0	0.29	0.01	0.003	0.013	0.293	170500 (d)	131000	120000	6.7	6.8	30.9
Udimet 500-4	1700	12000	100.0	0.06	nil	0.56	0.56	0.61	104000	98600	79300	0.8	2.3	30.4
									Izod Impact Strength (ft.lb.)					
Rene 41	As Heat Treated								1					
Rene 41-3	1200	75000	100.0	0.36	0.05	-0.01	0.04	0.35	1					
Rene 41-5	1700	12000	100.0	0.06	nil	0.22	0.22	0.28	0					
Udimet 500	As Heat Treated								1					
Udimet 500-3	1200	75000	18.5						<1					
Udimet 500-5	1700	12000	100.0	0.06	nil	0.30	0.30	0.36	<1					

< Less than

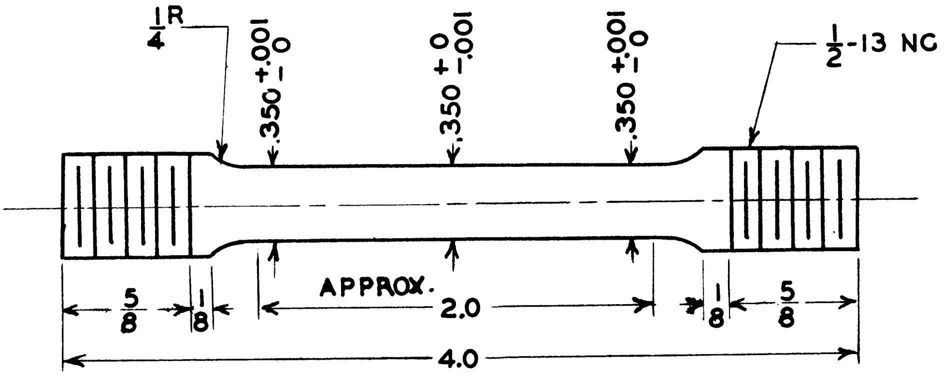
> Greater than

(a) 2150°F--1hr + Oil Quench; 1650°F--4 hr + Air Cool

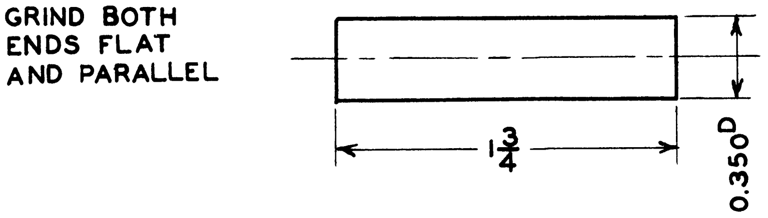
(b) 2150°F--2 hr + Air Cool; 1975°F--4 hr + Air Cool; 1550°F--24 hr + Air Cool; 1400°F--16 hr + Air Cool

(c) Broke in threads; elongation measurements made on unbroken gauge section

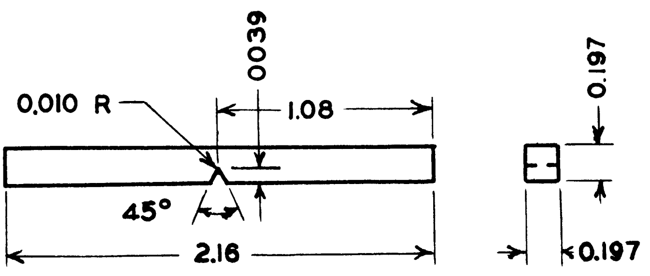
(d) Broke at fillet



TENSILE AND CREEP SPECIMEN



COMPRESSION SPECIMEN



TYPE-W IMPACT SPECIMEN

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

FIGURE 1. Details of Test Specimens

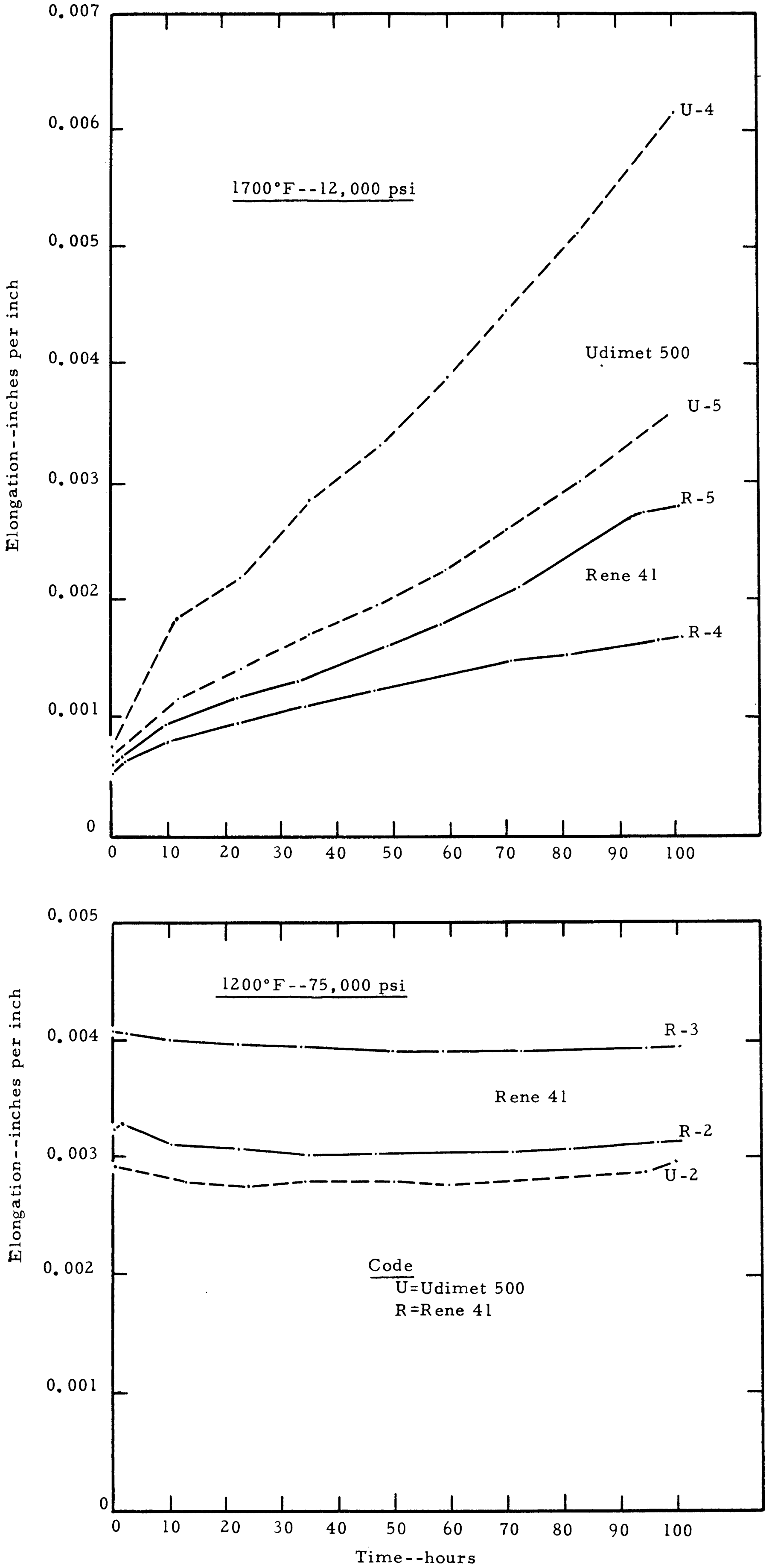


FIGURE 2: Time-Elongation Curves for Creep-Exposures of Rene 41 and Udimet 500 at 1200°F or 1700°F

Electron Micrograph

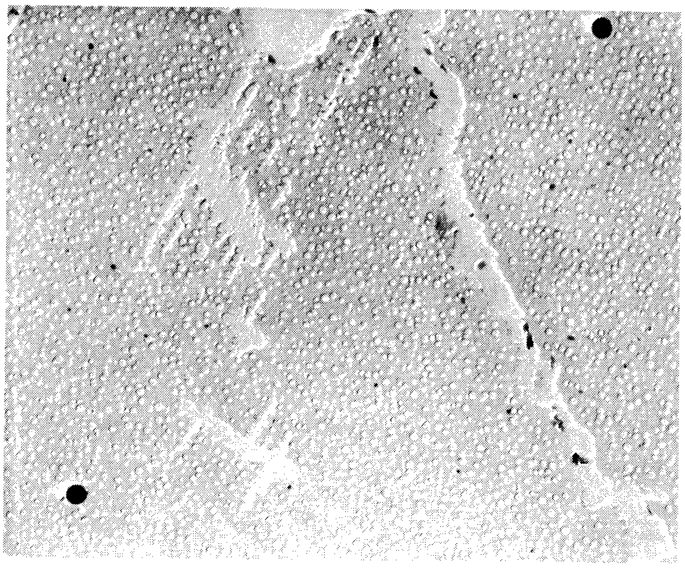


Figure 3. Rene 41--As Heat Treated (2150°F-1 hr + Oil Quench 1650°F-4 hr + Air Cool)

G-etch
8200 X

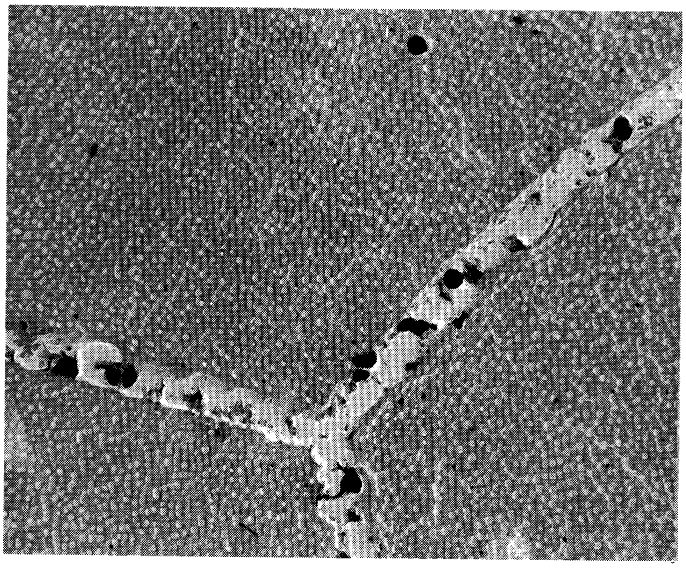


Figure 4. Rene 41--Exposed 100 hours at 1200°F and 75,000 psi

G-etch
8200 X

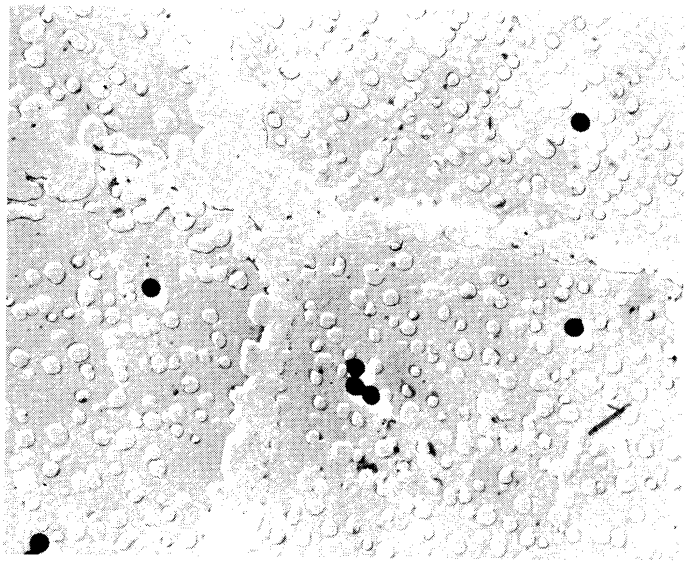


Figure 5. Rene 41--Exposed 100 hours at 1700°F and 12,000 psi

G-etch
8200 X

Electron Micrographs

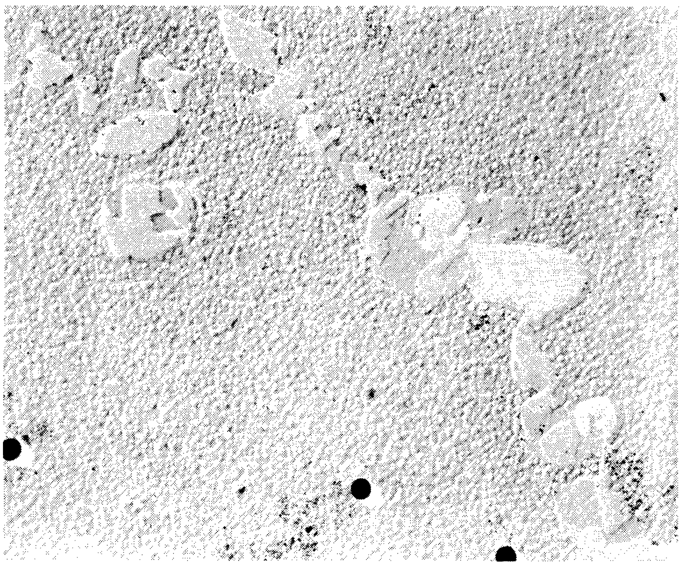


Figure 6. Udimet 500-As Heat Treated
(2150°F-2 hr + Air Cool
1975°F-4 hr + Air Cool
1550°F-24 hr + Air Cool
1400°F-16 hr + Air Cool

G-etch
8200 X

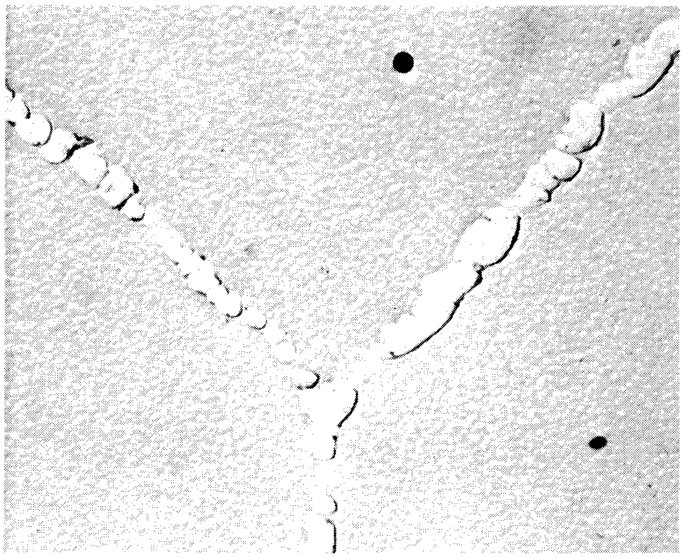


Figure 7. Udimet 500--Exposed 100 hours at 1200°F and 75,000 psi

G-etch
8200 X

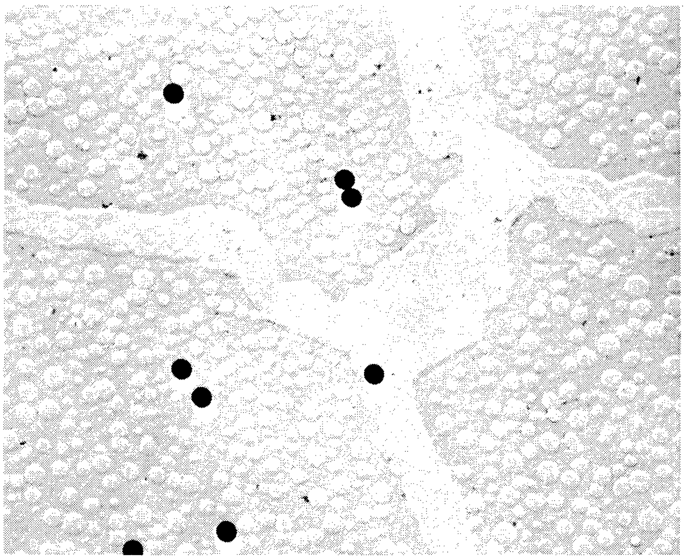


Figure 8. Udimet 500--Exposed 100 hours at 1700°F and 12,000 psi

G- etch
8200

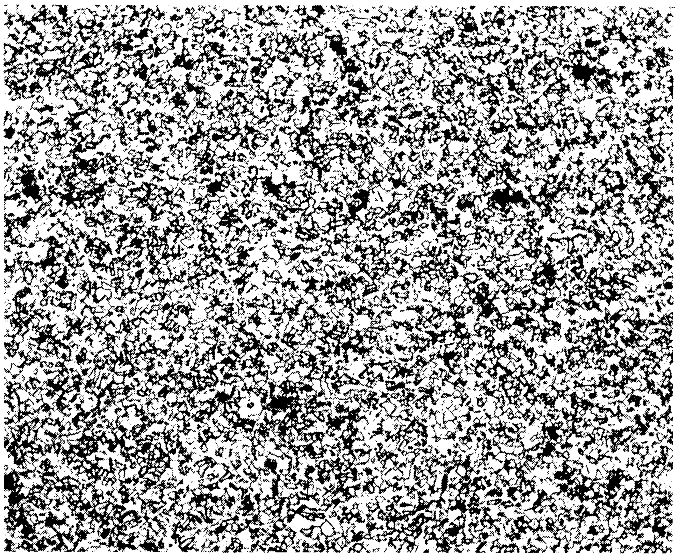


Figure 9. Chromel-AS
As Hot Rolled

Marble's Reagent
100 X

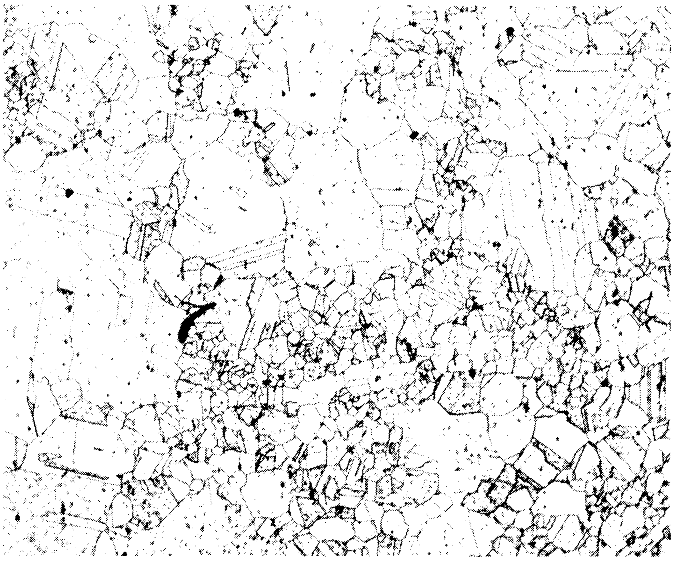


Figure 10. Chromel-AS
2000°F--1/2 hr + Air Cool

Marble's Reagent
100 X

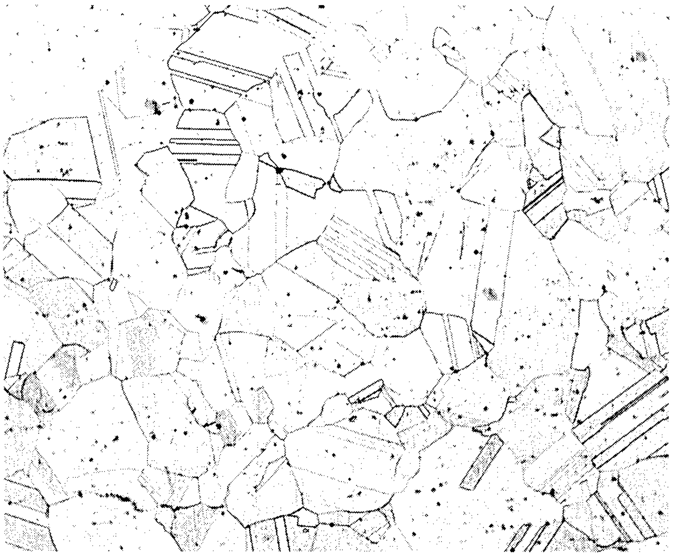


Figure 11. Chromel-AS
2100°F--1 hr + Air Cool

Marble's Reagent
100 X

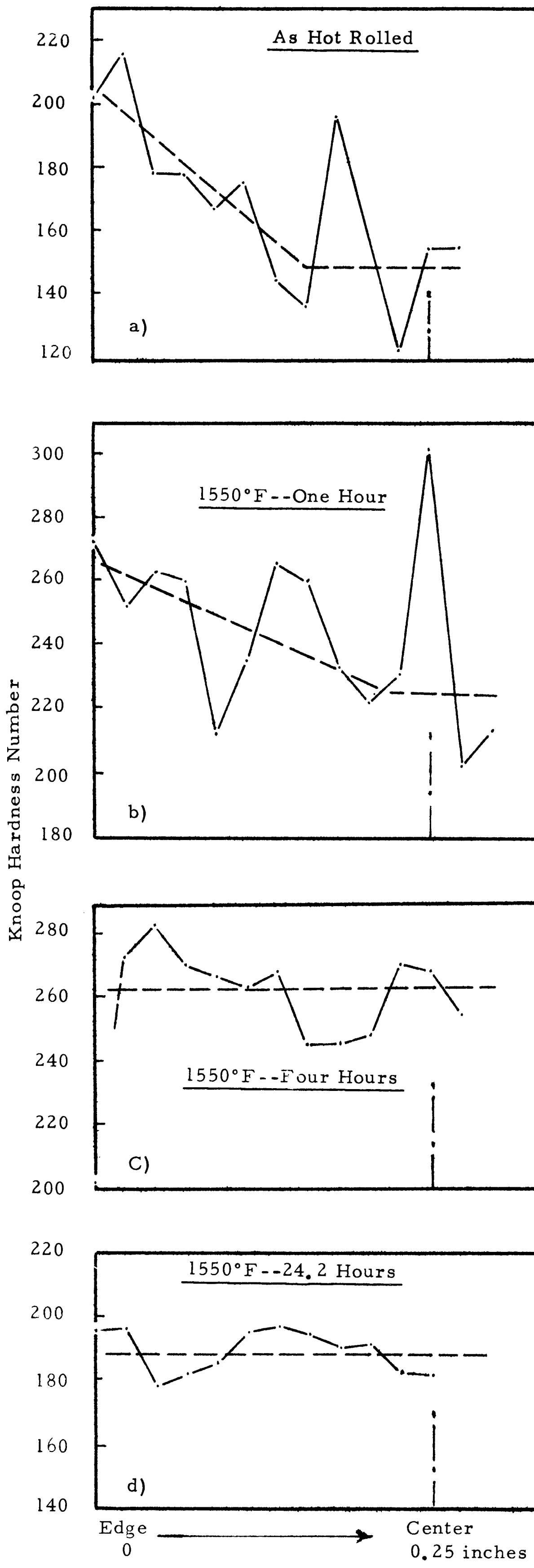


FIGURE 12. Knoop Micro Hardness Surveys of Annealed Chromel-AS

Electron Micrographs

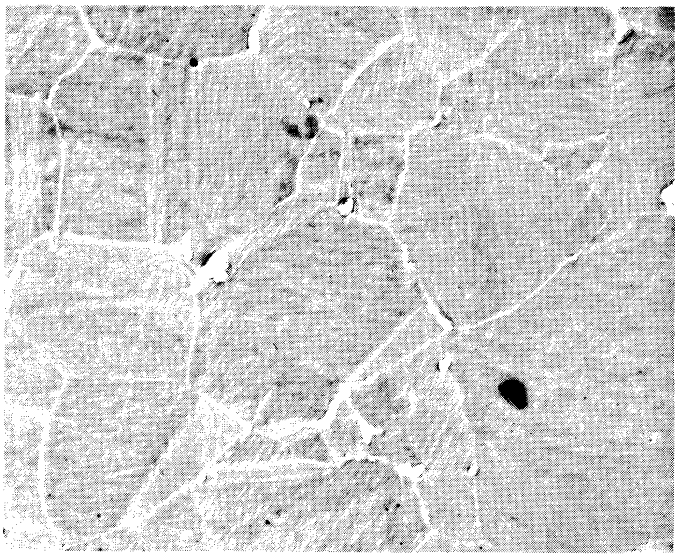


Figure 13. Chromel-AS
As Hot Rolled

Marble's Reagent
3500 X

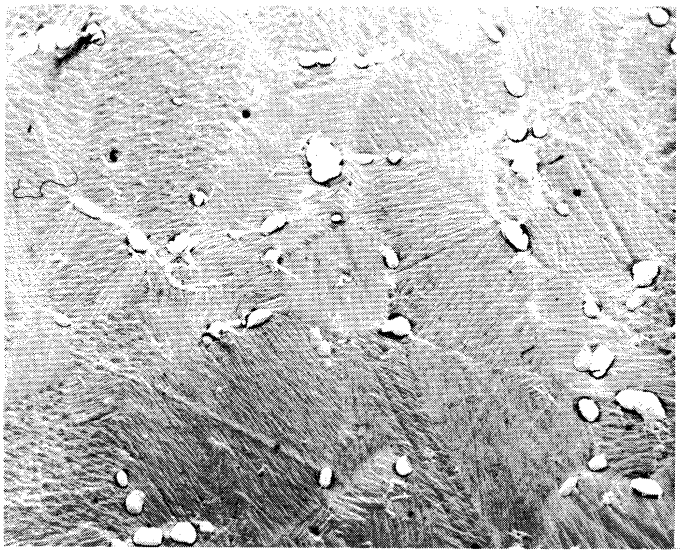


Figure 14. Chromel-AS
1550°F--four hour + Air Cool

Marble's Reagent
3500 X