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FINAL REPORT

DEVELOPMENT OF ADVANCED HIGH STRENGTH TANTALUM BASE ALLOYS

PHASE III – INFLUENCE OF METALLURGICAL CONDITION ON THE MECHANICAL PROPERTIES OF ASTAR-811C SHEET

BY

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16. Abstract

Metallurgical condition was shown to have a significant effect on the creep properties of ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C) sheet. As cold worked (33% Red) material exhibited creep rates 30 times higher than solution annealed (≥ 1/2 hour at 3600°F) material and 10 times greater than for recrystallized (1 hr. at 3000°F) material. Both grain size and the carbide morphology changed as the final annealing temperature was raised from 3000°F to 3600°F. However, the lowest creep rates were achieved for material which retained the high temperature form of the Ta_2C precipitate. Samples with GTA weldments had essentially identical properties as recrystallized base metal. Cooling rates from 3600°F of 5, 50, and 800 F°/min. had little effect on the 2000 and 2400°F creep behavior of ASTAR-811C.

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FOREWORD

The work described in this report was performed by the Westinghouse Electric Corporation, Astronuclear Division, during the period October, 1967 through July, 1970. Technical administration at the Astronuclear Laboratory was under the direction of Mr. R. W. Buckman while Mr. P. Moorhead served as the NASA Project Manager.

Astronuclear Laboratory

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1.0 SUMMARY

The purpose of this investigation was to delineate the effects of metallurgical conditions on the mechanical properties of ASTAR-811C (Ta-8W-1RE-0.7Hf-0.025C) sheet (0.035 inch thick). Under NAS 3-2542 the 2400°F creep properties were shown to be improved by increasing the final annealing temperature from 3000°F to 3600°F.

Cold worked ASTAR-811C (0.035 inch thick) sheet annealed for 5, 30 and 60 minutes at 3000 to 3800°F recrystallized to give an equiaxed grain size ranging from 0.03 to .38 mm. Differential temperature-stress creep testing showed that there was essentially no difference in creep properties for material annealed above the carbon solvus (~3600°F) even though the grain size varied by a factor of 5. The creep rates of ASTAR-811C annealed above the carbon solvus (≥3600°F) were significantly less (~1/5) than for material annealed below the carbon solvus. The creep rates observed for material annealed below the carbon solvus exhibited too much scatter to clearly define a trend. Since both grain size and precipitate distribution were altered over the range of heat treatment investigated, it would appear that the changes in creep behavior were caused primarily by changes on the precipitate morphology. Creep tests on material heat treated to retain the high temperature form of the carbide precipitate in a relatively fine grain size matrix (~0.03 mm) confirmed this observation.

The minimum annealing treatment necessary to produce the high temperature form of the Ta_2C precipitate is 1/2 hour at 3600°F. This annealing treatment was used as both a final annealing treatment as well as for in process annealing. This treatment resulted in optimum creep properties in ASTAR-811C over the temperature range of 2000-2600°F.

Cooling from the final annealing temperature of 3600°F at rates of 5, 50, and approximately 700 F°/minutes had little effect on creep properties at 2000 and 2400°F. Metallographic analysis, both optical and TEM,* indicated that the strengthening of the carbide precipitate must

*Transmission Electron Microscopy

be related to grain boundary precipitate. There was insufficient grain volume precipitate observed by TEM to account for the strengthening increments attributable to the carbide dispersion.

The creep properties of GTA welded ASTAR-811C was similar to that for the base metal. During GTA welding, the fusion zone exceeds the carbon solvus and cools sufficiently fast to retain the high temperature form of the carbide. The grain size in the fusion zone varied from 0.03 to 0.07 mm.

2.0 INTRODUCTION

During the development of ASTAR-811C under Contract NAS 3-2542, it was observed that the creep properties of ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C) could be substantially altered by increasing the final annealing temperature from 3000 to 3600°F[1]. Since ASTAR-811C was developed primarily for long time service applications in advanced space nuclear power systems, creep strength is the primary design criterion. Creep of metals and alloys is a structure sensitive property[2]; therefore, it was of importance to investigate structural effects on the creep behavior of ASTAR-811C. The primary objective of the investigation described in this report was to delineate the influence of metallurgical condition on the creep behavior of ASTAR-811C sheet (0.035 inch thick). Creep properties were determined on material in the as-worked, annealed, and the GTA welded condition.

3.0 EXPERIMENTAL PROGRAM AND PROCEDURES

3.1 EXPERIMENTAL PROGRAM

The experimental program for determining the effects of metallurgical condition on the creep behavior of ASTAR-811C was conducted sequentially following the four tasks outlined in Figures 1-4.

3.2 STARTING MATERIAL

Two heats of ASTAR-811C were required to conduct the program and the ingot analysis for the material is listed in Table 1. With the exception of the specimens used for the thermal mechanical processing study discussed in Section 4.3 of this report, all test specimens were from the NASV-20 heat of ASTAR-811C.

Heat NASV-20 was prepared from sandwich-type electrodes which were double consumable electrode vacuum melted to yield an 80 pound, 4 inch diameter ingot.[1] A one-inch high right circular section of the ingot was upset forged at 2550°F by a single blow on a Dynapak to give a thickness reduction of 59 per cent. After annealing one hour at 3000°F, the forged disc was rolled first at 900°F and then at room temperature to give a combined 89 per cent thickness reduction. Following another anneal for one hour at 3100°F, the material was cold rolled 33 per cent at room temperature to give a finished sheet thickness of 0.035 inch from which test specimens were prepared.

3.3 HEAT TREATMENTS

Heat treatments were done using a cold wall vacuum furnace at $\leq 1 \times 10^{-5}$ torr. At temperatures up to 3200°F, an elastomer sealed polymer pumped system was utilized for heat treatment. Above 3200°F, decarburization of the 0.035 inch ASTAR-811C occurred very rapidly (see Figure 5) when heat treated in a conventional elastomer sealed, polymer pumped, unbaked

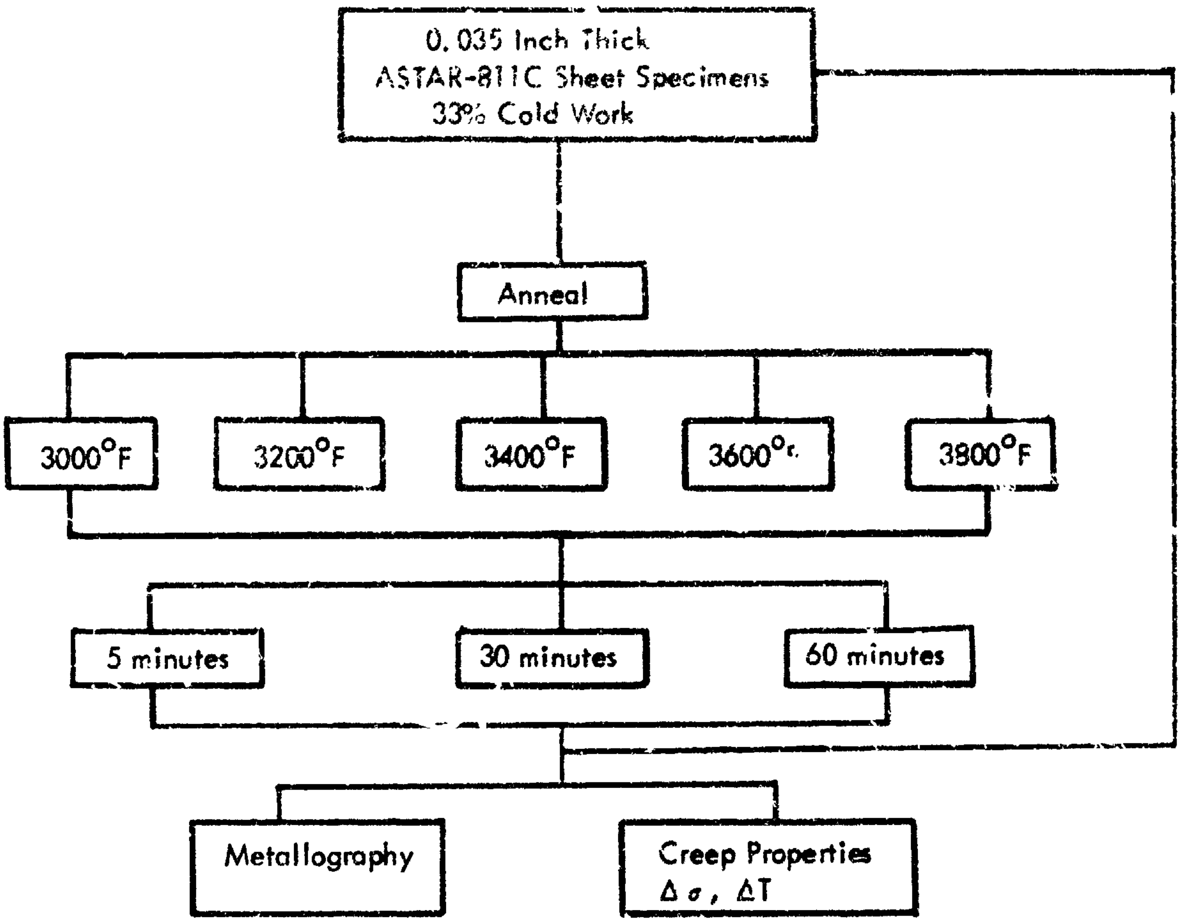


Figure 1. TASK I - Effect of Thermal Treatment on Grain Size and Creep Strength of ASTAR-811C

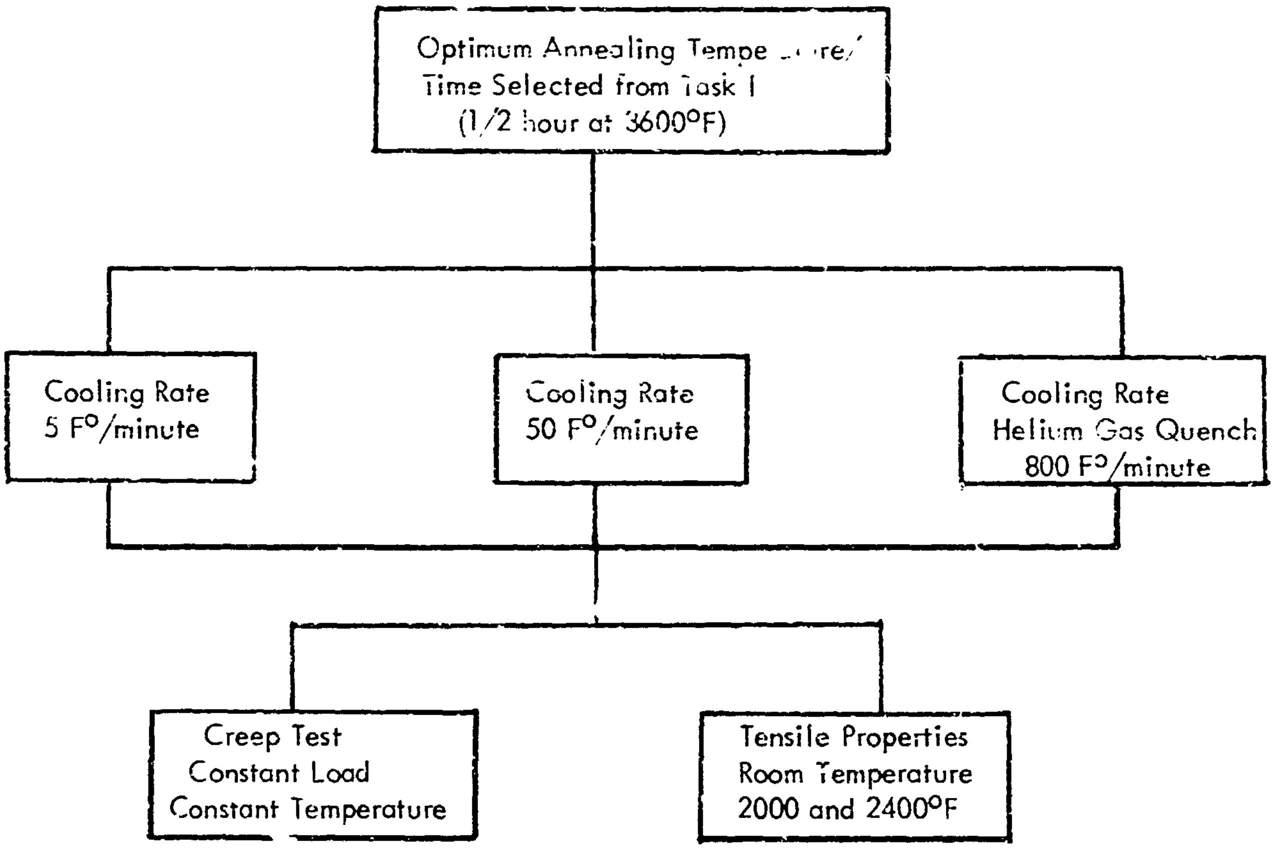


Figure 2. TASK II - Effect of Cooling Rate on Mechanical Properties of ASTAR-811C

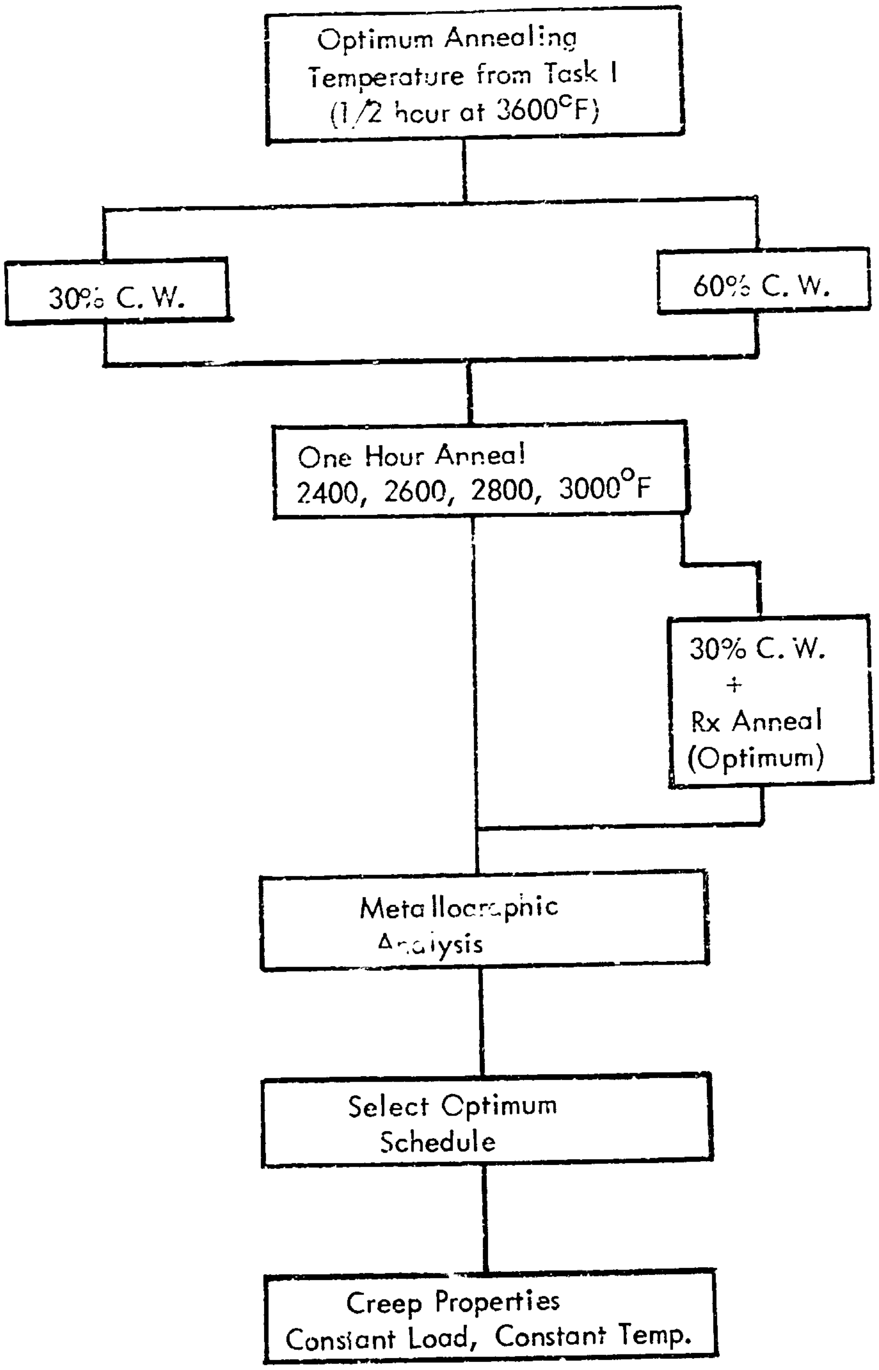


Figure 3. TASK III - Effect of Thermal Mechanical Processing on Creep Properties of ASTAR-811C

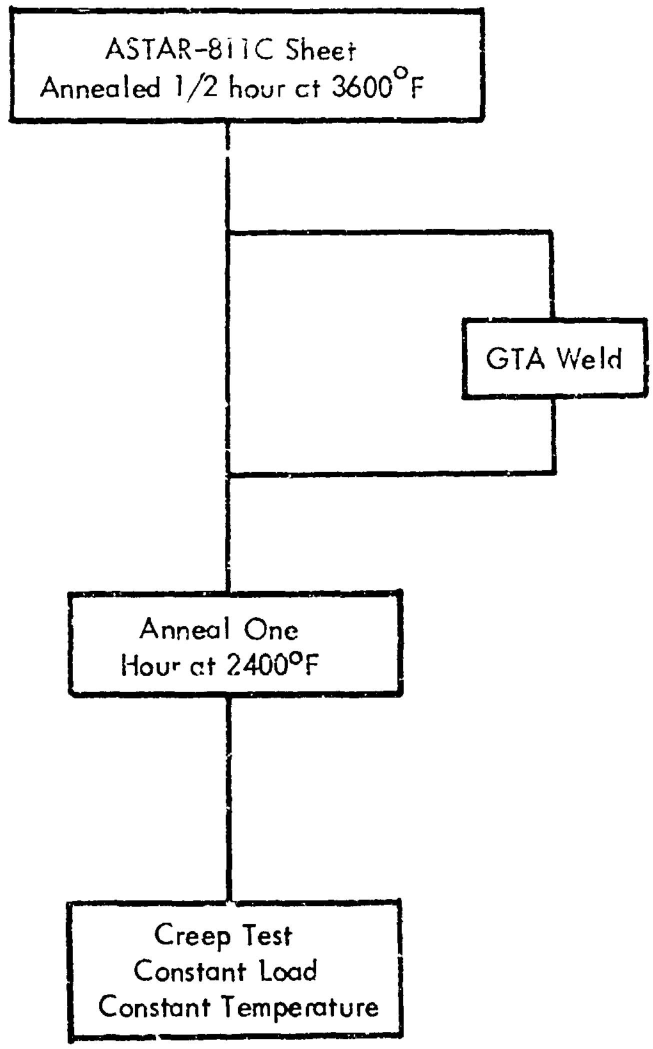


Figure 4. TASK IV - Effect of GTA Welding on Creep Properties of ASTAR-811C

Table 1. Composition of ASTAR-811C Material

Heat No.	Sample Location	W	Re	Hf	C	O	N
NASV-20*	Ingot Top	7.4	1.04	0.71	0.024	0.0006	0.0013
	Ingot Bottom	7.2	0.92	1.01	0.023	0.0021	0.0027
WC-650078	**	8.0	1.4	0.9	0.0235	0.0050	<0.0010

* Original development heat produced under Contract NAS 3-2542 - see Reference 1.

** Vendor reported analysis for 1/8 in. thick plate. Material produced from 8 in. diameter ingot by Wah Chang, Albany, Oregon.

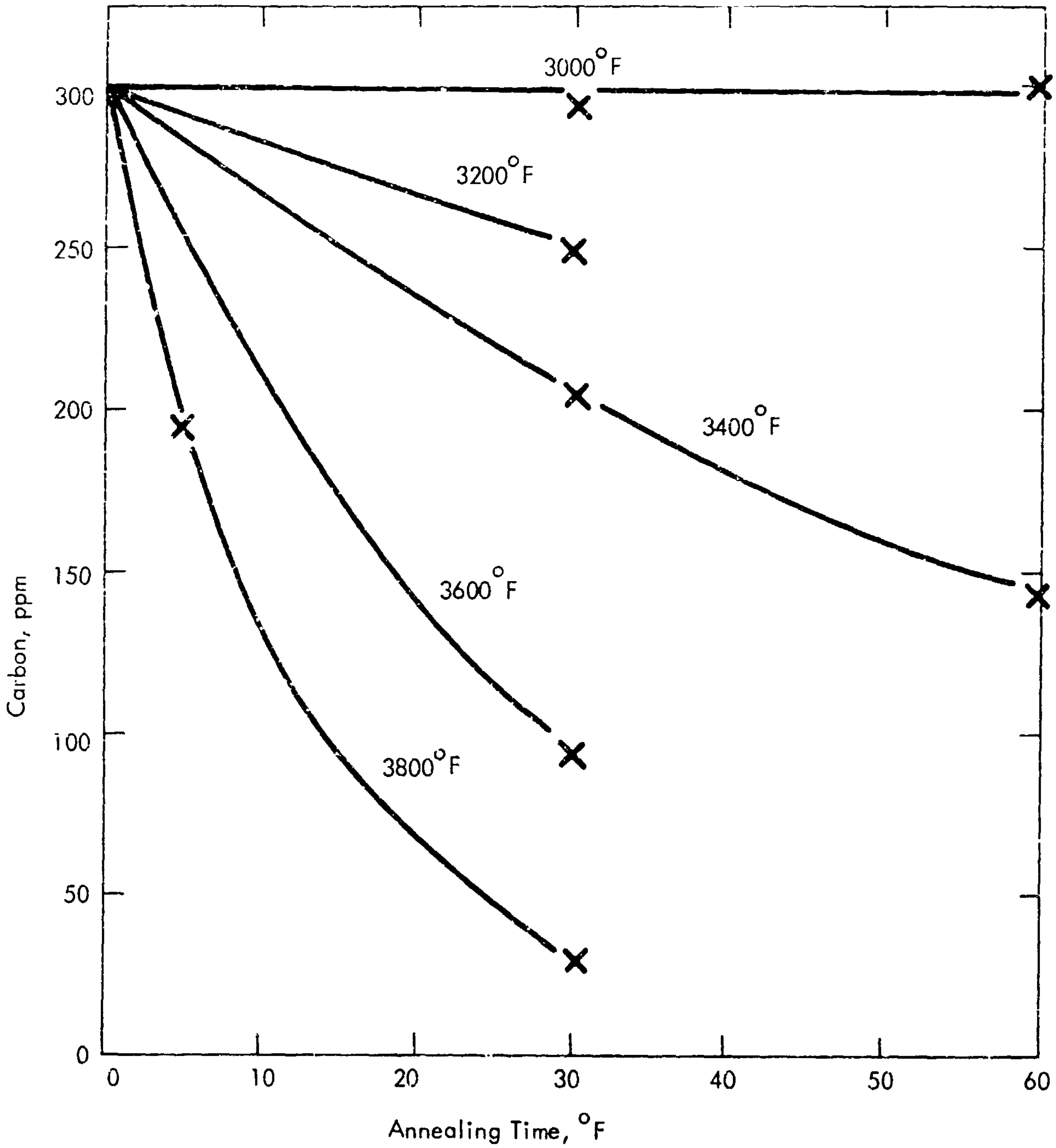


Figure 5. Effect of Annealing Temperature and Time on the Decarburization of 0.035 inch thick ASTAR-811C Sheet

(Note elastomer sealed polymer, pumped nonbaked cold wall vacuum system operating at 1 x 10^{-6} to 1 x 10^{-5} torr) vacuum system. To avoid decarburization, specimens were either annealed in sputter ion pumped bakeable metal sealed systems, or the samples were encapsulated in a T-222 foil envelope which was evacuated to $\leq 1 \times 10^{-5}$ torr and sealed by electron beam welding. Specimens encapsulated in this fashion did not decarburize even when heated at 10^{-5} torr at temperatures up to and including 3800°F.

3.4 MECHANICAL PROPERTIES

Creep and tensile test specimens were of the pin loaded type having a one inch gage length and 1/4 inch width in the gage section. All creep testing was done in sputter ion pumped systems operating at $\leq 1 \times 10^{-8}$ torr. Creep elongation was measured optically using lines scribed at the extremes of the reduced gage section for reference marks. Heating was by radiation from a split wall tantalum resistance heated element. A uniaxial stress was applied by dead weight loading. Details of the ultra-high vacuum creep test units have been described elsewhere[(3)].

Tensile testing was conducted in an elastomer sealed vacuum chamber fitted to a 500 lb. capacity screw driven Instron testing frame. Crosshead motion was used to record specimen elongation during testing. All elevated temperature tensile testing was done at a pressure of $\leq 1 \times 10^{-5}$ torr.

4.0 EXPERIMENTAL RESULTS AND DISCUSSION

4.1 EFFECT OF THERMAL TREATMENT ON GRAIN SIZE AND CREEP PROPERTIES

4.1.1 Grain Size

The effect of annealing conditions (temperature and time) on the grain size of ASTAR-811C was determined using heat NASV-20 (35 mil sheet, 33% cold worked). Specimens were annealed for times of 5, 30, and 60 minutes at temperatures ranging from 3000 to 3800°F in 200F° increments.

The as-machined specimens were pickled and wrapped in clean tantalum foil. Tungsten-rhenium thermocouples were spot welded to the tantalum foil and the temperatures measured using the calibration data supplied by the Hoskins Manufacturing Company. The specimens were heated in vacuum to 2000°F and held there for outgassing until the pressure dropped to $< 1 \times 10^{-5}$ torr. This took about 10-15 minutes. The specimens were then heated to the annealing temperature as rapidly as possible. The heating time varied from about 30 to 60 seconds. Cooling was done in helium gas with the time to cooldown to 2000°F varying from about 45 to 60 seconds. By way of comparison, it takes about 50% longer to cool in vacuum.

The grain size results are shown in Figure 6, and the following points are noted:

(a) The grain size was measured on photomicrographs at 100X by scribing four lines which ran from corner to corner and mid-face to mid-face. Measurements were made on both longitudinal (L) and transverse (T) sections and occasionally in the rolling plane (RP).

(b) The points plotted at zero time refer to the starting, as-worked material.

(c) After recrystallization, no difference was noted in the grain size as measured on the L, T, and RP sections.

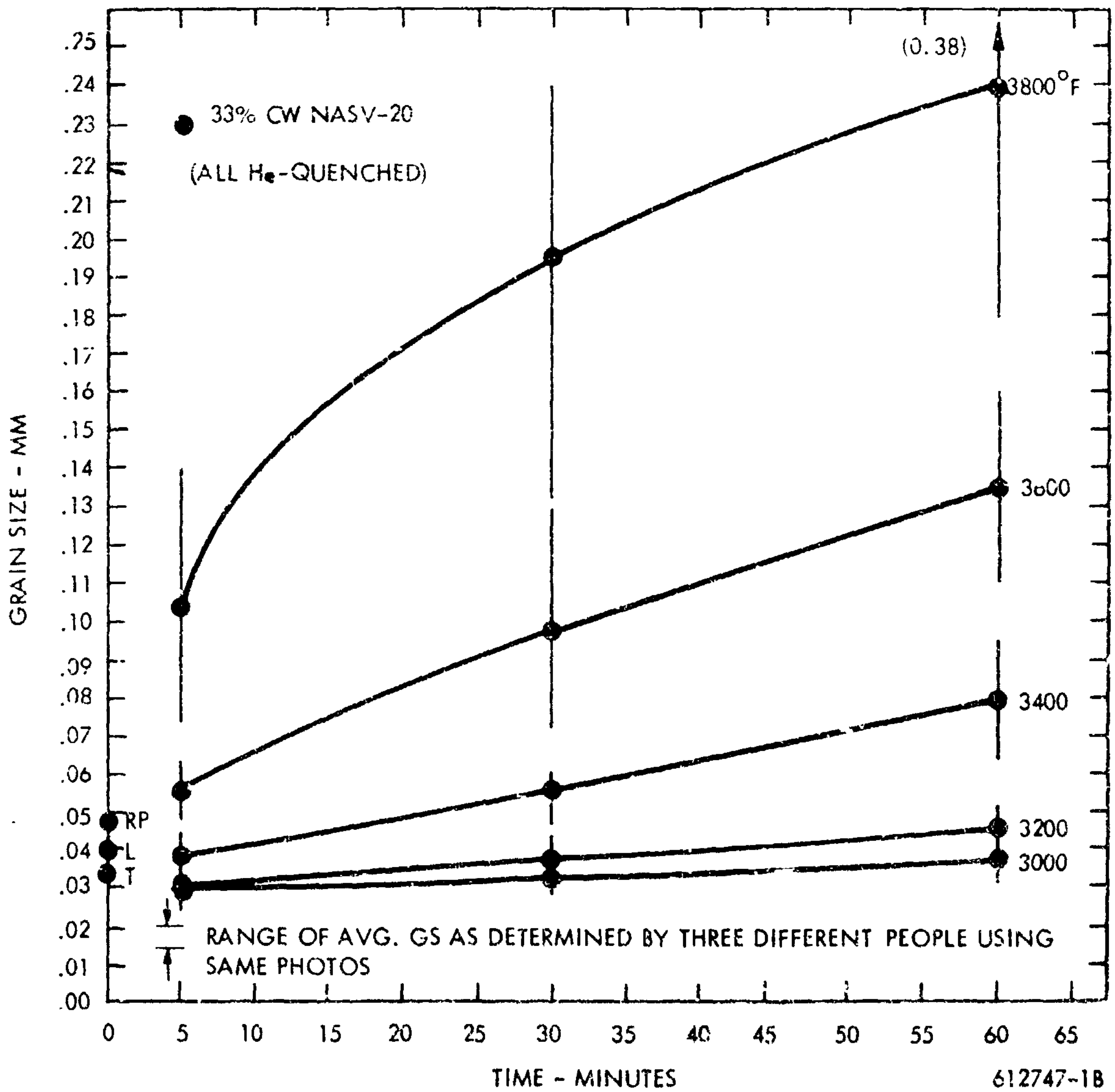


Figure 6. Effect of Annealing Time and Temperature on Grain Size of ASTAR-811C Sheet, 0.035 Inch Thick, 33% Cold Work

(d) The circular points give the average grain size, i.e., the average of at least eight values, four for each micrograph for both L and T sections. The vertical bands depict the range of all the individual values. Note that at the larger grain sizes there are only three or four grains across the thickness of the sheet. It is also to be noted that the height of the vertical bands does not necessarily give a correct measure of the inherent variation in grain size since it also reflects variation due to the particular measurement procedures employed.

The average values shown in Figure 6 are replotted in Figure 7a and compared with previous data on ASTAR-811C in Figure 7-b, c, d.

4.1.2 Creep Behavior

Incremental type creep tests were employed in which the stress and temperature were progressively changed, beginning generally at 2200°F and 25 ksi and ending after about 300 hours at 2600°F and stresses as low as 5 ksi. An example of the type of creep curves obtained is shown in Figure 8.*

The specimens all showed between 0.1 and 0.2 per cent strain on loading to a stress level of 25 ksi at 2200°F. Most of the segments of the creep curves were linear. Where nonlinear segments were observed, the minimum creep rate is reported. The applied stresses were based upon the initial cross-sectional area. Thus, the true stress on the specimen during the later stages of test are higher than the values reported by up to about 5 per cent. The reported creep rates have not been corrected for this effect, except for the following described test.

* Complete creep curves for each test are in Appendix A.

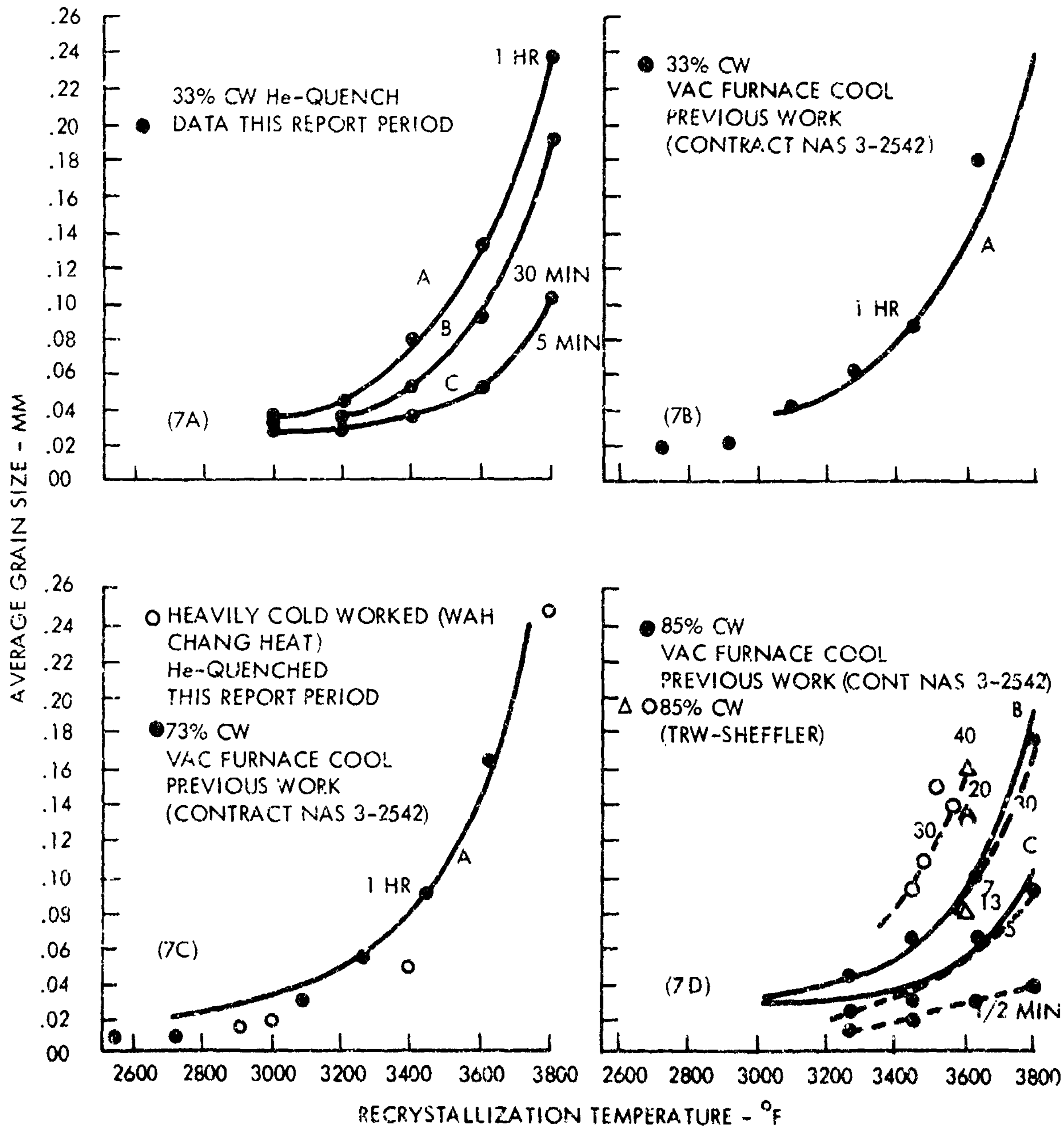


Figure 7. Grain Size-Temperature-Time Data on ASTAR-811C Alloy
(Heat No. NASV-20 except as noted)

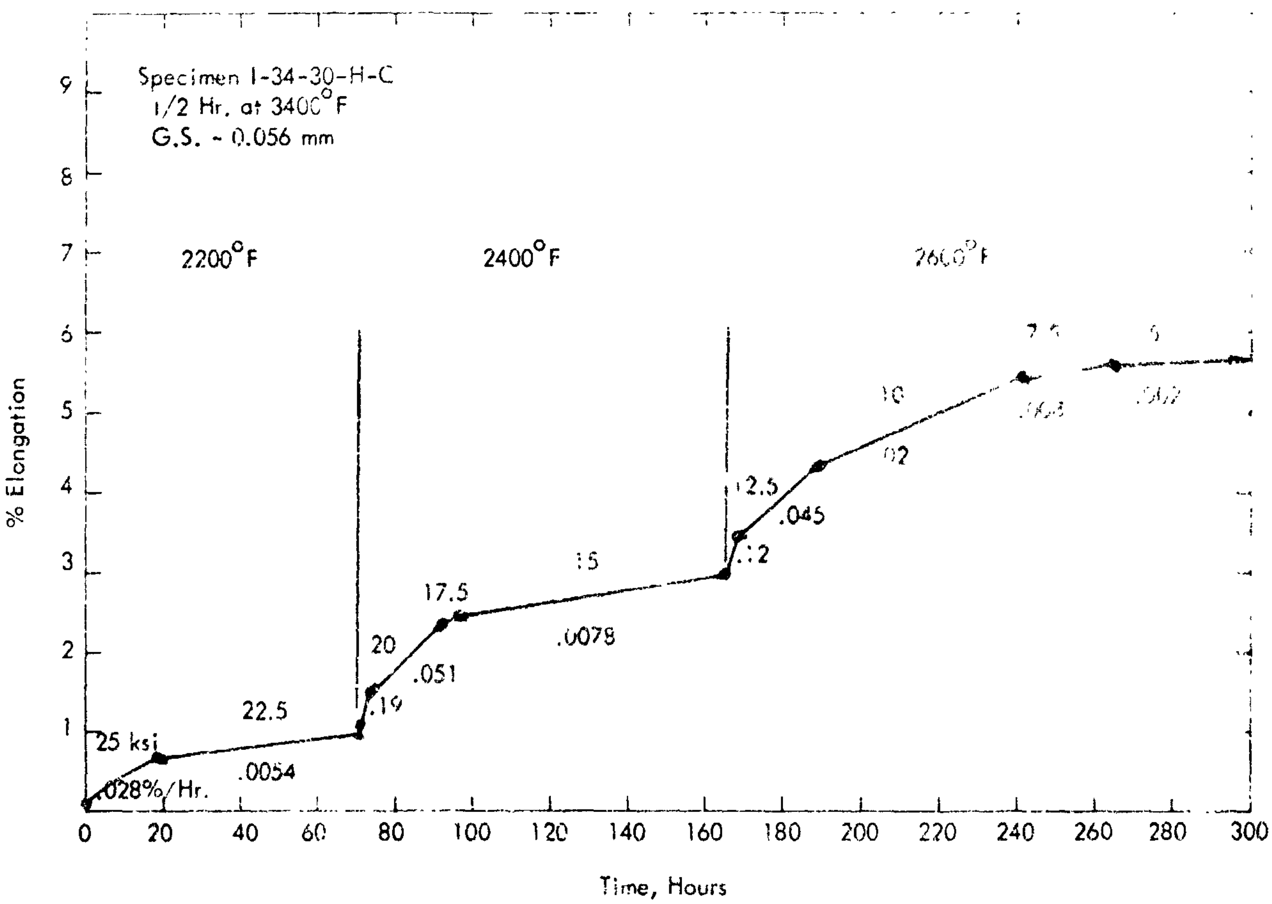


Figure 8. Creep Behavior of ASTAR-811C During Incremental Stress and Temperature Testing. (Specimen 1-34-30-H-C; prior treatment, 1/2 hour at 3400°F; grain size, 0.056 millimeter.)

To provide some measure of the effect of the prior creep strain on the creep rate at a given instant in the latter part of the test, a test like that shown in Figure 8 was run and then the sequence was reversed. The creep rates for the up-cycle and down-cycle were compared after correcting for the true stress effect; and as shown in Figure 9, they are in good agreement.

The effect of annealing temperature on the creep behavior of ASTAR-811C is summarized in the Larson-Miller plot shown in Figure 10. The times to 1 per cent strain values were calculated from the creep rate data obtained during the incremental temperature-stress tests. It is apparent that recrystallized ASTAR-811C has significantly better creep properties than the cold worked material. Increasing the final annealing temperature results in still further improvement in creep properties. The change is gradual though, with specimens annealed at 3400°F and below showing considerable data scatter, while there is little if any difference between the specimens annealed at 3600° and 3800°F. The spread in the band for the annealed specimens amounts to a vertical separation of about 5 ksi, or a horizontal separation of about 150°F in test temperature. (A change by one unit in the Larson-Miller parameter in Figure 10 is equivalent to a change in test temperature of about 50 F°.)

In Figure 11 the creep rate is plotted against stress for each test temperature. The circular symbols in this figure are for specimens annealed between 3000 and 3400°F, while the triangles represent specimens annealed at 3600 and 3800°F, the annealing time being 5, 30, and 60 minutes at each temperature. The trend of decreasing creep rate with increasing annealing temperature is again evident.

The solid-line and dashed-line curves in Figure 11 are plots of equations (1) and (2), respectively:

$$\dot{\epsilon} = A e^{B\sigma} e^{-Q/RT} \qquad (1)$$

$$\dot{\epsilon} = A \sigma^{n} e^{-Q/RT} \qquad (2)$$

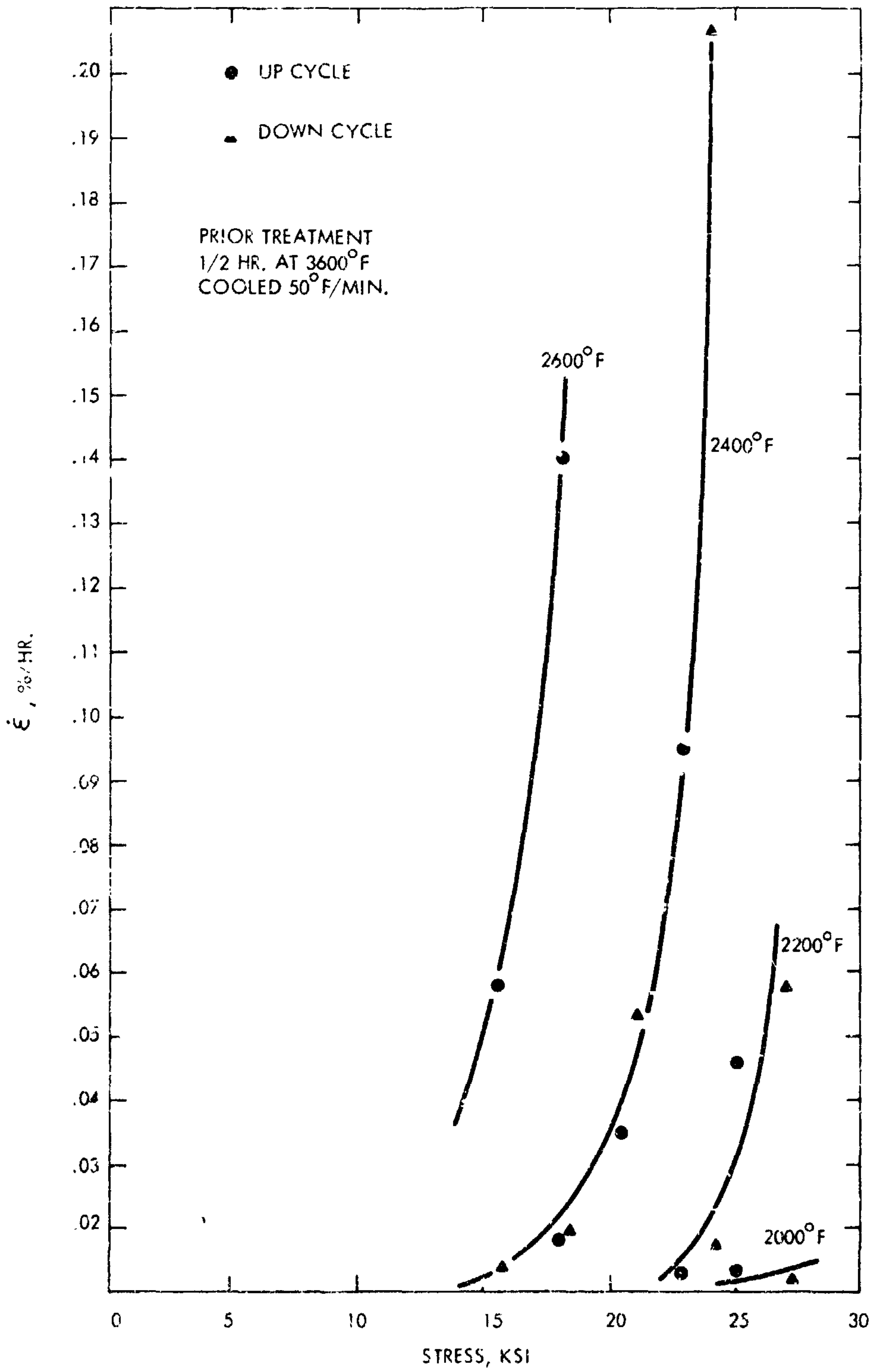


Figure 9. Effect of Test Sequence on Creep Behavior of ASTAR-811C During Incremental Stress and Temperature Testing. (Prior treatment, 1/2 hour at 3600°F, cooled 50F° per minute.)

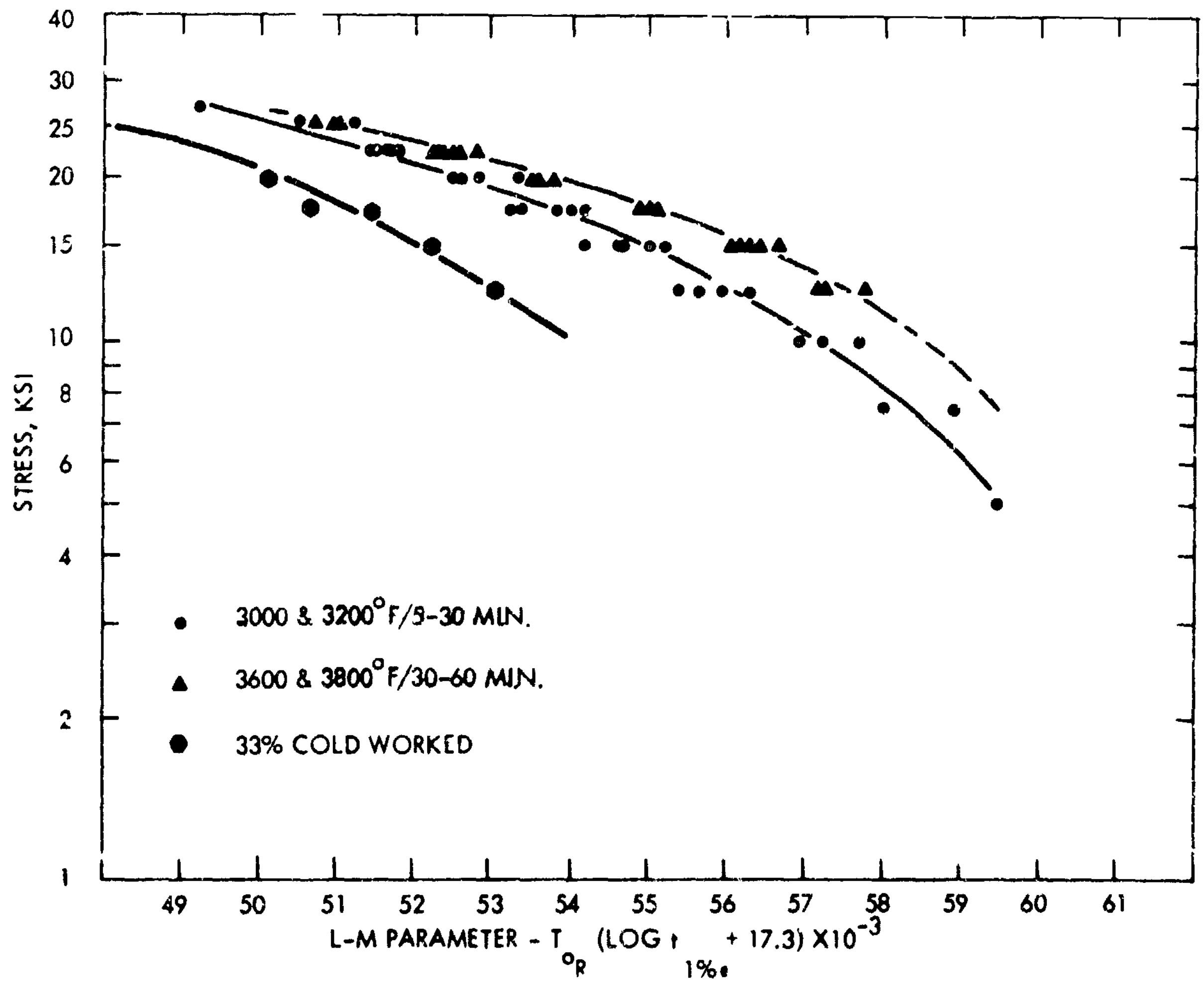


Figure 10. Larson-Miller plot of incremental creep test data on ASTAR-811C.

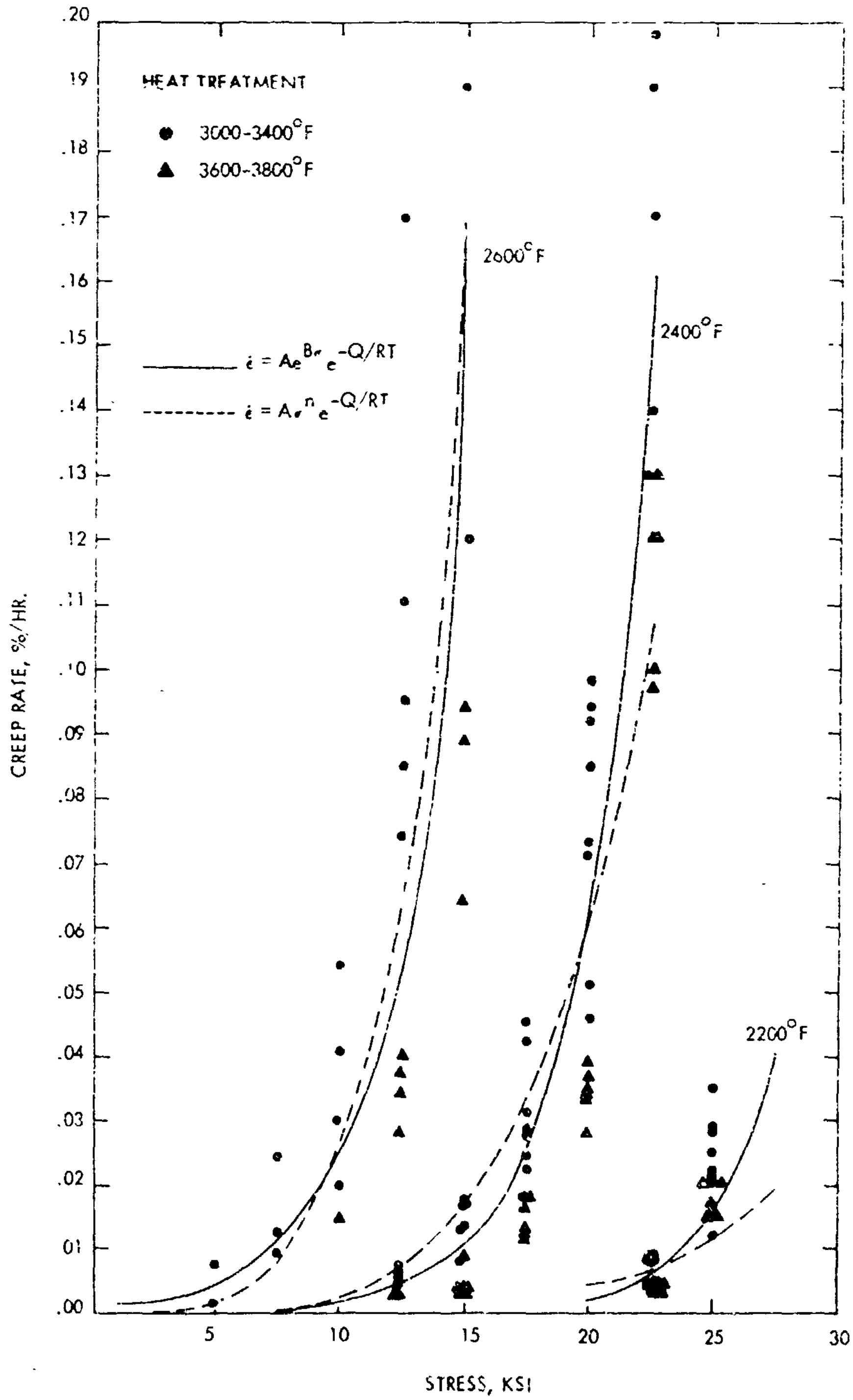


Figure 11. Temperature and stress dependence of creep of ASTAR-811C.

These curves were drawn using values for the constants in equations (1) and (2) determined by treating all of the creep data as a group, that is, irrespective of heat treat condition. The values for these constants determined by a least-squares-computer program are as follows:

Eq. (1) ($e^{B\sigma}$)			Eq. (2) (σ^n)		
A, %/hr.	B, $(ksi)^{-1}$	Q, kcal/mole	A, $(\%/hr)/(ksi)^n$	n, dimensionless	Q, kcal/mole
1.6×10^{14}	3.73	135.5	1.5×10^{8}	4.61	111.8

No particular physical significance is attached to the values obtained for these constants. However, equation (1), in particular, is useful for estimation purposes.

The effect of heat treatment on creep is shown in Figure 12 in terms of conventional (constant T, σ) creep curves. The specimen annealed at 3000°F shows a monotonically increasing creep rate, while the sample annealed at 3600°F shows a linear creep rate out to at least 1000 hours. The difference in their creep behavior therefore increases with increasing time or strain. These two creep curves also depict the magnitude of the effect on creep due to the range of heat treatments being investigated. It is noted that the two curves start out about the same, and even after 50 hours it is difficult to distinguish between them. This suggests the possibility that the incremental creep tests may minimize the differences in creep behavior due to heat treatment effects because of the short time increments employed at each combination of temperature and stress.

4.1.3 Microstructure

After moderate to heavy cold working, ASTAR-811C recrystallizes in 1 hour at about 2400 to 2600°F. With increasing annealing temperature and time the grain size increases (Figure 6), and the precipitates present in the cold worked material are gradually taken into solution

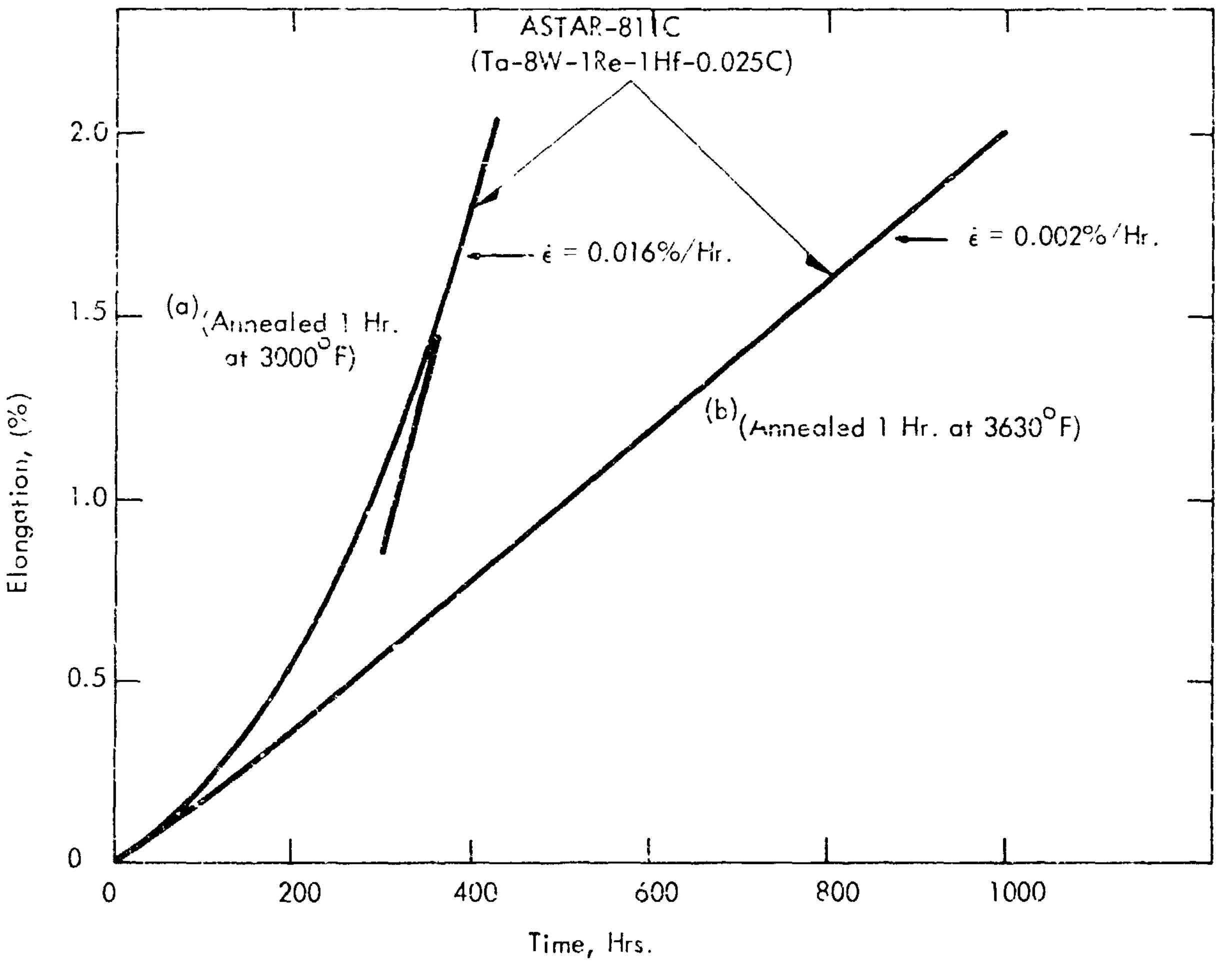


Figure 12. Effect of Heat Treatment on Creep of ASTAR-811C in Conventional Test at Constant Stress (15 ksi) and Constant Temperature (2400°F).

(Figures 13 to 15). The precipitates in ASTAR-811C have been identified as the hexagonal close packed tantalum rich dimetal-carbide, $(Ta, W)_2C$, regardless of the mechanical-thermal conditions.

The optical microstructure of samples annealed at 3600°F and above appears to be single phase (Figure 15). There is no doubt that the carbides have been taken completely into solution. However, reprecipitation on a scale unresolvable by optical microscopy occurs during cooling, as shown by the electron transmission micrographs in Figure 16.

The precipitates shown in Figure 17 were extracted from creep specimens after being tested as shown in Figure 12 . The initial precipitates (Figure 16) in the specimen annealed at 3630°F grew into long, thin platelets (Figure 17 b) during creep testing. These precipitates are still much smaller than those in the specimen annealed at 3000°F (Figure 17(a)). Figure 18 shows a transmission micrograph of the creep specimen which had been annealed at 3630°F prior to testing. The dislocations appear to be pinned by the small precipitates and tangled around the larger ones.

4.1.4 Discussion

The two principal microstructural features which change for the range of heat treatments investigated are grain size and precipitate dispersion. Since both of these features can influence creep deformation, it is of interest to consider their relative contribution to the creep resistance of ASTAR-811C. Creep rate (from incremental tests) is plotted as a function of grain size in Figure 19 for general combinations of test temperature and stress. The circular points represent specimens annealed at 3400°F (below carbon solvus) and below while the triangular points are for specimens annealed at 3600°F (above carbon solvus) and above. There is little difference in the creep rate of specimens annealed at the higher temperatures (above the carbon solvus), even though the grain size varied by a factor of 4 or 5. In view of the observed

Figure 13. Microstructure of ASTAR-811C as-cold-worked 33 percent. Optical micrographic montage, X1500.

Figure 14. Microstructure of ASTAR-811C after annealing cold-worked material for 5 minutes at 3000°F. Optical micrographic montage, X1500.

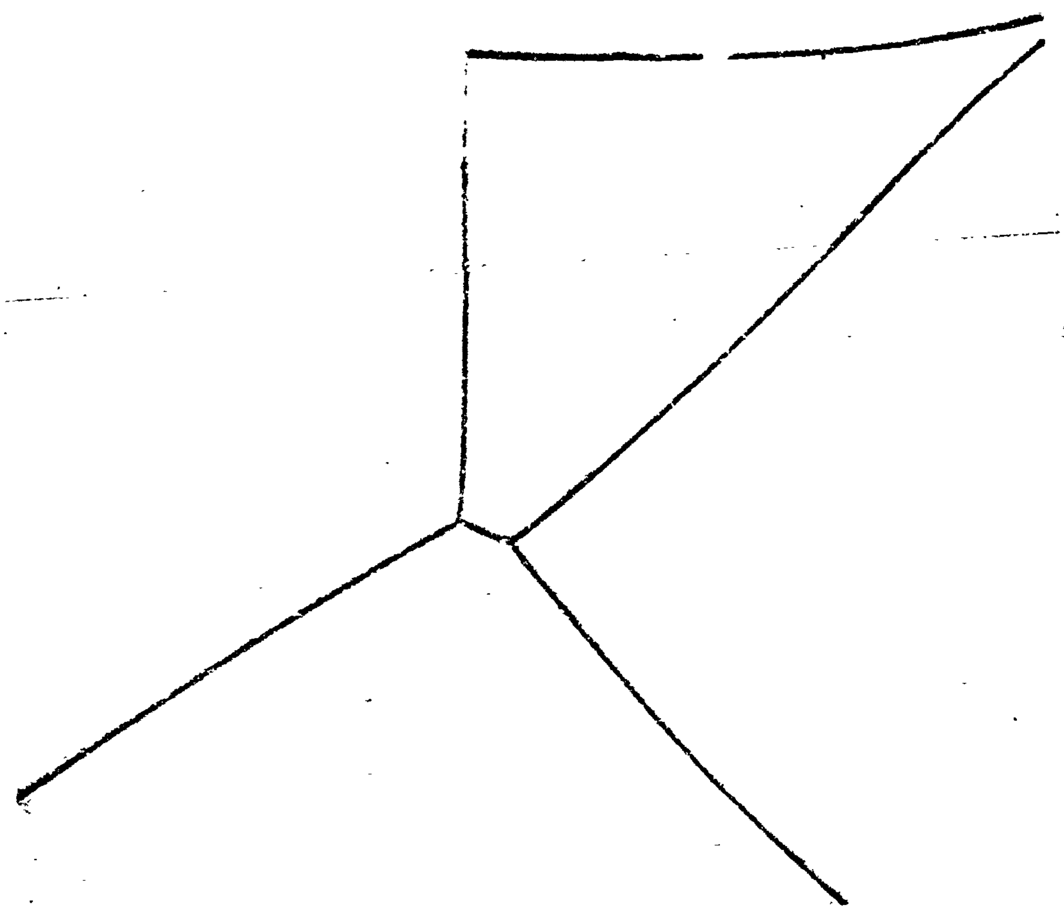
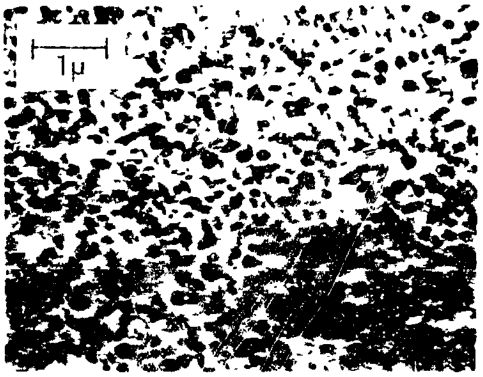


Figure 15. Microstructure of ASTAR-811C after annealing cold-worked material (fig. 13) for 30 minutes at 3600°F. Optical micrographic montage, X1500.



(a)

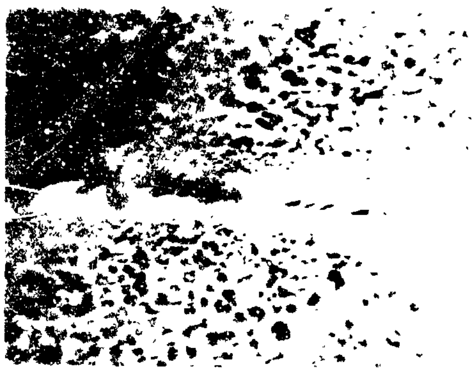
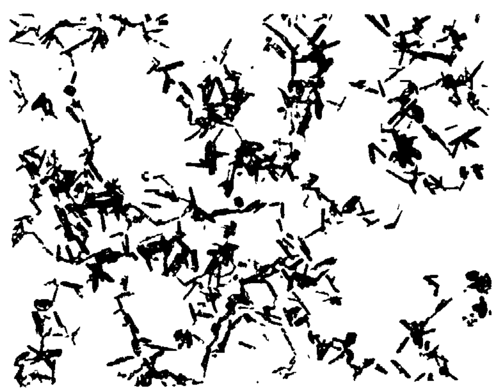
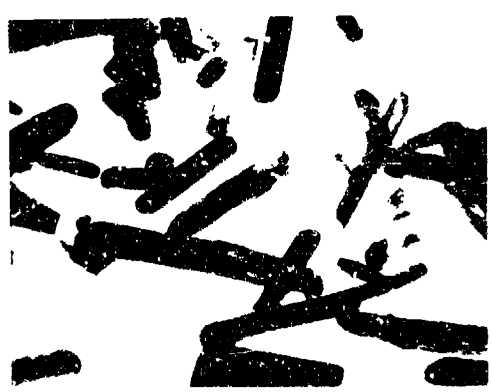


Figure 16. Electron Transmission Micrographs of ASTAR-811C After Annealing for 1 hour at 3630°F

(a) Annealed 1 Hr/3000°F & Creep Strained 2% in 500 Hrs.



(b) Annealed 1 Hr/3630°F & Creep Strained 2% in 1000 Hrs. Same Magnification as (a)



(c) Same as (b) Except at 40,000X

Figure 17. Electron Micrographs of Tantalum Dimetal Carbide (Ta_2C) Precipitate Extracted from Creep Specimens (fig. 12) Tested at 2400°F and 15 ksi.

Figure 18. Electron Transmission Micrograph of ASTAR-811C Creep Specimen After Testing at 2400°F and 15 ksi to 2 percent Strain in 1000 Hours. (Pretest heat treatment, 1 hour at 3630°F.)

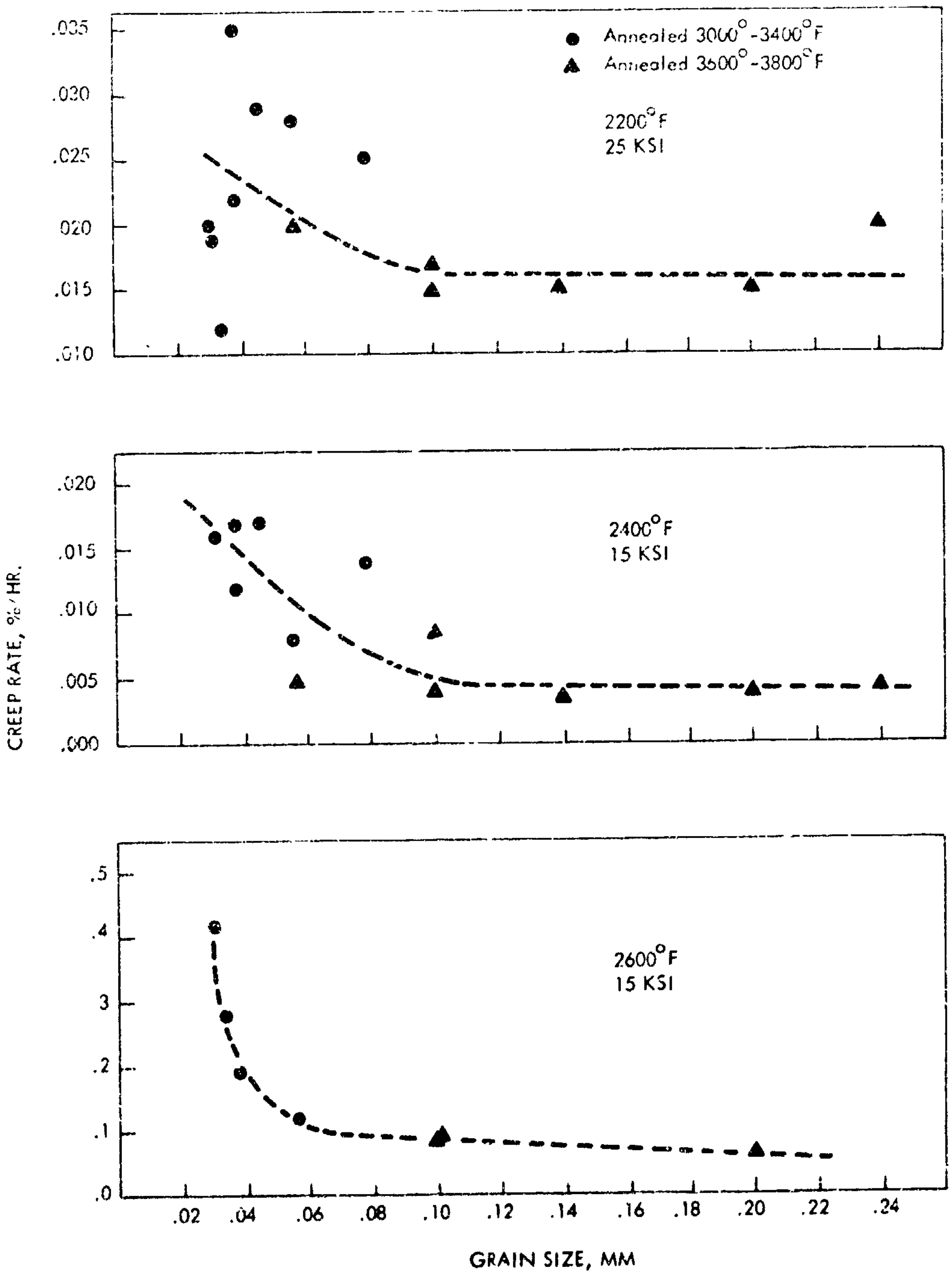


Figure 19. Creep Rate (incremental tests) of ASTAR-811C as a Function of Grain Size for Several Combination of Test Temperature and Stress

changes in precipitate structure, it is believed that the trends shown in Figure 19 reflect for the most part the contribution of the precipitate dispersion.

4.2 EFFECT OF COOLING RATE ON MECHANICAL PROPERTIES

The effect of cooling rate was investigated using sheet tensile and creep specimens of ASTAR-811C, NASV-20, cooled at three given rates after a 1/2 hour anneal at 3600°F. The three cooling rates chosen were helium gas quench, which was equivalent to a cooling rate of 700 to 800 F°; 50 F° and 5F° per minute, respectively. The specimens were encapsulated in evacuated tantalum envelopes to prevent carbon loss at the 3600°F annealing temperature. Sample cooling rates were controlled from 3600°F to 2000°F. The slower cooled samples were helium gas quenched from 2000°F to ambient temperature. The helium gas quenched samples required approximately two minutes to reach 2000°F. The cooling rates of the slower cooled samples were controlled by programming furnace element power to achieve the desired time-temperature relationships. A cooling rate of 50 F° per minute required 32 minutes to reach 2000°F from 3600°F. The 5 F° per minute cooling rate required 5 hours and 20 minutes for completion of the cooling process.

The cooling rates represent a broad spectrum of conditions which might be encountered in commercial heat treatment applications of various component sizes and shapes. The results of the Task I study indicated that the larger grain size produced by annealing above the carbon solvus followed by rapid cooling produced the optimum or minimum creep rate for ASTAR-811C. In that study all the samples were helium gas quenched from the final annealing temperature. It was recognized that slower cooling rates would provide an opportunity for the carbide phase to precipitate within the matrix at temperatures below the carbon solvus. Since the precipitation process is strongly temperature dependent, the morphology and distribution of the carbide phase would be a function of the cooling rate and hence could have a significant effect on mechanical property behavior.

4.2.1 Microstructure

The microstructures of the samples cooled at various rates are shown in Figure 20. The structures are typical of classical precipitation phenomenon. The helium gas quenched sample appears to be single phase at low magnification (~200x). At 650x, as shown in Figure 20a, a finely divided precipitate can be seen in the grain matrix with denuded zones associated with grain boundaries. During the rapid quench from the annealing temperature many second phase precipitates were nucleated. As the residence time at elevated temperature increased, agglomermation of the carbide phase proceeded producing the microstructures shown in Figures 20b and 20c. The intermediate cooling rate, 50F° per minute, produced a duplex type structure of finely divided precipitate along with larger plate-like precipitates at grain boundaries and within the grains. The 5F° per minute cooling rate produced a structure with very little fine precipitate in evidence but dominated with large massive blocky type precipitate associated primarily with grain boundaries.

4.2.2 Tensile Properties

Samples cooled at various cooling rates from final annealing temperature were tensile tested at room temperature and at elevated temperatures. The room temperature tensile results are given in Table 2 and the elevated temperature test results are given in Table 3. The data are summarized in Figure 21. Included for comparison are tensile data for heat NASV-20 taken from earlier work(1). The prior tensile data were for material annealed one hour at 3000°F and furnace cooled.

The yield strengths of the helium gas quenched material and the material cooled at 50F° per minute were significantly higher than the yield strength of the material heat treated and cooled in the more conventional manner. The finely divided carbide phase evident in the microstructures for these materials may have been responsible for the increase in yield strength. Initial dislocation motion may have been inhibited by the finely dispersed carbide phase

(a) ASTAR-811C, 1/2 hr. at 3600°F, Helium Quenched (Cooling Rate ~800°F/min.)
DPH 313
(a)

(b) ASTAR-811C, 1/2 hr. at 3600°F, Cooling Rate 50 F°/min.
DPH 317
(b)

(c) ASTAR-811C, 1/2 hr. at 3600°F, Cooling Rate 5 F°/min.
DPH 278
(c)

Figure 20. Microstructures of ASTAR-811C Subjected to Various Cooling Rates from Final Annealing Temperature

TABLE 2

ROOM TEMPERATURE TENSILE PROPERTIES OF ASTAR-811C SUBJECTED TO VARIOUS COOLING RATES FROM FINAL ANNEALING TEMPERATURE

Specimen History	0.2 Yield Strength (KSI)	Ultimate Strength (KSI)	Elongation Uniform (%)	Elongation Total (%)	Reduction In Area (%)
1	100.0	101.7	15.4	24.0	22
2	98.8	102.6	13.9	23.0	26
3	83.8	87.5	15.7	23.0	27
4	85.0	104.4	16.3	26.6	49

All Material Heat NASV-20

Strain Rate 0.05 in/min.

1. Annealed, 1/2 Hour 3600°F, Helium Gas Quenched
2. Annealed, 1/2 Hour 3600°F, Cooled 50F°/min.
3. Annealed, 1/2 Hour 3600°F, Cooled 5F°/min.
4. Annealed, 1 Hour 3000°, Furnace Cooled [1]

causing the yield strength to increase. The yield strength of the material cooled at 5F° per minute was essentially the same as the conventionally heat treated material. The ultimate strength, however, was significantly lower. The lack of strain hardening in all three materials of this study is evident. The low temperature deformation behavior of BCC alloys are strongly dependent on dislocation and vacancy density as well as dislocation-interstitial interaction. These phenomenon which are strongly affected by final annealing temperature and cooling rate are undoubtedly responsible for the deformation behavior exhibited by these materials. A more definitive explanation of the low temperature deformation of these materials would require additional tests to confirm the influence of cooling rate on the low temperature deformation behavior of ASTAR-811C.

TABLE 3

ELEVATED TEMPERATURE TENSILE PROPERTIES OF ASTAR-811C SUBJECTED TO VARIOUS COOLING RATES FROM FINAL ANNEALING TEMPERATURE

Specimen History	Test Temperature (°F)	0.2 Yield Strength (KSI)	Ultimate Strength (KSI)	Elongation Uniform (%)	Elongation Total (%)	Reduction In Area (%)
1	2000	27.6	51.7	15.3	20.0	24
1*	2000	35.2	59.5	14.6	19.0	22
1	2400	26.3	36.0	13.5	23.0	17
1*	2400	23.3	37.0	11.8	22.0	25
2	2000	32.9	55.7	12.6	18.0	26
2*	2000	35.2	59.6	14.6	20.0	20
2	2400	29.4	39.1	11.2	23.0	32
2*	2400	28.7	41.4	12.7	25.0	34
3	2000	28.0	51.8	14.2	18.0	22
3*	2000	27.8	53.5	13.0	17.0	16
3	2400	26.2	35.9	11.8	25.0	23
3*	2400	25.9	39.4	13.2	21.0	21
4	2000	35.0	60.9	-	24.0	-
4	2400	30.4	49.9	-	28.8	-

All Material From Heat NASV-20

1. Annealed 1/2 Hour 3600°F, Helium Gas Quenched
2. Annealed 1/2 Hour 3600°F, Cooled 50F°/min.
3. Annealed 1/2 Hour 3600°F, Cooled 5F°/min.
4. Annealed 1 Hour 3000°F, Furnace Cooled(1)

Constant Strain Rate Test 0.05 in/min.

* Strain Rate Changed From 0.005 in/min. to 0.5 in/min. after 3% Plastic Strain

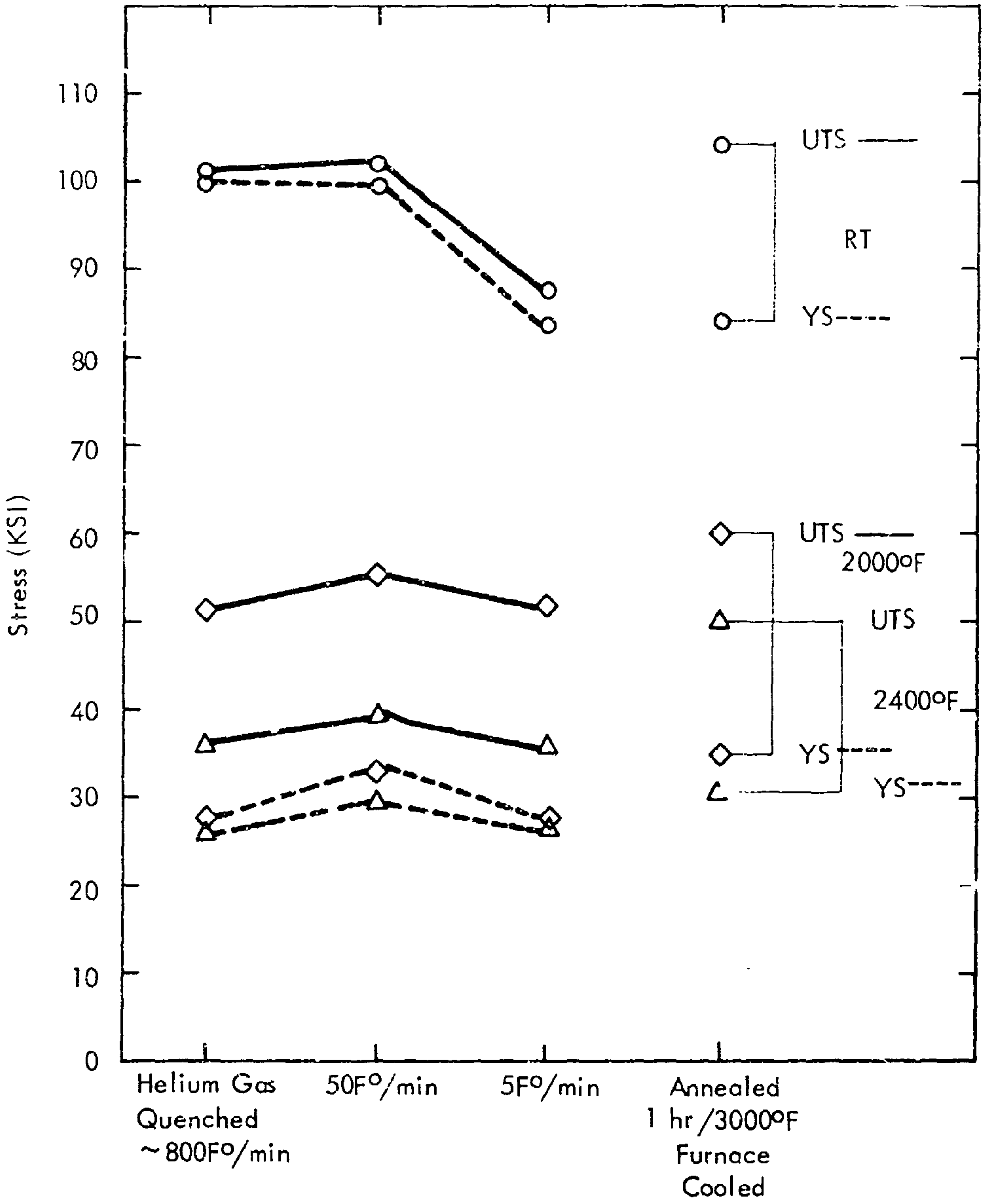


Figure 21. Tensile Properties of ASTAR-811C Subjected to Various Cooling Rates from Final Annealing Temperature

The elevated temperature tensile properties for material subjected to the various cooling rates are also presented in Figure 21. Very little difference in yield and ultimate strength can be seen for the materials evaluated as a function of cooling rate. The ASTAR-811C annealed at 3000°F exhibited higher mechanical properties at both test temperatures. These results tend to emphasize the effect of final annealing temperature on mechanical behavior. The cooling rate test material of this study was annealed 1/2 hour at 3600°F, which was above the carbon solvus. The ASTAR-811C annealed at 3000°F (below the carbon solvus) had a smaller grain size (0.037 mm vs. 0.10 mm). These factors are most probably responsible for the difference in elevated temperature mechanical property behavior.

In addition to the constant strain rate tests carried out at 2000 and 2400°F, a strain rate change test was also performed. An initial strain rate of 0.005 in/in/minute was used to 3 percent plastic strain then the strain rate was increased to 0.5 in/in/minute to failure. The test results are included in Table 3

At the 2400°F test temperature the lower strain rate (0.005 in/in/minute) produced a slightly lower yield strength and the higher strain rate (0.5 in/in/minute) produced a slightly higher ultimate strength compared to the constant strain rate (0.05 in/in/minute) test results. This behavior which was the same for all three test materials, is typical for stable material. At the 2000°F test temperature, test results were not the same. The lower strain rate produced a higher yield strength in helium gas quenched and 50 F°/min. cooled material, where as the slowest cooled material, 5F°/min., behaved in the conventional manner. This anomolous behavior was most likely due to an occurrence of a thermally activated process such as precipitation of the carbide phase during testing. The slower strain rate provided sufficient time at test temperature to permit precipitation of the carbide phase (Ta_2C) from supersaturation produced by rapid cooling. Material cooled at the slowest cooling rate, 5F° per minute, approached more nearly equilibrium conditions and thus exhibited more conventional behavior.

4.2.3 Creep Properties

Material subjected to the various cooling rates were also creep tested. The test program was limited to two creep specimens per cooling rate. The specimens were tested at two temperatures, 2000 and 2400°F, at constant stresses of 27.5 and 15 ksi, respectively, Creep data are given in Table 4 and complete creep curves are presented in Figures 22 and 23.

At 2400°F, there does not appear to be any significant effect of cooling rate on the creep behavior of ASTAR-811C. The data appears to be well within experimental error or what would be normal scatter for a given material. At 2000°F, there does appear to be some effect of cooling rate on creep behavior. The material cooled at the slowest cooling rate, 5F°/min., appears to have a somewhat higher creep rate compared to material cooled at the intermediate rate, 50F°/min. The creep rate of the helium gas quenched material was slightly higher but not enough to be considered significant.

Samples were cut from the gage section of the creep specimens for optical and transmission electron microscopic evaluation. The optical micorgraphs are shown in Figures 24, 25, and 26. Included with the micrographs of the creep specimens are micrographs of the starting material for comparison. In each case the morphology and distribution of carbide phase, Ta_2C, was altered by the thermal exposure during creep testing. The trend toward redistribution of the carbide phase to the grain boundaries as shown in the slowest cooled material, 5F°/min., is evident in the creep samples of the helium gas quenched and 50F°/min. cooled materials. The amount of second phase present was greater in the 2400°F creep specimens. The process of dissolution of the smaller carbide precipitates, diffusion of the carbon, and the redeposition of the carbide phase on larger particles is most likely more efficient at the higher temperature.

Transmission electron microscopic examination of the starting material and creep tested

TABLE 4

CREEP PROPERTIES OF ASTAR-811C SUBJECTED TO VARIOUS COOLING RATES FROM FINAL ANNEALING TEMPERATURE

Specimen History	Test Temp. (°F)	Stress (KSI)	Strain On Loading (%)	Test Time (Hrs.)	Total Elongation (%)	Secondary Creep Rate (%/Hr.)	Time to 1% Elongation (Hrs.)	Larson-Miller Parameter $P \times 10^3$
1	2000	27.5	0.20	738	1.97	0.00186	540	49.2
2	2000	27.5	0.13	666	1.21	0.00122	820	49.2
3	2000	27.5	0.25	498	2.42	0.00364	275	48.5
1	2400	15.0	0.10	962	4.71	0.00365	273	56.5
2	2400	15.0	0.08	648	1.93	0.0026	385	56.8
3	2400	15.0	0.10	577	1.68	0.00278	360	56.8

$P = T_{^oR} (17.3 + \text{Log } t) \times 10^{-3}$

1. Annealed 1/2 hour at 3600°F, helium gas quenched
2. Annealed 1/2 hour at 3600°F, cooled 50F°/min.
3. Annealed 1/2 hour at 3600°F, cooled 5F°/min.

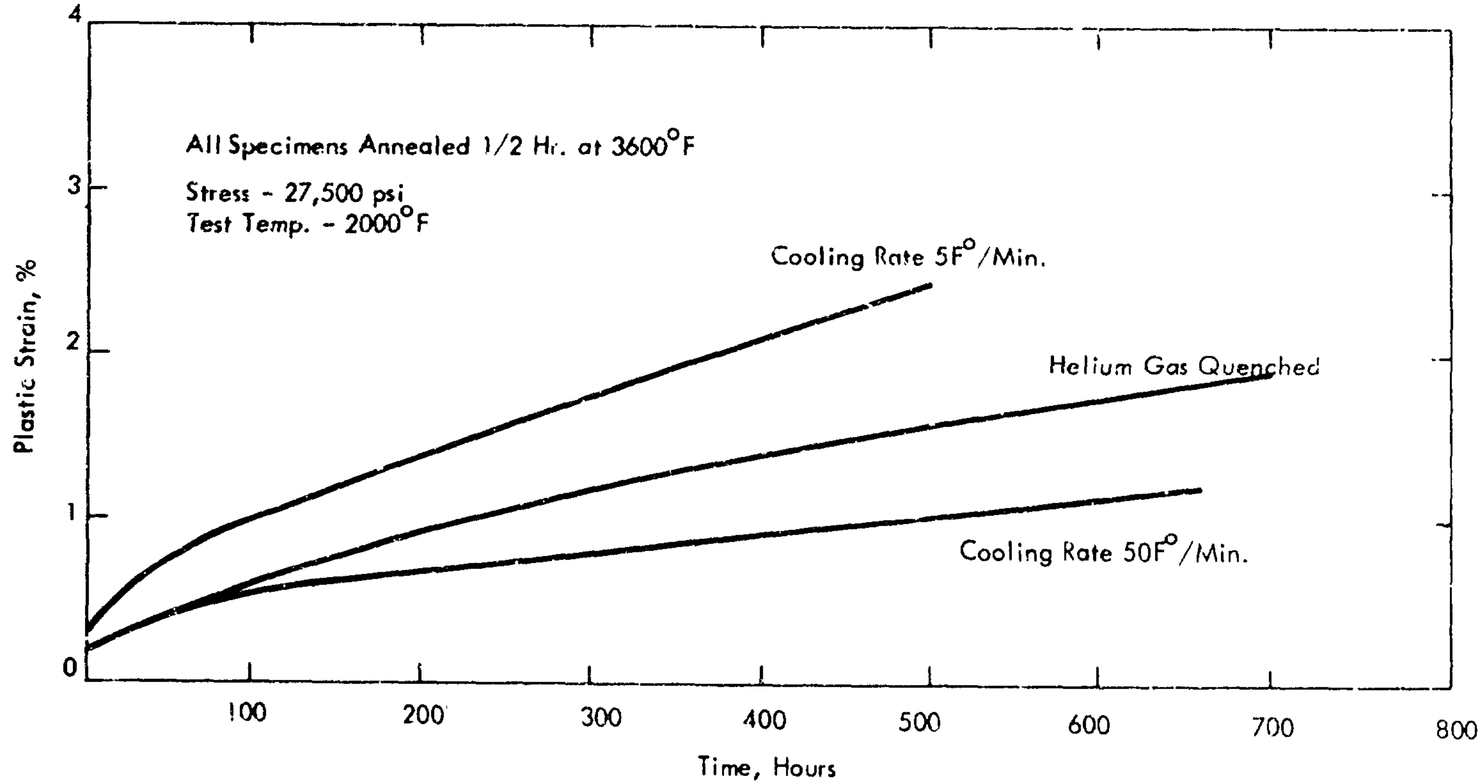


FIGURE 22- Effect of Cooling Rate on the Creep Strength of ASTAR-811C (NASV-20)

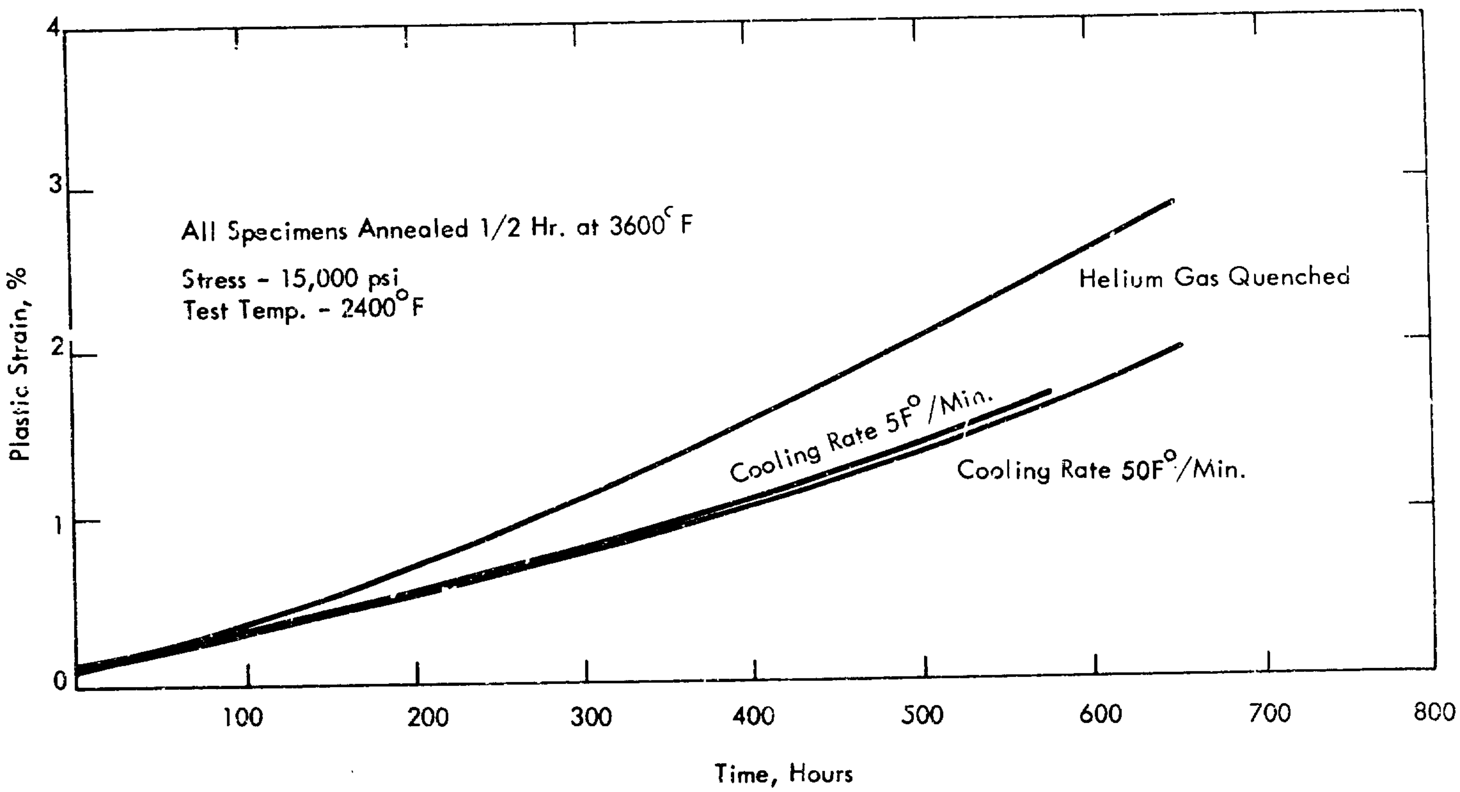


FIGURE 23- Effect of Cooling Rate on the Creep Strength of ASTAR-811C (NASV-20)

A. Helium Gas (Quenched)
Starting Material

200X 313 DPH

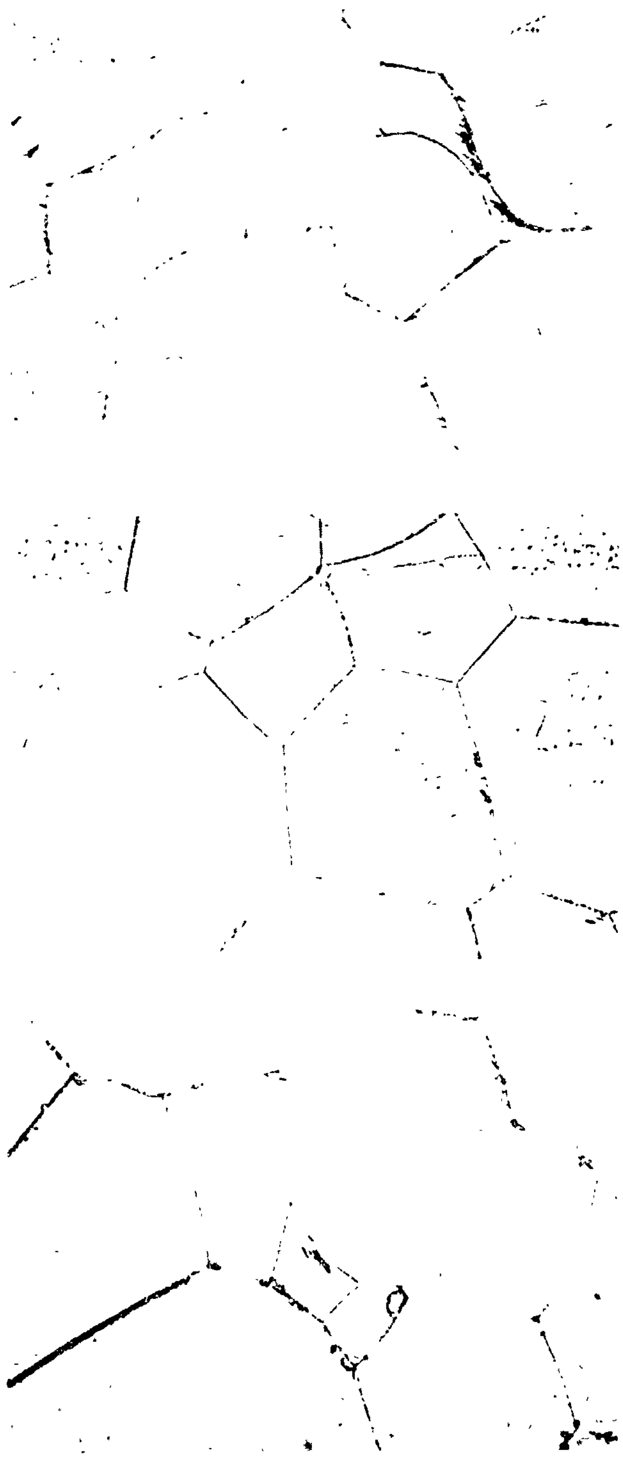
B. Helium Gas (Quenched)
Creep Tested 738 hrs
at 2000°F, 27.5 KSI

200X 209 DPH

C. Helium Gas (Quenched)
Creep Tested 962 hrs
at 2400°F, 15 KSI

200X 218 DPH

Figure 24. Microstructures of Helium Gas Quenched (from 3600°F) Creep Specimens of ASTAR-811C



A. Cooled 50F°/min
Starting Material

200X 317 DPH

B. Cooled 50F°/ min
Creep Tested 666 hrs
at 2000°F, 27.5 KSI

200X 214 DPH

C. Cooled 50F°/min
Creep Tested 648 hrs
at 2400°F, 15 KSI

200X 221 DPH

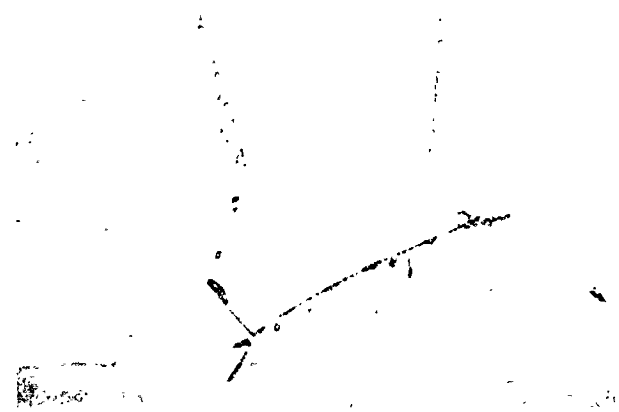
Figure 25. Microstructures of ASTAR-811C Creep Specimens Cooled 50F°/min from 3600°F

A. Cooled 5F°/min
Starting Material

200X 278 DPH

B. Cooled 5F°/min
Creep Tested 498 hrs
at 2000°F, 27.5 KSI

200X 228 DPH



C. Cooled 5F°/min
Creep Tested 577 hrs
at 2400°F, 15 KSI

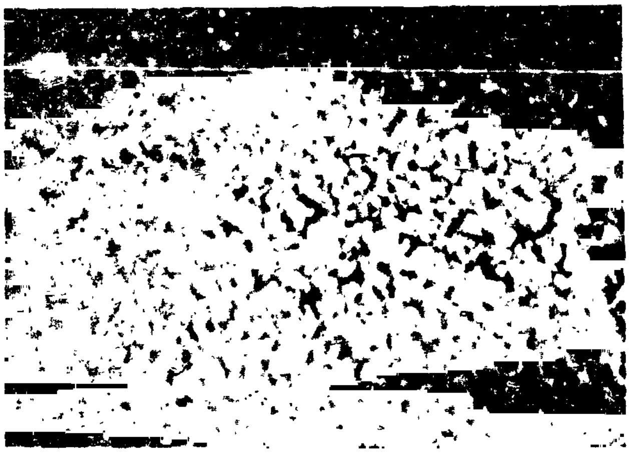
200X 212 DPH

Figure 26. Microstructures of ASTAR-811C Creep Specimens Cooled at 5F°/min from 3600°F

material was also conducted. The objective of this study was to determine the role of the carbide phase in the creep behavior of ASTAR-811C. The transmission electron micrographs are shown in Figures 27, 28, and 29. The starting helium gas quenched material exhibited an extremely fine precipitate on the order of (.025 μm) in diameter. The 50F°/min. cooling rate produced particles approximately 0.2 μm in diameter which increased to 0.4 μm at the 5F°/min. cooling rate. The micrographs of the creep tested material show in each case that a significant change occurred with respect to the size and quantity of the carbide phase present. The quantity of microscopic carbide phase was significantly reduced for each creep test temperature. Some evidence of a possible carbide particle dislocation interaction can be seen for the 2000°F tested material. At 2400°F, the fine carbide phase had virtually disappeared. The fine carbide precipitate apparently redissolved with the carbon diffusing to and reprecipitating on larger, more stable, grain boundary particles. This process was more efficient at the higher test temperature, 2400°F. The transmission electron micrographs give evidence that the role of intragranular carbide precipitate in the creep behavior of ASTAR-811C is rather limited. The intergranular or grain boundary precipitate may be of more significance to the creep behavior of ASTAR-811C.

4.3 EFFECT OF THERMAL-MECHANICAL PROCESSING AND WELDING ON CREEP PROPERTIES

In the study of the effect of grain size on the creep behavior of ASTAR-811C, it was discovered that the creep properties were independent of grain size so long as the final anneal was conducted at a temperature above the carbon solvus. Annealing above the solvus, 3600°F, produces a grain size on the order of 0.1 mm after 1/2 hour at temperature. In this study an attempt was made to produce a fine (≤.03mm)grain size material which retained the high temperature carbide precipitate morphology produced by rapid cooling from above the carbon solvus. By keeping the precipitate form constant, and producing a fine grained material, the effect of grain size and carbide precipitate morphology on creep behavior can be separated.



A. Helium Gas Quenched
Starting Material

46, 000X

B. Helium Gas Quenched
Creep Tested 738 hrs
at 2000°F, 27.5 KSI

56, 000X

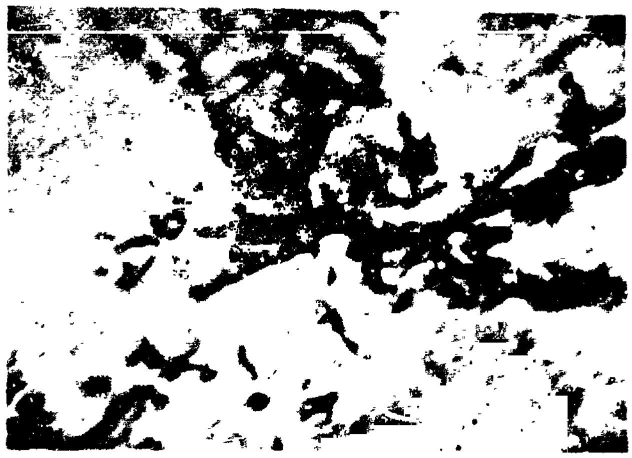
C. Helium Gas Quenched
Creep Tested 962 hrs
at 2400°F, 15 KSI

46, 000X

Figure 27. Transmission Electron Micrographs of Helium Gas Quenched (from 3600°F) Creep Specimens of ASTAR-811C

A. Cooled 50°F/min
Starting Material

56,000X



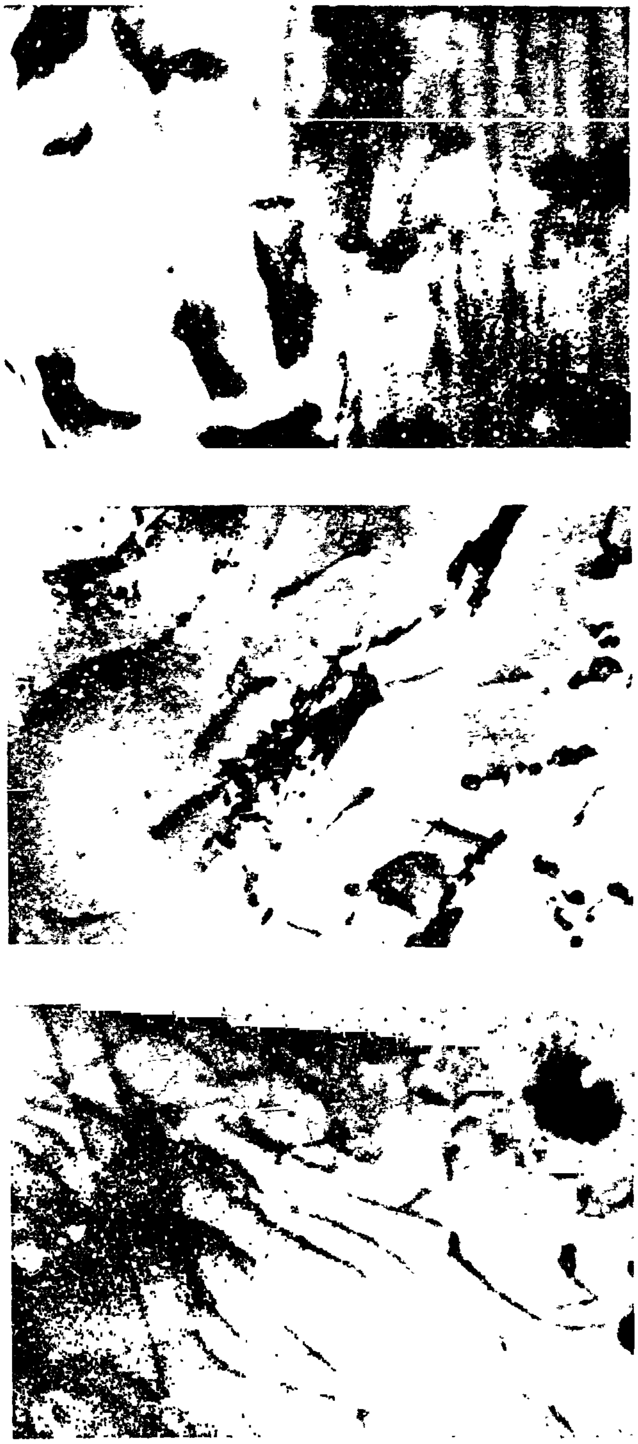
B. Cooled 50F°/min
Creep Tested 666 hrs
at 2000°F, 27.5 KSI

56,000X

C. Cooled 50F°/min
Creep Tested 648 hrs
at 2400°F, 15 KSI

36,000X

Figure 28. Transmission Electron Micrographs of ASTAR-811C Creep Specimens Cooled at 50F°/min from 3600°F



A. Cooled 5F°/min
Starting Material

36, 000X

B. Cooled 5F°/min
Creep Tested 498 hrs
at 2000°F, 27.5 KSI

36, 000X

C. Cooled 5F°/min
Creep Tested 577 hrs
at 2400°F, 15 KSI

56, 000X

Figure 29. Transmission Electron Micrographs of ASTAR-811C Creep Specimens Cooled at 5F°/min from 3600°F

4.3.1 Material Preparation

The initial program plan called for the development of a processing schedule which would produce the desired microstructure, fine grain with the high temperature form of the carbide phase present. For this study, another heat of ASTAR-811C was required, WC 650078. Chemistry of this heat is given in the Experimental Procedure Section. Two one inch wide strips were rolled to 0.143 and 0.082 inch thick respectively. The strips were encapsulated in evacuated envelopes of T-222 (Ta-10W-2.4Hf-0.01C), annealed 1/2 hour at 3600°F and helium gas quenched. The thicker sheet was rolled to a reduction of 60 percent, 0.057 inch. The thinner sheet was rolled to a reduction of 30 percent, to a thickness of 0.057 inch. The sheets were cut into three equal parts. One strip of 60 percent reduced material and one strip of 30 percent material were annealed at 2400, 2600, and 2800°F. After the heat treatment, metallographic samples were taken and the remaining sheet of both starting thicknesses were reduced 30 percent to a uniform finished thickness of 0.040 inch. Each lot of finished material was annealed at the same temperature as the intermediate heat treatment. The rolling and heat treatment schedule is outlined in Figure 30.

Metallographic examination of the initial twelve sheet samples revealed that recrystallization had not occurred in any of the sheets. Typical microstructures for sheet reduced 60 and 30 percent are shown in Figures 31 and 32. Reducing the sheet an additional 30 percent did not promote recrystallization. Samples of material initially reduced 60 percent were annealed for 2 and 4 hours respectively with the same results. The combination of cold work and annealing temperature was apparently insufficient to cause recrystallization to occur. Samples of ASTAR-811C reduced 30 and 40 percent and annealed 1/2 hour at 3000°F were successfully recrystallized. The microstructures are shown in Figure 33. The material rolled 60 percent had a more uniform grain size (0.03 mm) than the 30 percent reduced material. The high temperature form of the carbide precipitate appears to have been retained as indicated by the 1500 x micrographs. The grain boundaries appear devoid of the large block

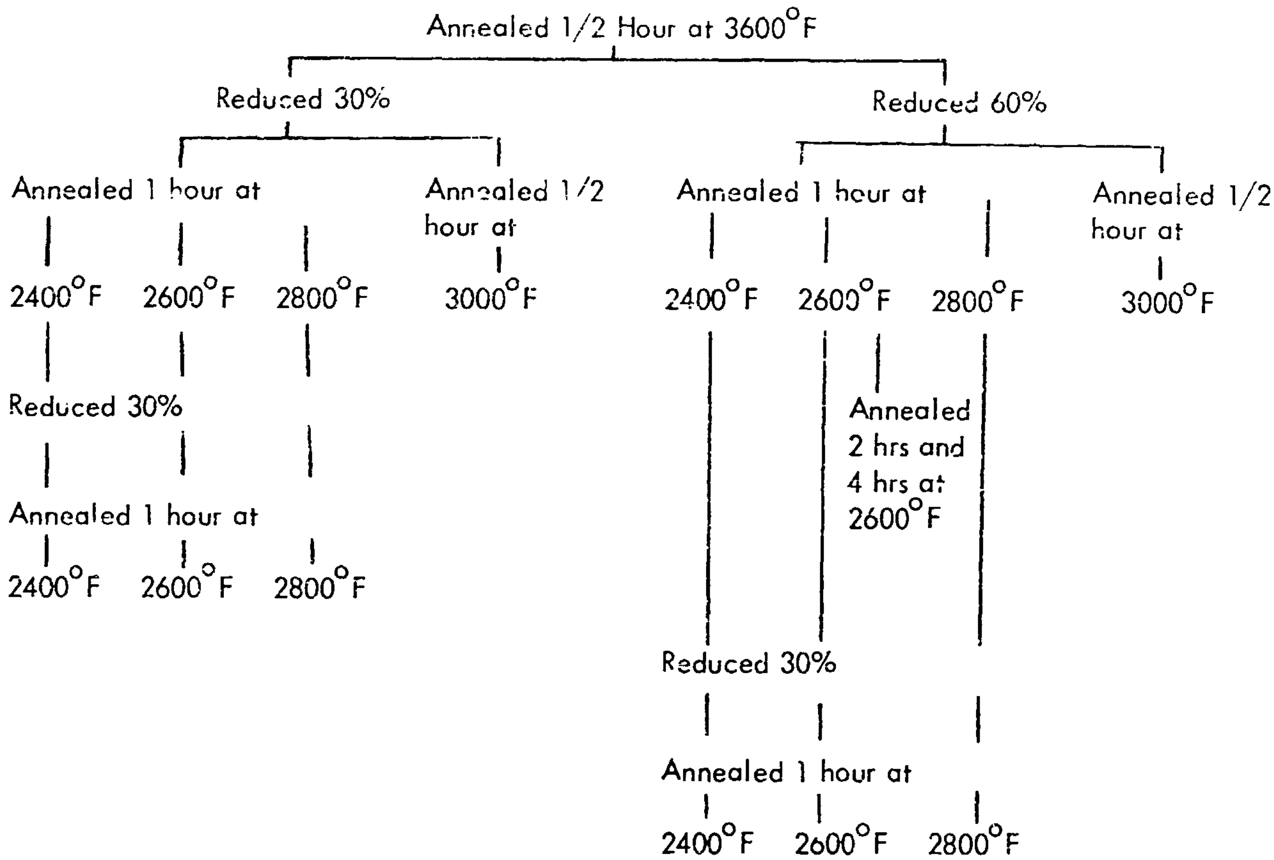
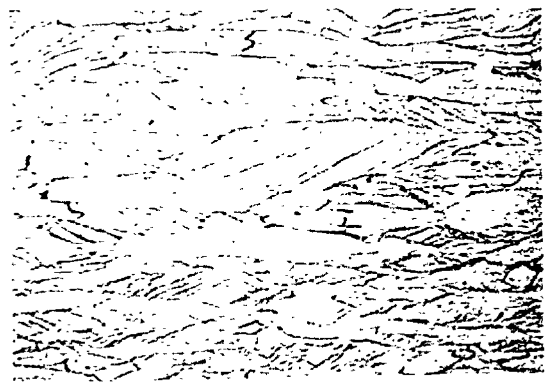


Figure 30 Thermal-Mechanical Processing of ASTAR-811C

A As Rolled DPH 382



B 1 hr/2400°F DPH 314

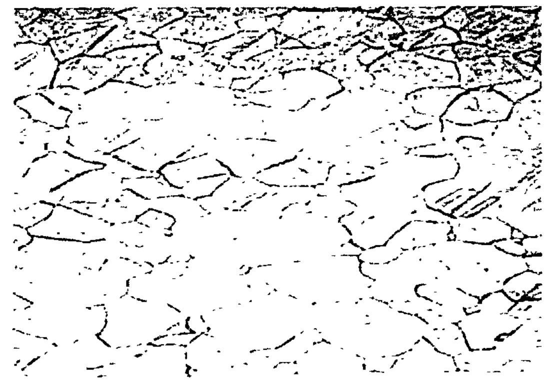
C 1 hr/2600°F DPH 287

D 1 hr/2800°F DPH 316

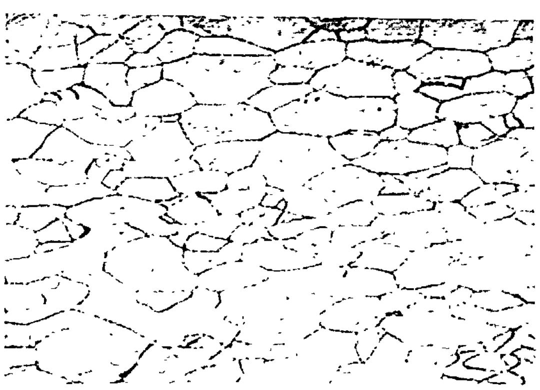
100X

Figure 31. Recrystallization Behavior of ASTAR-811C Annealed 1/2 Hour at 3600°F and Cold Rolled 60 Percent

A As Rolled DPH 360



B 1 hr/2400°F DPH 297

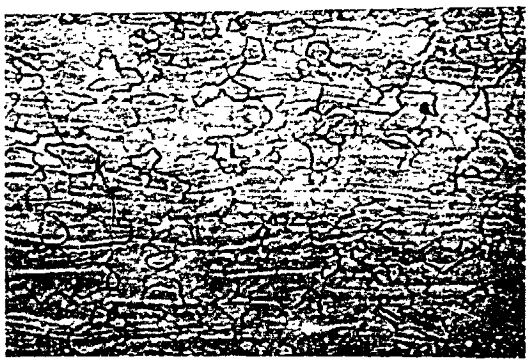


C 1 hr/2600°F DPH 296

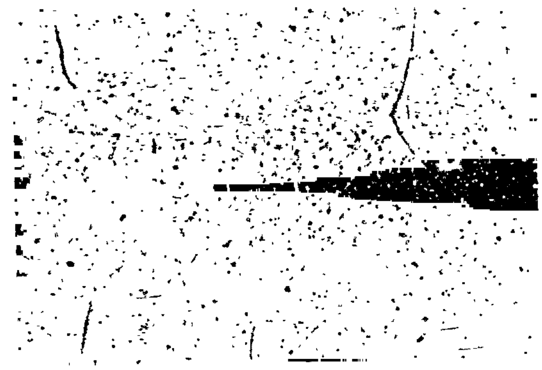
D 1 hr/2800°F DPH 300

100X

Figure 32 Recrystallization Behavior of ASTAR-811C Annealed 1/2 Hour at 3600°F and Cold Rolled at 30 Percent



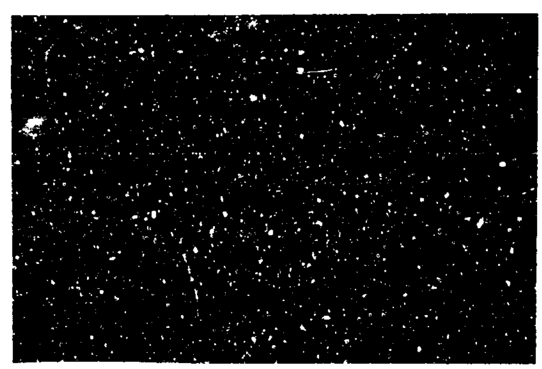
A DPH-307 100X



B 1500X

Reduced 60%, Annealed
1/2 Hour at 3000°F

C DPH-280 100X



D 1500X

Reduced 30%, Annealed
1/2 Hour at 3000°F

Figure 33 Microstructures of ASTAR-811C Subjected to Various Thermal-Mechanical Processing type carbide precipitate while evidence of a finely divided general precipitate can be seen with the grain.

As a result of the process evaluation, a procedure consisting of 1/2 hour anneal at 3600°F followed by a reduction of 60 percent, with a final anneal of 1/2 hour at 3000°F was chosen. Material sufficient to produce four creep specimens was processed accordingly.

4.3.2 Creep Testing

The material processed to 0.040 inch thick sheet using the procedure developed in the thermal-mechanical evaluation was machined from four creep test specimens. Creep tests were conducted at two stress levels at two temperatures. The creep data are listed in Table 5.

4.3.3 Effect of GTA Welding on The Creep Properties of ASTAR-811C

The objective of this evaluation was to determine the creep behavior of ASTAR-811C weld metal. ASTAR-811C sheet, 0.040 inch thick from heat NASV-20 was used for this study. Two strips approximately 1-1/2 inches wide by 6 inches long were annealed for 1/2 hour at 3600°F and helium gas quenched. A bead-on-plate weld was made on each sheet using welding procedures developed on previous programs[1,4]. The welding parameters used were as follows:

Speed	15 ipm
Current	140 Amps
Voltage	~18 Volts
Clamp Spacing	3/8 Inch

A weldmetal zone approximately 1/4 inch wide at the upper surface was produced in each sheet. Regular sheet creep specimens were machined with the 1/4 inch wide weld metal zone coincidental with the 1/4 inch wide gage section of the creep specimen. The specimens

TABLE 5

CREEP PROPERTIES OF ASTAR-811C SUBJECTED TO THERMAL MECHANICAL PROCESSING*

Tesr Time (°F)	Stress (KSI)	Strain On Loading (%)	Test Time (Hrs.)	Total Strain (%)	Secondary Creep Rate (%/Hr.)	Time To 1% Strain (Hrs.)	P***
2000	27.5	0.2	1283	.57	0.000184	5200	51.7
2000**	25.0	0.1	503	-	-	-	-
2100	25.0	-	1027	.39	0.00038	2560	53.0
2400	15.0	0.05	388	1.21	0.00290	344	56.7
2400	12.5	0.05	602	1.02	0.0016	620	57.5

* Annealed 1/2 Hour At 3600°F Helium Gas Quenched, Rolled 60%, Annealed 1/2 Hour At 3000°F and Furnace Cooled

** No Measureable Creep Temperature Increased To 2100°F

*** $P = T_{^{\circ}R} (17.3 + \text{Log } t_{1\%E}) \times 10^{-3}$

were surface ground to a final thickness of 0.035 inch to remove any surface irregularities. The creep specimens were then annealed 1 hour at 2400°F prior to creep testing. The weld creep test data are listed in Table 6.

TABLE 6

CREEP PROPERTIES OF GTA WELDED ASTAR-811C

Test Temp. (°F)	Stress (KSI)	Strain On Loading (%)	Test Time (Hrs.)	Total Strain (%)	Secondary Creep Rate (%/Hr.)	Time To 1% Strain (Hrs.)	P*
2000	27.5	0.18	1129	0.70	0.00046	2180	50.8
2000	25.0	0.15	850	0.30	0.000177	5650	51.8
2050	25.0	-	766	0.28	0.000365	2740	52.0
2400	15.0	0.10	386	1.84	0.00416	240	56.3
2400	12.5	0.10	592	1.29	0.0020	500	57.2

$^*P = T^o_R\ (17.3 + \mathrm{Log}\, t_{1\%E}) \times 10^{-3}$

4.4 DISCUSSION OF RESULTS

The results of the ASTAR-811C creep testing conducted under this program are summarized in Figure 34. The dash-line curves represent the creep data for the stress and temperature change creep tests conducted as part of this program. The solid-line curves represent constant load and temperature data from prior work(1). The multiload-temperature creep tests tend to give results which were conservative compared to constant load, constant temperature test results. This behavior is not unexpected due to the nature of the load-temperature change tests. The time for one percent strain for each test condition was determined by extrapolating creep rates. Small strain increments which were necessary to cover the test rates

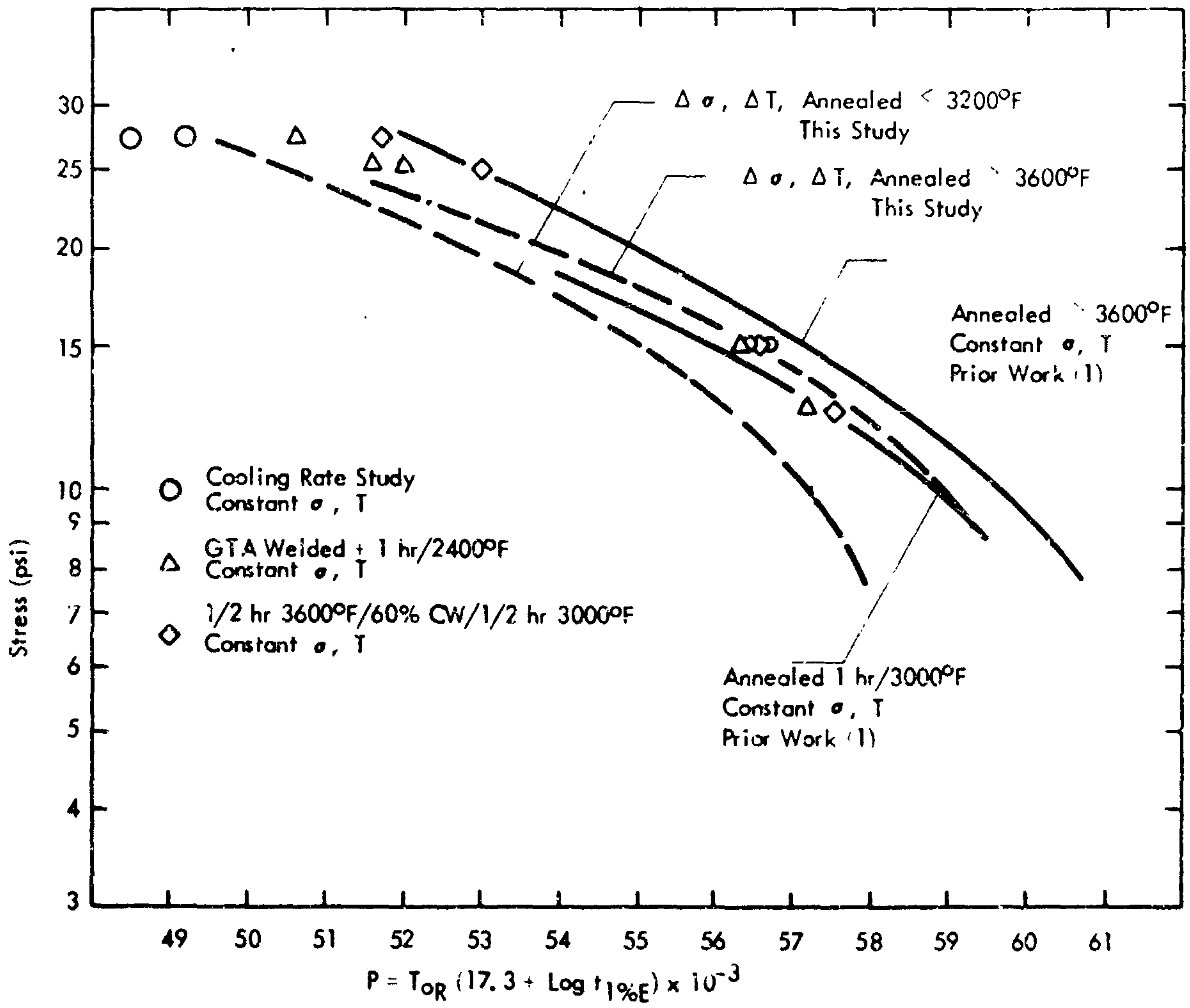


Figure 34. Summary Larson-Miller Plot of Creep Test Results for ASTAR-811C

and thus shift the Larson-Miller parameter to lower values. The open points represent constant stress and temperature results for the cooling rate, the thermal-mechanical processing and welding effect investigations. The test results were determined in many cases using actual time to one percent strain or extrapolating secondary creep rates. To permit valid comparison with multiload-temperature tests strain on loading and primary creep was neglected. At the lower temperatures and higher stress levels, the material subjected to various cooling rates from the final 3600°F heat treatment exhibited creep behavior which was significantly lower than expected. The fine grained material produced to retain the high temperature form of the carbide precipitate displayed creep properties at 2000°F which were comparable to the larger grained material. The creep properties of the welded ASTAR-811C at 2000°F was slightly lower than the larger grained material. The creep curves for the 2000°F, 27.5 ksi test conditions are shown in Figure 35. The creep curves for the cooling rate study show in addition to a larger secondary creep rate, a significant primary creep stage. There was no indication of a primary creep stage for the welded material. The small grained material produced by the thermal-mechanical processing study produced a small but extended primary creep stage. At the 2400°F test temperature and 15 ksi stress level, the data were clustered, showing little effect of material condition. The creep curves, Figure 36, were similar in appearance and typical of the low stress-high temperature creep behavior of ASTAR-811C. The higher temperature apparently minimizes the effect of cooling rate and grain size on the creep behavior of ASTAR-811C.

The conclusions which can be drawn from the results obtained under this program must be evaluated in light of the quantity of data generated. In the initial investigation of final annealing temperature and grain size, sufficient data were produced to permit firm conclusions to be drawn. In the remaining investigations, the limited number of test specimens available restricted the conclusion to the delineation of trends.

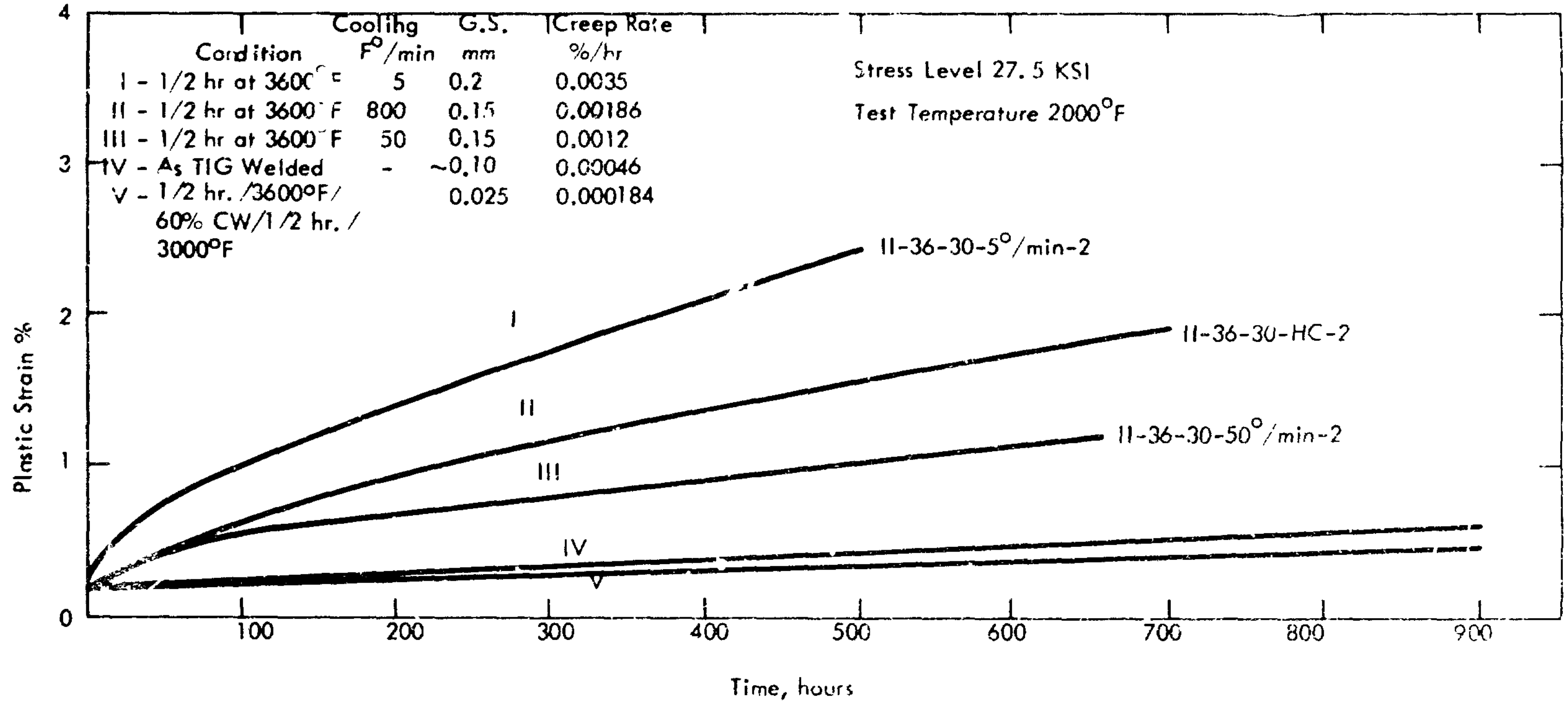


Figure 35. Effect of Cooling Rate on the Creep Strength (2000°F - 27.5 KSI) of ASTAR-811C

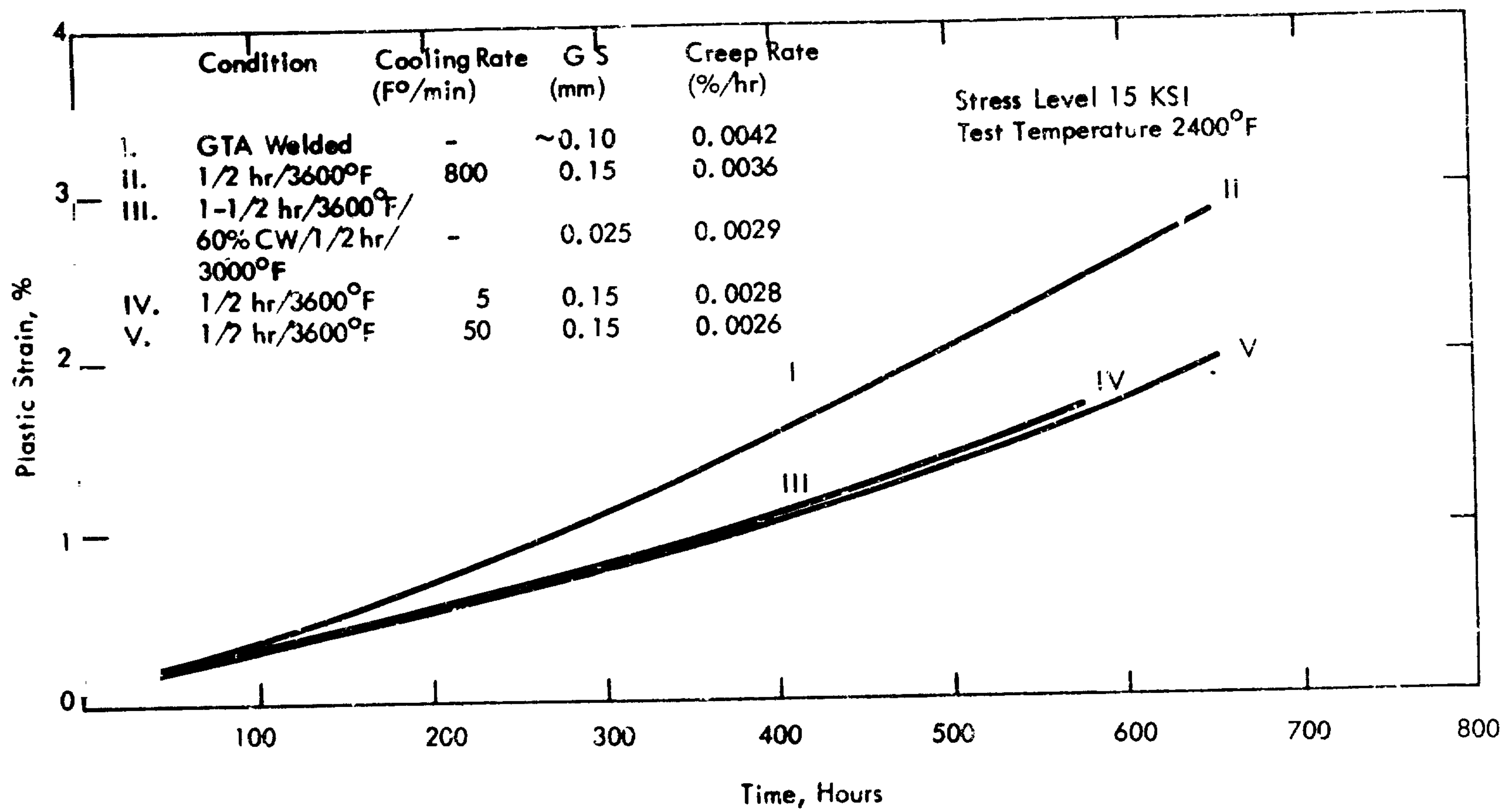


Figure 36. Effect of Cooling Rate on the Creep Strength (2400°F - 15KSI) of ASTAR-811C

5.0 CONCLUSIONS

1. The final annealing temperature of ASTAR-811C has a strong influence on creep properties. ASTAR-811C sheet annealed above the carbon solvus, $\geq$ 3600°F, exhibited the creep rates in the range of 2000° to 2600°F ~1/5 of that for material annealed at 3000°F.

2. The precise role of carbon and the carbide phase, Ta_2C, remains undefined since the evidence of direct precipitate-dislocation interaction detected in creep specimens tested at 2000 and 2400°F was insufficient to account for the strengthening increments measured. It would appear, however, that grain boundary precipitate interactions may be controlling.

3. The effect of grain size on the creep behavior of ASTAR-811C appears to be minimal. No significant effect on creep rate was noted for grain sizes which ranged from 0.025 to 0.25 mm, provided the disposition of carbide phase was the same.

4. Cooling rate from final annealing temperature had little effect on the 2000 and 2400°F creep behavior of ASTAR-811C sheet.

5. GTA welding of ASTAR-811C sheet does not adversely effect the creep behavior.

6.0 RECOMMENDATIONS

Based on the conclusions that the carbide morphology is controlling the creep behavior of ASTAR-811C, the following heat treatment is recommended to produce optimum creep properties in ASTAR-811C sheet.

- Intermediate anneal 1/2 hour at 3600°F*, rapid cool, (<100F°/min) final reduction (~60%) by rolling at room temperature, final anneal at 1/2 hour at 3000°F.

(NOTE: Same creep properties can be achieved by final annealing only at 3600°F for 1/4 hour followed by rapid cool. However, final grain size will be approximately five times that of recommended treatment.)

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*Although current industrial vacuum heat treating furnaces are generally limited to a maximum temperature of 1700°F at 1×10^{-5} torr, demonstrated technology exists for heat treating at temperatures up to 4000°F at pressure $\leq 1 \times 10^{-8}$ torr. Thus if a requirement would arise for processing large quantities of ASTAR-811C material with optimum creep properties, the heat treatment capability could be provided. Laboratory heat treatment at temperatures up to 3600°F and 1×10^{-5} torr of tubular shapes up to 1 1/2 inch in diameter x 10 feet long are currently available at the Astronuclear Laboratory.

REFERENCES

(1) R. W. Buckman and R. . Goodspeed, "Development of Precipitation Strengthened Tantalum Base Alloys", NASA CR-1642, WANL PR-Q-017

(2) D. Garafalo, "Fundamentals of Cree, and Creep-Rupture in Metals", MacMillan Series in Materials Science, 1965

(3) R. W. Buckman and J. J. Hetherington, "Apparatus for Determining Creep Behavior Under Conditions of Ulta-High Vacuum", Review of Scientific Instruments, Vol. 37, No. 8, pp 999 - 10003, August, 1966

(4) R. E. Gold and G. G. Lessman, "Influence of Restraint and Thermal Exposure on Welds in T-111 and ASTAR-811C," NASA CR-72858, WANL-PR(VVV)-001

APPENDIX A

TIME-ELONGATION CURVES FOR TEMPERATURE-STRESS CHANGE TESTS ON ASTAR-811C

* Specimen Notation

I-30-5-H-C

I - denotes Task I specimen

30 - annealing temperature x 10^{-2} in ^{o}F

5 - annealing time in minutes

H - helium gas cooled

C - creep specimen

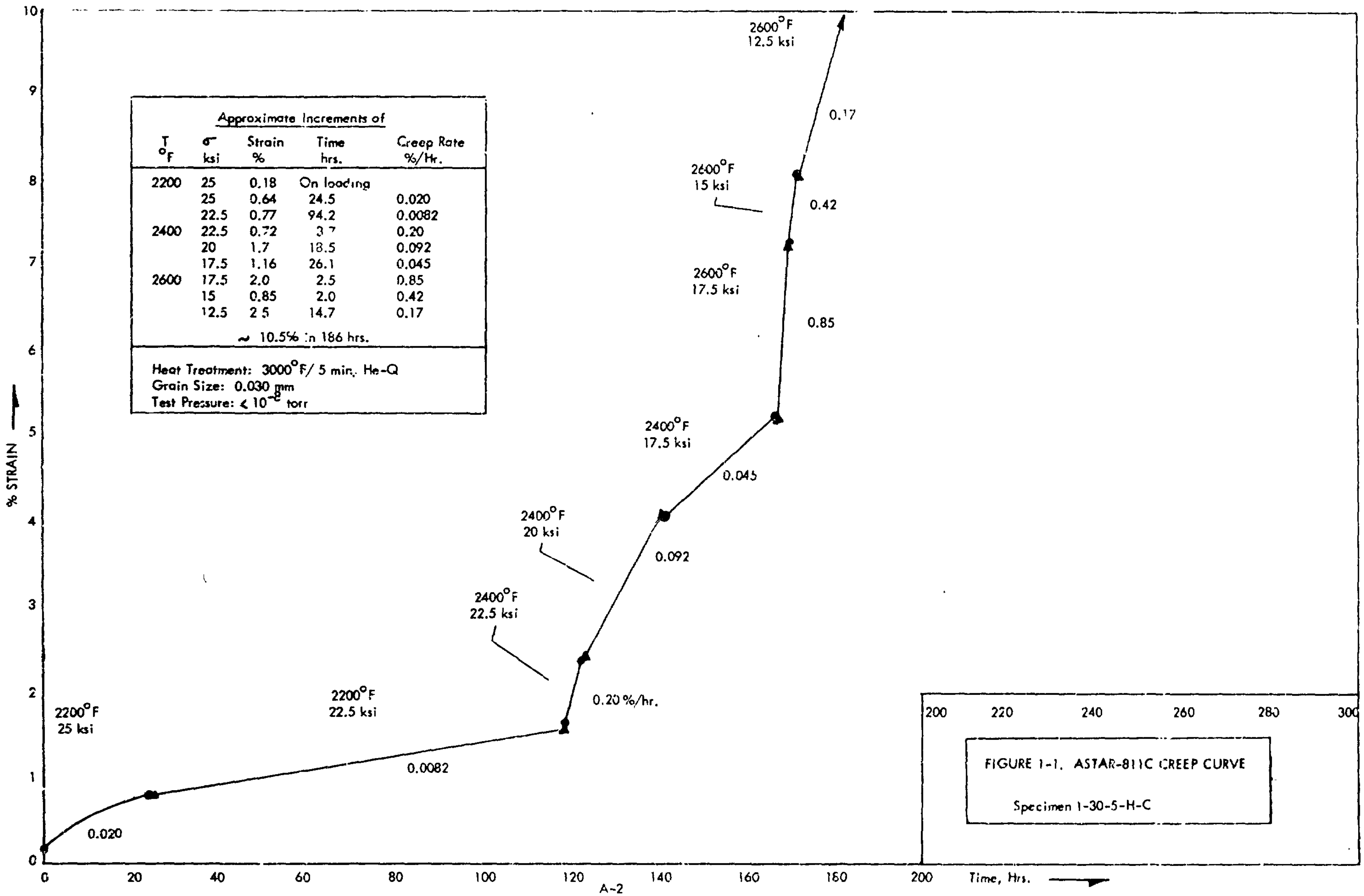
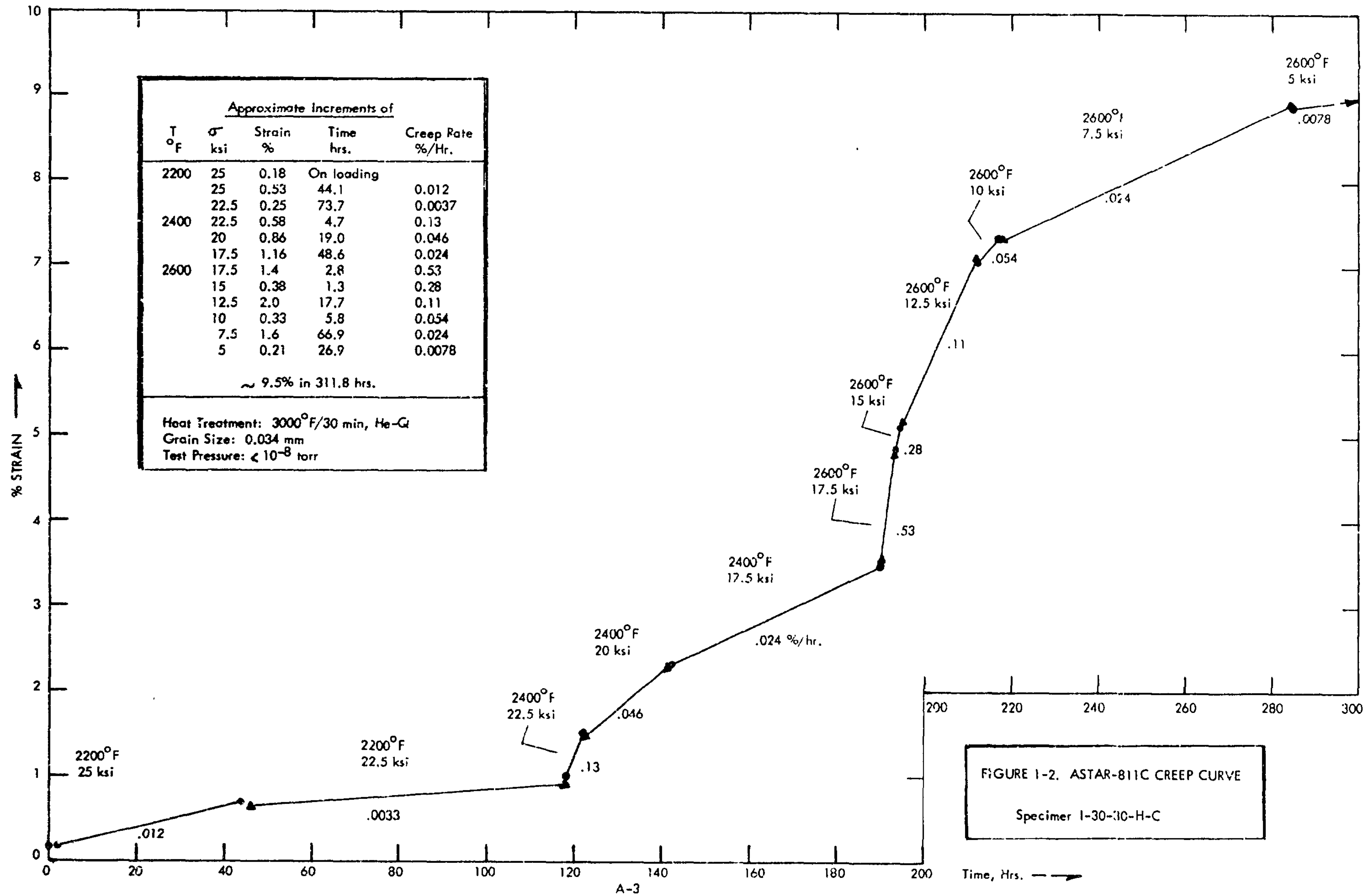


FIGURE 1-1. ASTAR-811C CREEP CURVE

Specimen 1-30-5-H-C



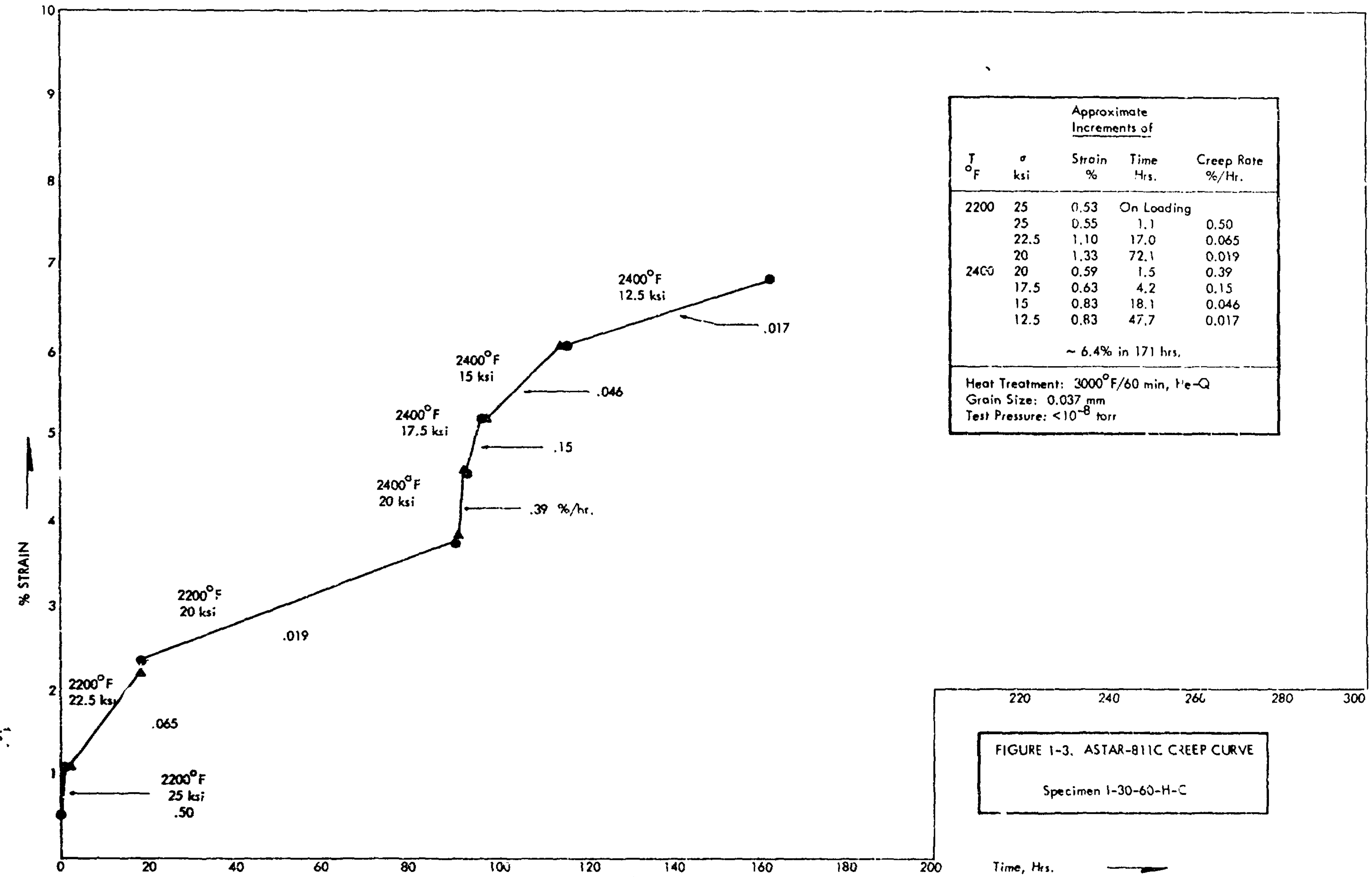
T °F	σ ksi	Approximate Increments of Strain %	Time hrs.	Creep Rate %/Hr.
2200	25	0.18	On loading	
	25	0.53	44.1	0.012
	22.5	0.25	73.7	0.0037
2400	22.5	0.58	4.7	0.13
	20	0.86	19.0	0.046
	17.5	1.16	48.6	0.024
2600	17.5	1.4	2.8	0.53
	15	0.38	1.3	0.28
	12.5	2.0	17.7	0.11
	10	0.33	5.8	0.054
	7.5	1.6	66.9	0.024
	5	0.21	26.9	0.0078

~ 9.5% in 311.8 hrs.

Heat Treatment: 3000°F/30 min, He-G
Grain Size: 0.034 mm
Test Pressure: < 10^{-8} torr

FIGURE 1-2. ASTAR-811C CREEP CURVE

Specimen 1-30-10-H-C



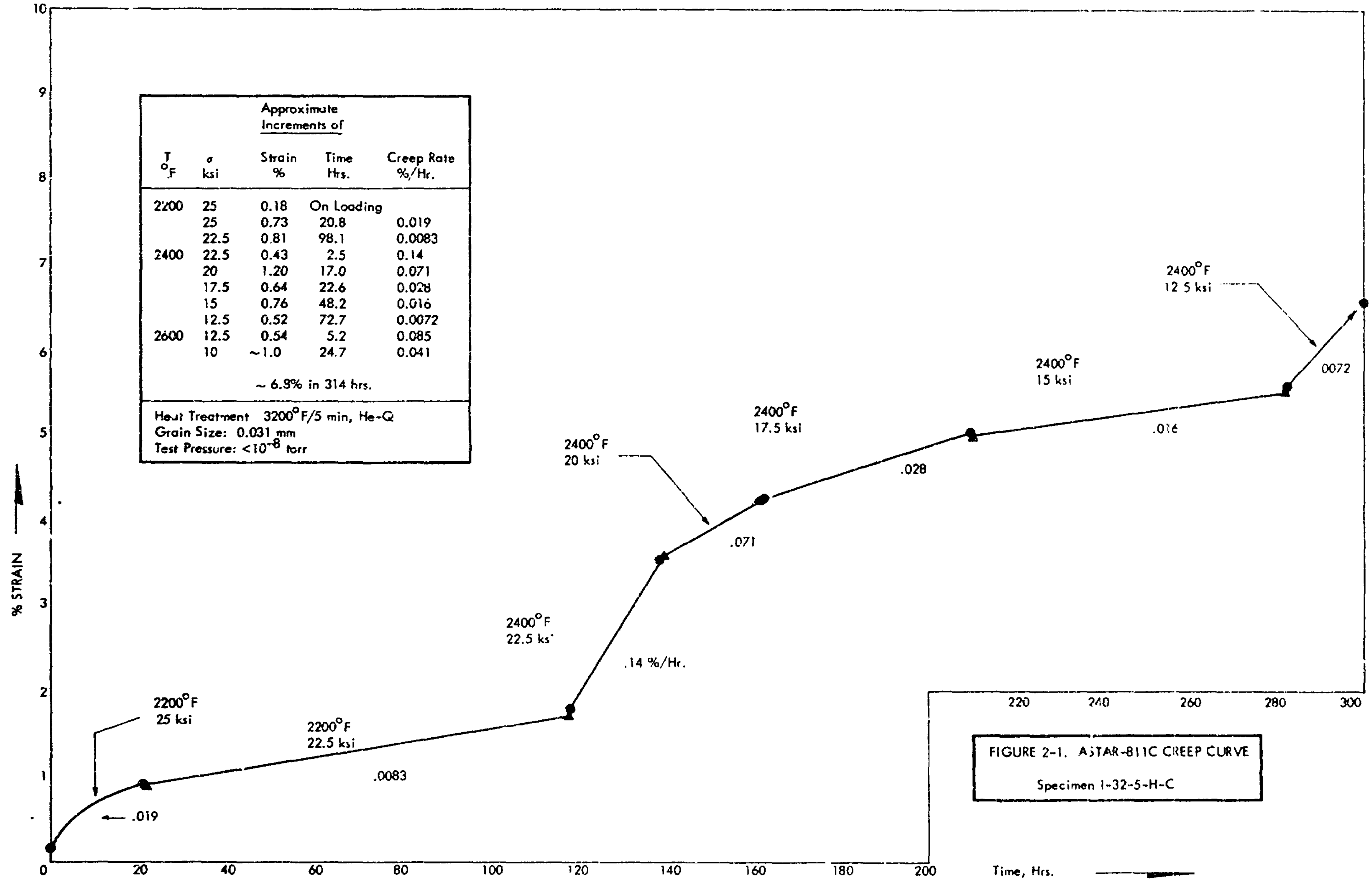
T °F	σ ksi	Approximate Increments of Strain %	Approximate Increments of Time Hrs.	Creep Rate %/Hr.
2200	25	0.53	On Loading	
	25	0.55	1.1	0.50
	22.5	1.10	17.0	0.065
	20	1.33	72.1	0.019
2400	20	0.59	1.5	0.39
	17.5	0.63	4.2	0.15
	15	0.83	18.1	0.046
	12.5	0.83	47.7	0.017

~ 6.4% in 171 hrs.

Heat Treatment: 3000°F/60 min, He-Q
Grain Size: 0.037 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 1-3. ASTAR-811C CREEP CURVE

Specimen 1-30-60-H-C



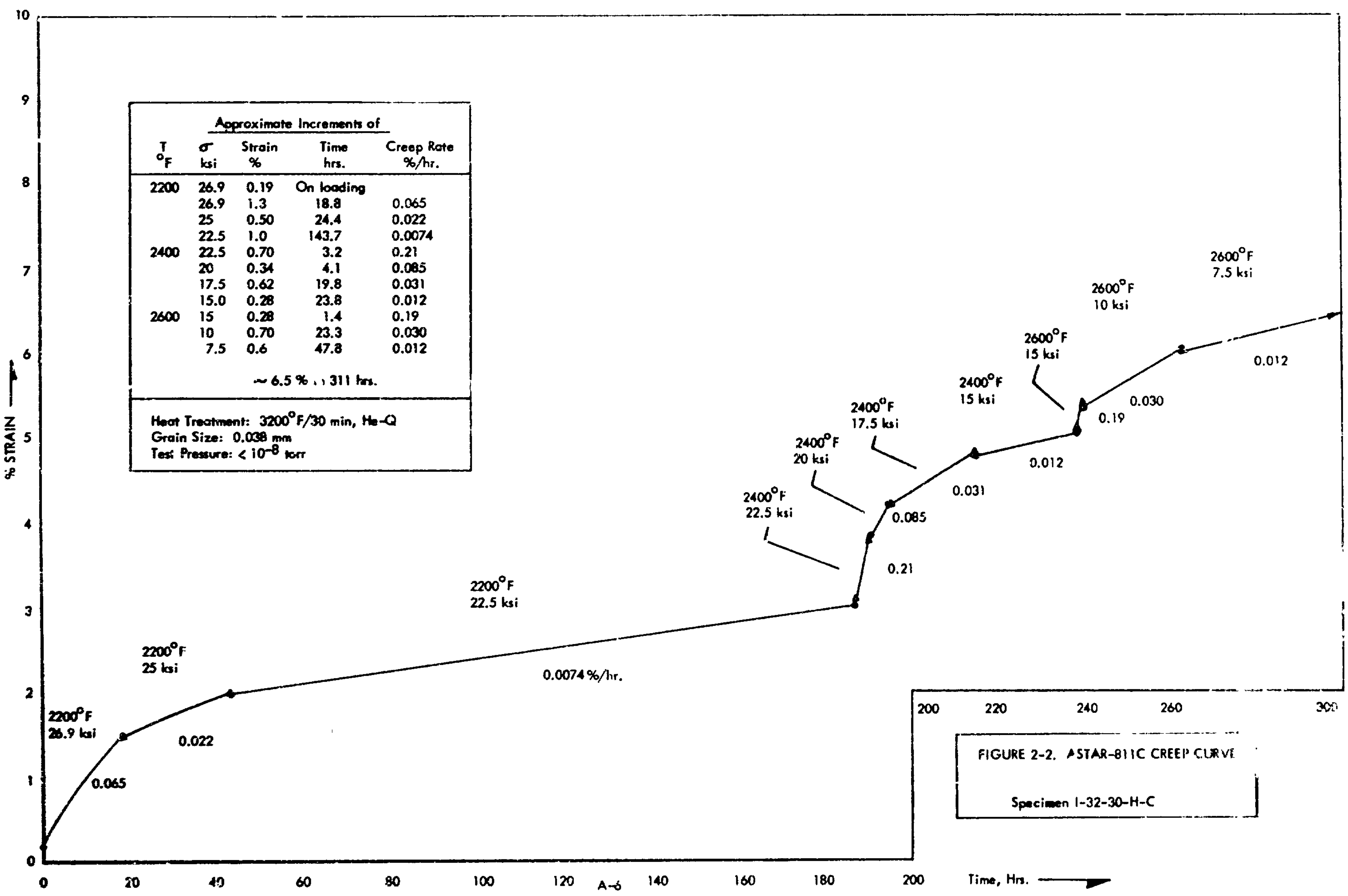
T °F	σ ksi	Approximate Increments of Strain %	Approximate Increments of Time Hrs.	Creep Rate %/Hr.
2200	25	0.18	On Loading	
	25	0.73	20.8	0.019
	22.5	0.81	98.1	0.0083
2400	22.5	0.43	2.5	0.14
	20	1.20	17.0	0.071
	17.5	0.64	22.6	0.028
	15	0.76	48.2	0.016
	12.5	0.52	72.7	0.0072
2600	12.5	0.54	5.2	0.085
	10	~1.0	24.7	0.041

~ 6.9% in 314 hrs.

Heat Treatment 3200°F/5 min, He-Q
Grain Size: 0.031 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 2-1. ASTAR-811C CREEP CURVE

Specimen I-32-5-H-C



Approximate Increments of

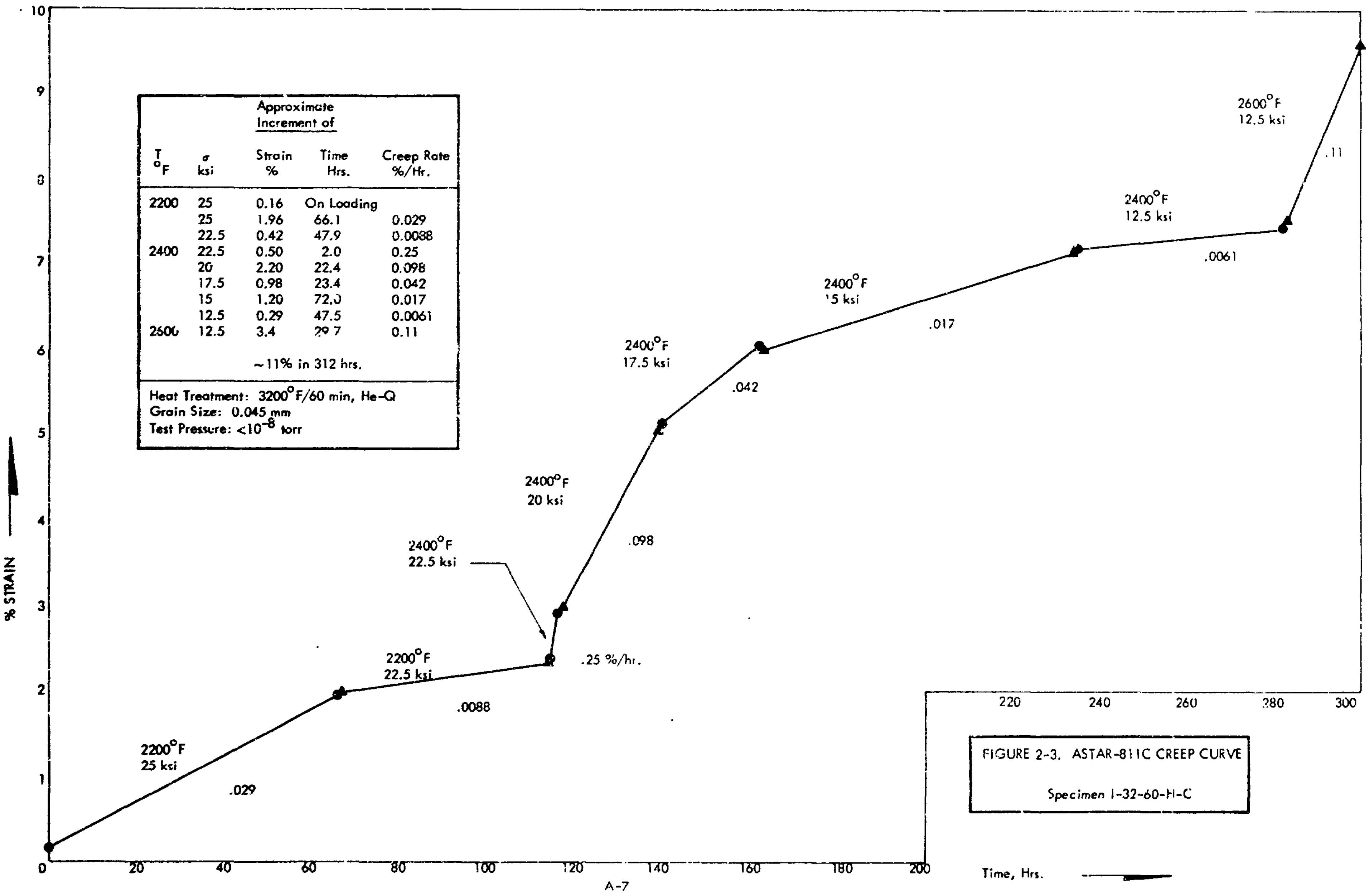
T °F	σ ksi	Strain %	Time hrs.	Creep Rate %/hr.
2200	26.9	0.19	On loading	
	26.9	1.3	18.8	0.065
	25	0.50	24.4	0.022
	22.5	1.0	143.7	0.0074
2400	22.5	0.70	3.2	0.21
	20	0.34	4.1	0.085
	17.5	0.62	19.8	0.031
	15.0	0.28	23.8	0.012
2600	15	0.28	1.4	0.19
	10	0.70	23.3	0.030
	7.5	0.6	47.8	0.012

~ 6.5 % , , 311 hrs.

Heat Treatment: 3200°F/30 min, He-Q
Grain Size: 0.038 mm
Test Pressure: < 10^{-8} torr

FIGURE 2-2. ASTAR-811C CREEP CURVE

Specimen I-32-30-H-C



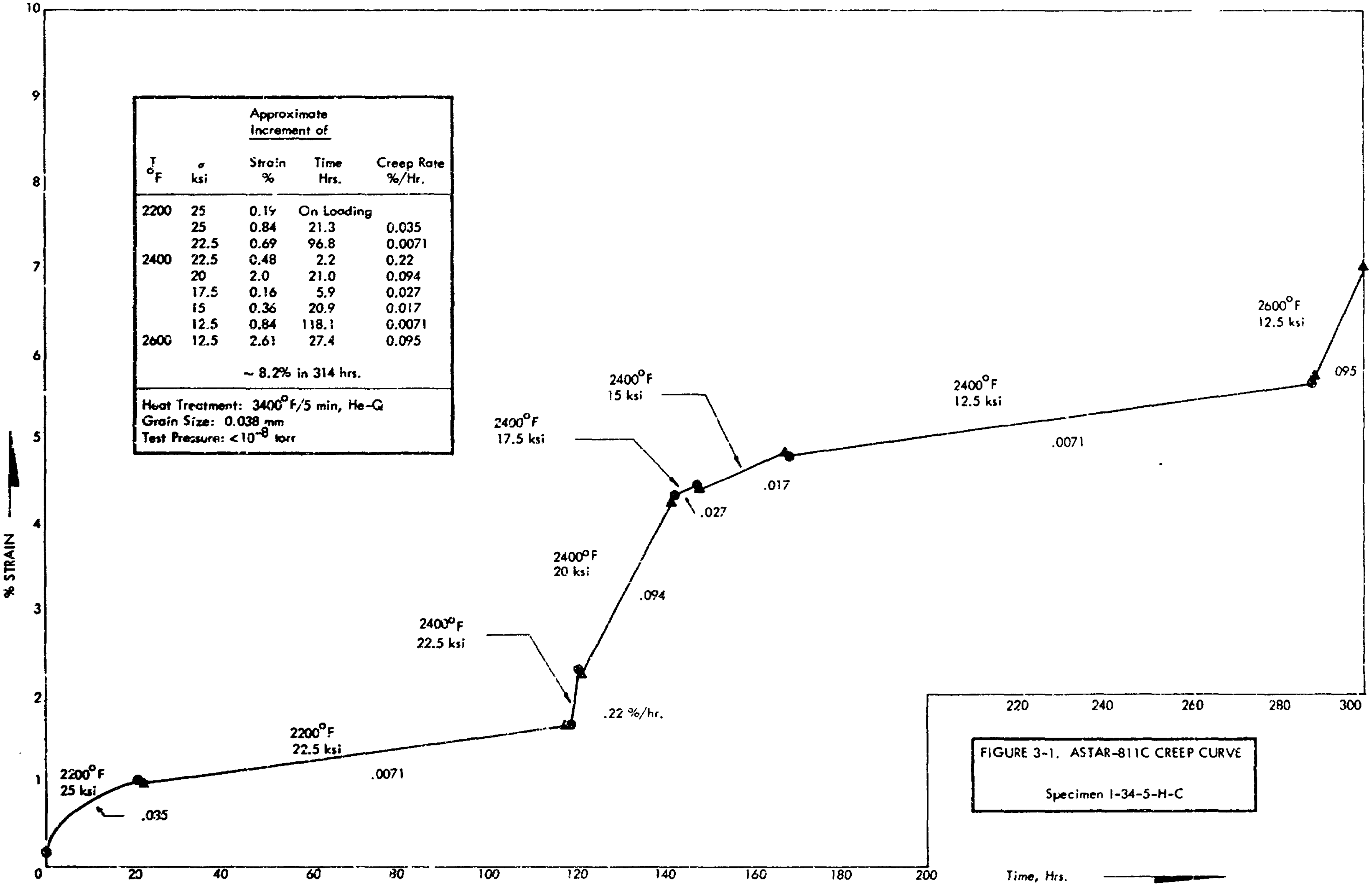
T °F	σ ksi	Approximate Increment of Strain %	Approximate Increment of Time Hrs.	Creep Rate %/Hr.
2200	25	0.16	On Loading	
	25	1.96	66.1	0.029
	22.5	0.42	47.9	0.0088
2400	22.5	0.50	2.0	0.25
	20	2.20	22.4	0.098
	17.5	0.98	23.4	0.042
	15	1.20	72.0	0.017
	12.5	0.29	47.5	0.0061
2600	12.5	3.4	29.7	0.11

~11% in 312 hrs.

Heat Treatment: 3200°F/60 min, He-Q
Grain Size: 0.045 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 2-3. ASTAR-811C CREEP CURVE

Specimen I-32-60-H-C



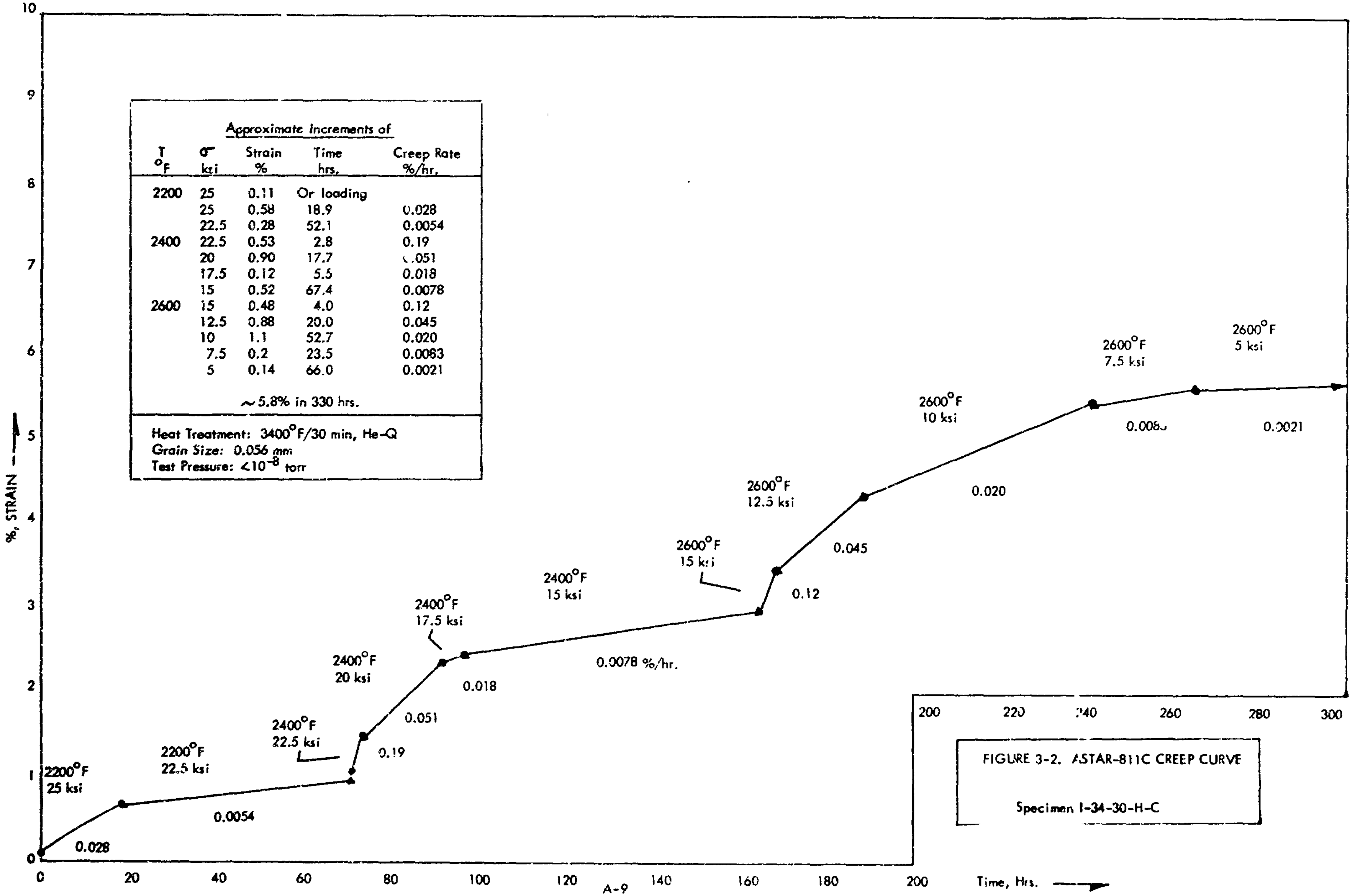
T °F	σ ksi	Approximate Increment of Strain %	Approximate Increment of Time Hrs.	Creep Rate %/Hr.
2200	25	0.19	On Loading	
	25	0.84	21.3	0.035
	22.5	0.69	96.8	0.0071
2400	22.5	0.48	2.2	0.22
	20	2.0	21.0	0.094
	17.5	0.16	5.9	0.027
	15	0.36	20.9	0.017
	12.5	0.84	118.1	0.0071
2600	12.5	2.61	27.4	0.095

~ 8.2% in 314 hrs.

Heat Treatment: 3400°F/5 min, He-Q
Grain Size: 0.038 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 3-1. ASTAR-811C CREEP CURVE

Specimen I-34-5-H-C



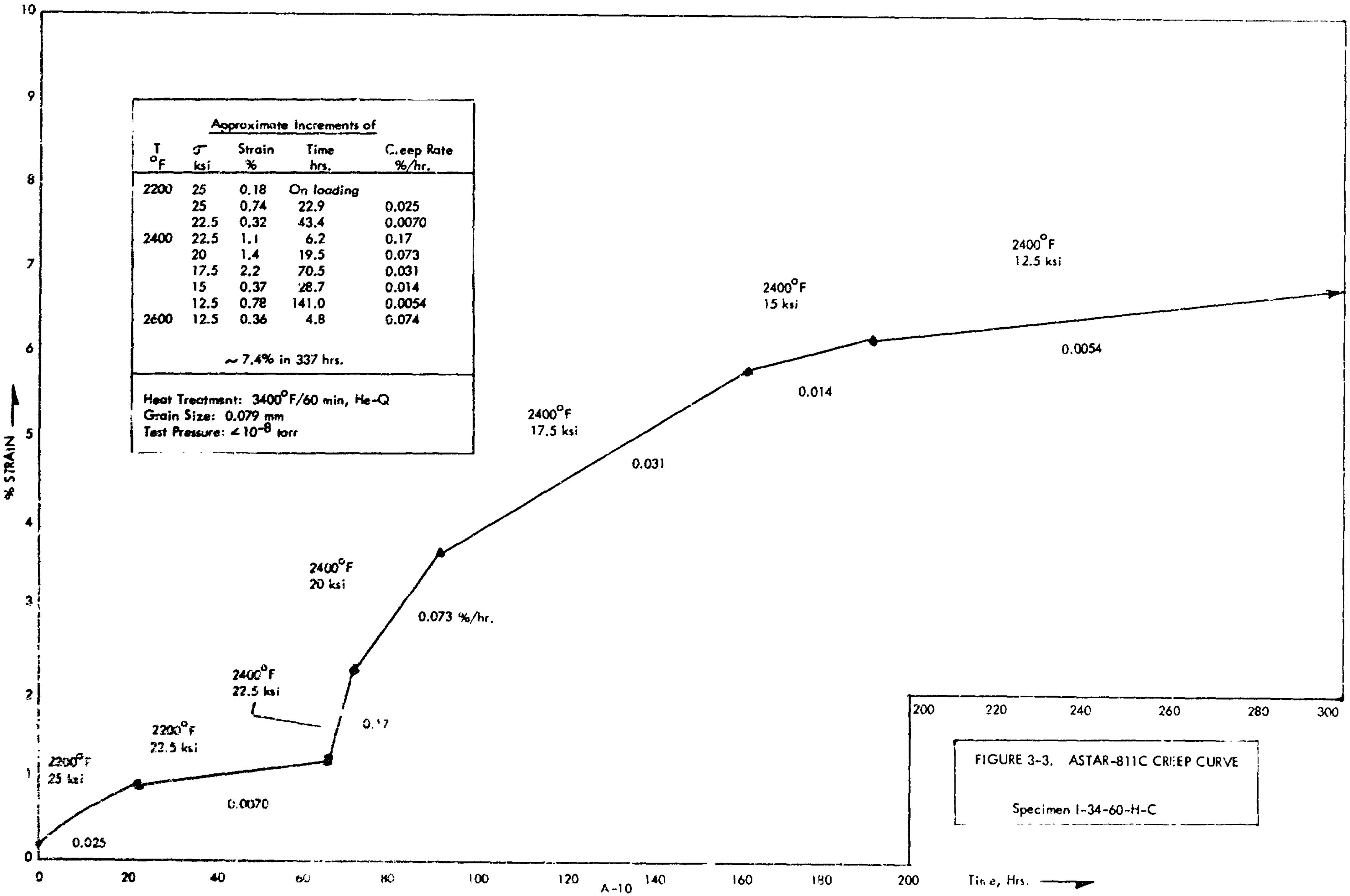
T °F	σ ksi	Approximate Increments of Strain %	Time hrs.	Creep Rate %/hr.
2200	25	0.11	On loading	
	25	0.58	18.9	0.028
	22.5	0.28	52.1	0.0054
2400	22.5	0.53	2.8	0.19
	20	0.90	17.7	0.051
	17.5	0.12	5.5	0.018
	15	0.52	67.4	0.0078
2600	15	0.48	4.0	0.12
	12.5	0.88	20.0	0.045
	10	1.1	52.7	0.020
	7.5	0.2	23.5	0.0083
	5	0.14	66.0	0.0021

~5.8% in 330 hrs.

Heat Treatment: 3400°F/30 min, He-Q
Grain Size: 0.056 mm
Test Pressure: <10^{-8} torr

FIGURE 3-2. ASTAR-811C CREEP CURVE

Specimen I-34-30-H-C



Approximate Increments of

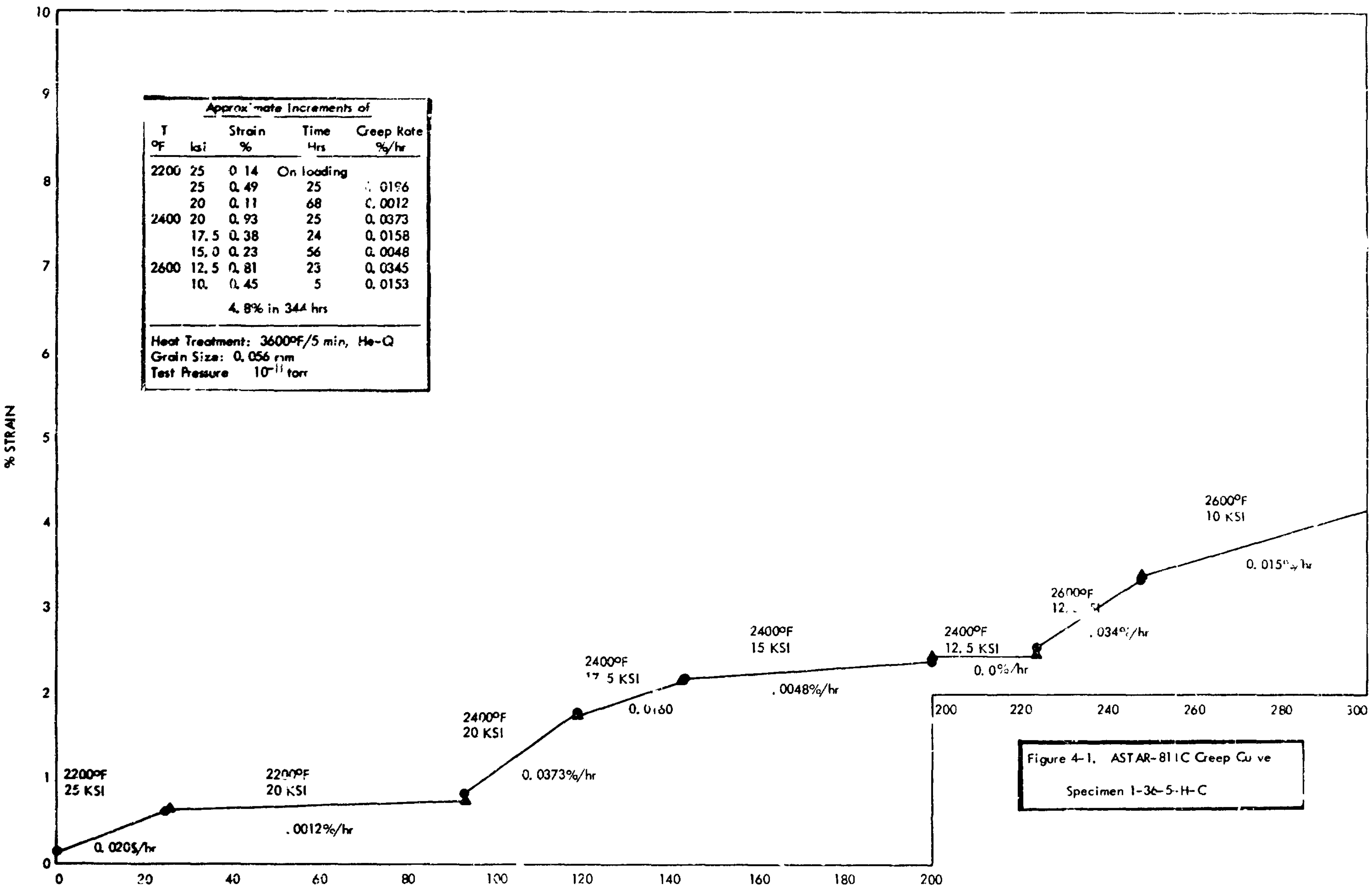
T °F	σ ksi	Strain %	Time hrs.	Creep Rate %/hr.
2200	25	0.18	On loading	
	25	0.74	22.9	0.025
	22.5	0.32	43.4	0.0070
2400	22.5	1.1	6.2	0.17
	20	1.4	19.5	0.073
	17.5	2.2	70.5	0.031
	15	0.37	28.7	0.014
	12.5	0.78	141.0	0.0054
2600	12.5	0.36	4.8	0.074

~ 7.4% in 337 hrs.

Heat Treatment: 3400°F/60 min, He-Q
Grain Size: 0.079 mm
Test Pressure: < 10^{-8} torr

FIGURE 3-3. ASTAR-811C CREEP CURVE

Specimen I-34-60-H-C



T °F	ksi	Strain %	Time Hrs	Creep Rate %/hr
2200	25	0 14	On loading	
	25	0. 49	25	. 0196
	20	0. 11	68	0. 0012
2400	20	0. 93	25	0. 0373
	17. 5	0. 38	24	0. 0158
	15. 0	0. 23	56	0. 0048
2600	12. 5	0. 81	23	0. 0345
	10.	0. 45	5	0. 0153

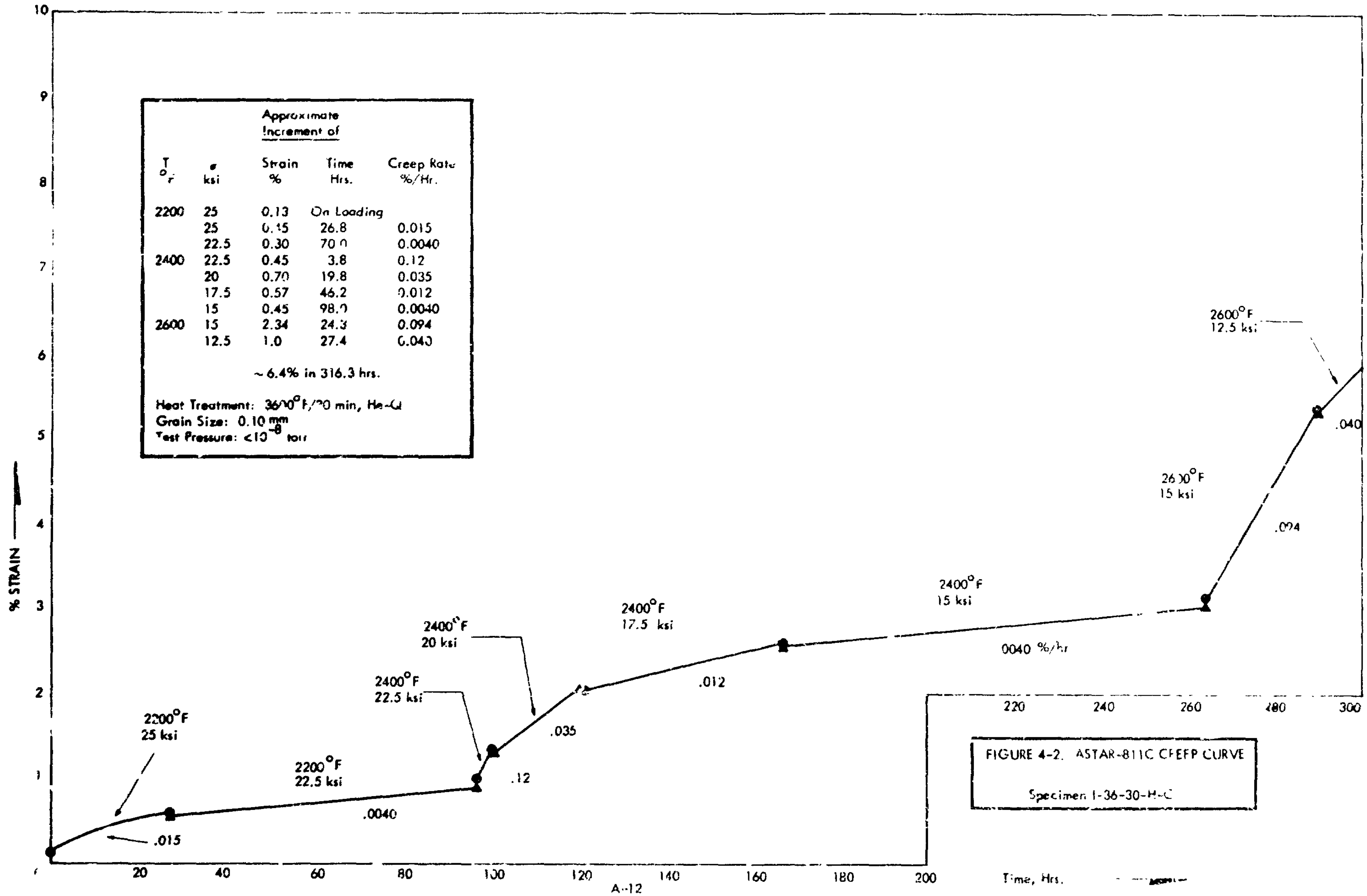
Approximate Increments of

4. 8% in 344 hrs

Heat Treatment: 3600°F/5 min, He-Q
Grain Size: 0. 056 mm
Test Pressure 10^{-11} torr

Figure 4-1. ASTAR-811C Creep Curve

Specimen 1-36-5-H-C



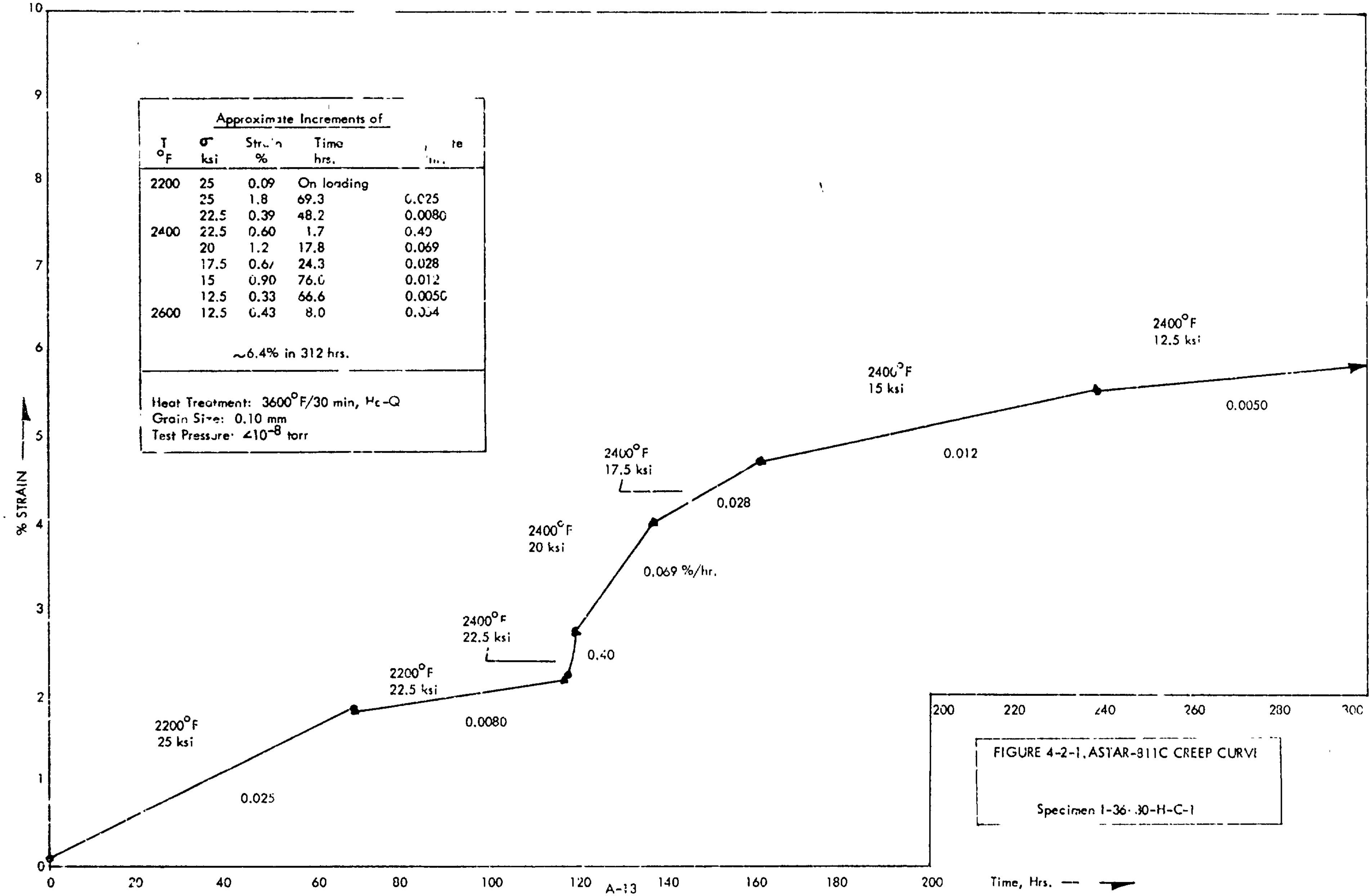
T °F	σ ksi	Approximate Increment of Strain %	Approximate Increment of Time Hrs.	Creep Rate %/Hr.
2200	25	0.13	On Loading	
	25	0.15	26.8	0.015
	22.5	0.30	70.0	0.0040
2400	22.5	0.45	3.8	0.12
	20	0.70	19.8	0.035
	17.5	0.57	46.2	0.012
	15	0.45	98.0	0.0040
2600	15	2.34	24.3	0.094
	12.5	1.0	27.4	0.040

~ 6.4% in 316.3 hrs.

Heat Treatment: 3600°F/20 min, He-Q
Grain Size: 0.10 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 4-2. ASTAR-811C CREEP CURVE

Specimen 1-36-30-H-C



Approximate Increments of

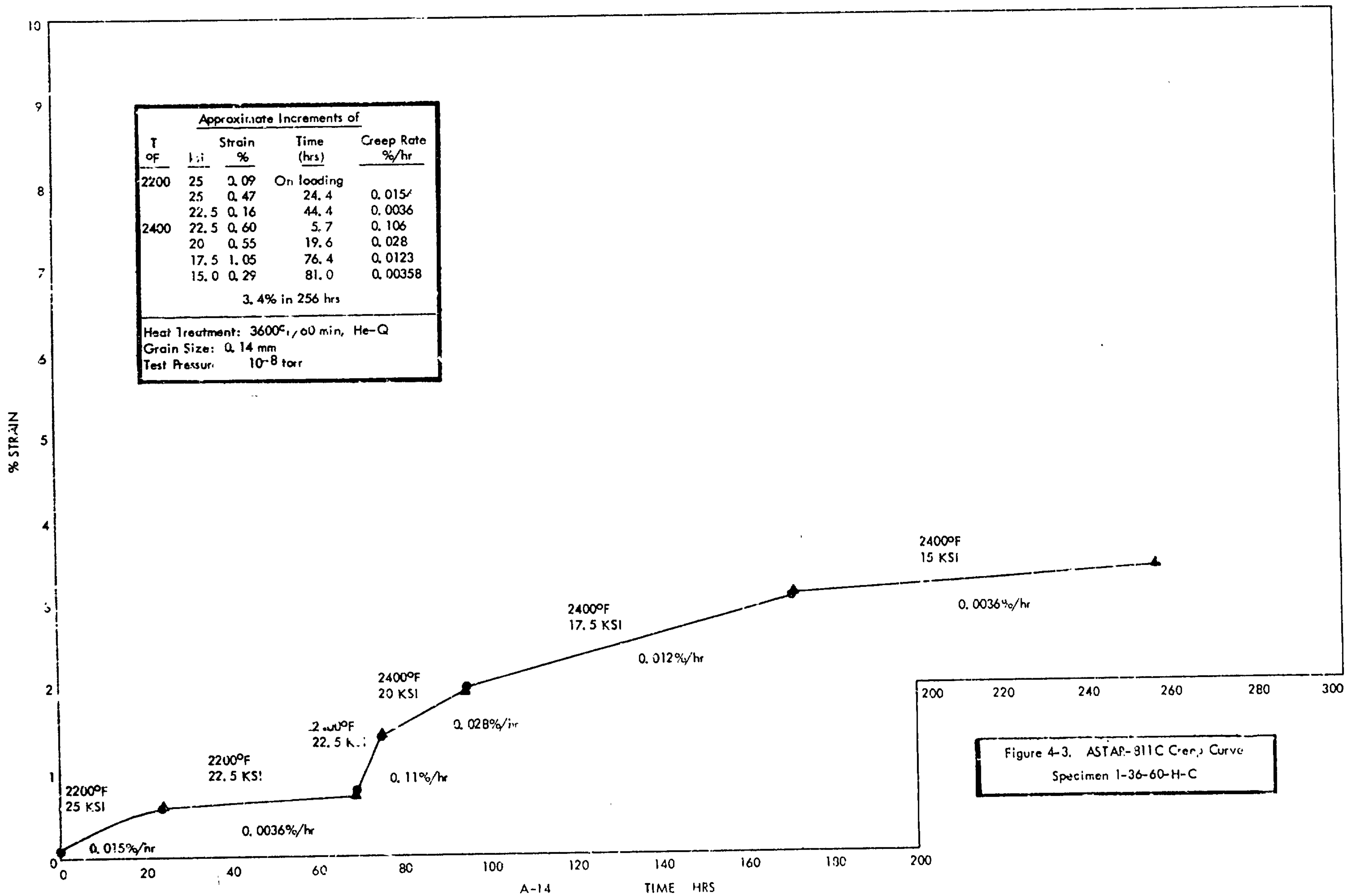
T °F	σ ksi	Strain %	Time hrs.	Rate %/hr.
2200	25	0.09	On loading	
	25	1.8	69.3	0.025
	22.5	0.39	48.2	0.0080
2400	22.5	0.60	1.7	0.40
	20	1.2	17.8	0.069
	17.5	0.67	24.3	0.028
	15	0.90	76.0	0.012
	12.5	0.33	66.6	0.0050
2600	12.5	0.43	8.0	0.054

~6.4% in 312 hrs.

Heat Treatment: 3600°F/30 min, He-Q
Grain Size: 0.10 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 4-2-1. ASTAR-811C CREEP CURVE

Specimen 1-36-30-H-C-1

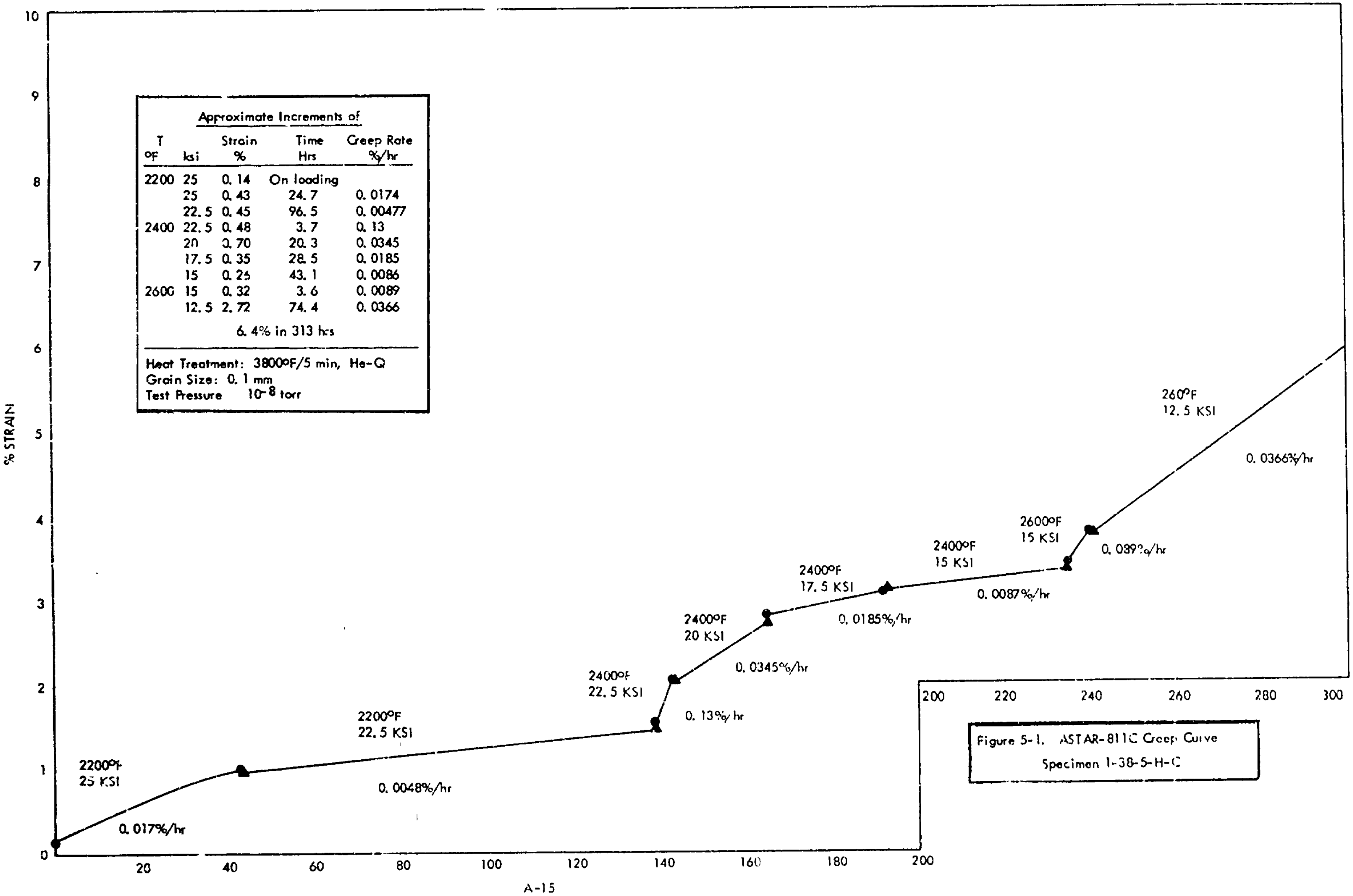


T °F	ksi	Strain %	Time (hrs)	Creep Rate %/hr
		Approximate Increments of		
2200	25	0. 09	On loading	
	25	0. 47	24. 4	0. 0154
	22. 5	0. 16	44. 4	0. 0036
2400	22. 5	0. 60	5. 7	0. 106
	20	0. 55	19. 6	0. 028
	17. 5	1. 05	76. 4	0. 0123
	15. 0	0. 29	81. 0	0. 00358

3. 4% in 256 hrs

Heat Treatment: 3600°F / 60 min, He-Q
Grain Size: 0. 14 mm
Test Pressure 10^-8 torr

Figure 4-3. ASTAR-811C Creep Curve
Specimen 1-36-60-H-C



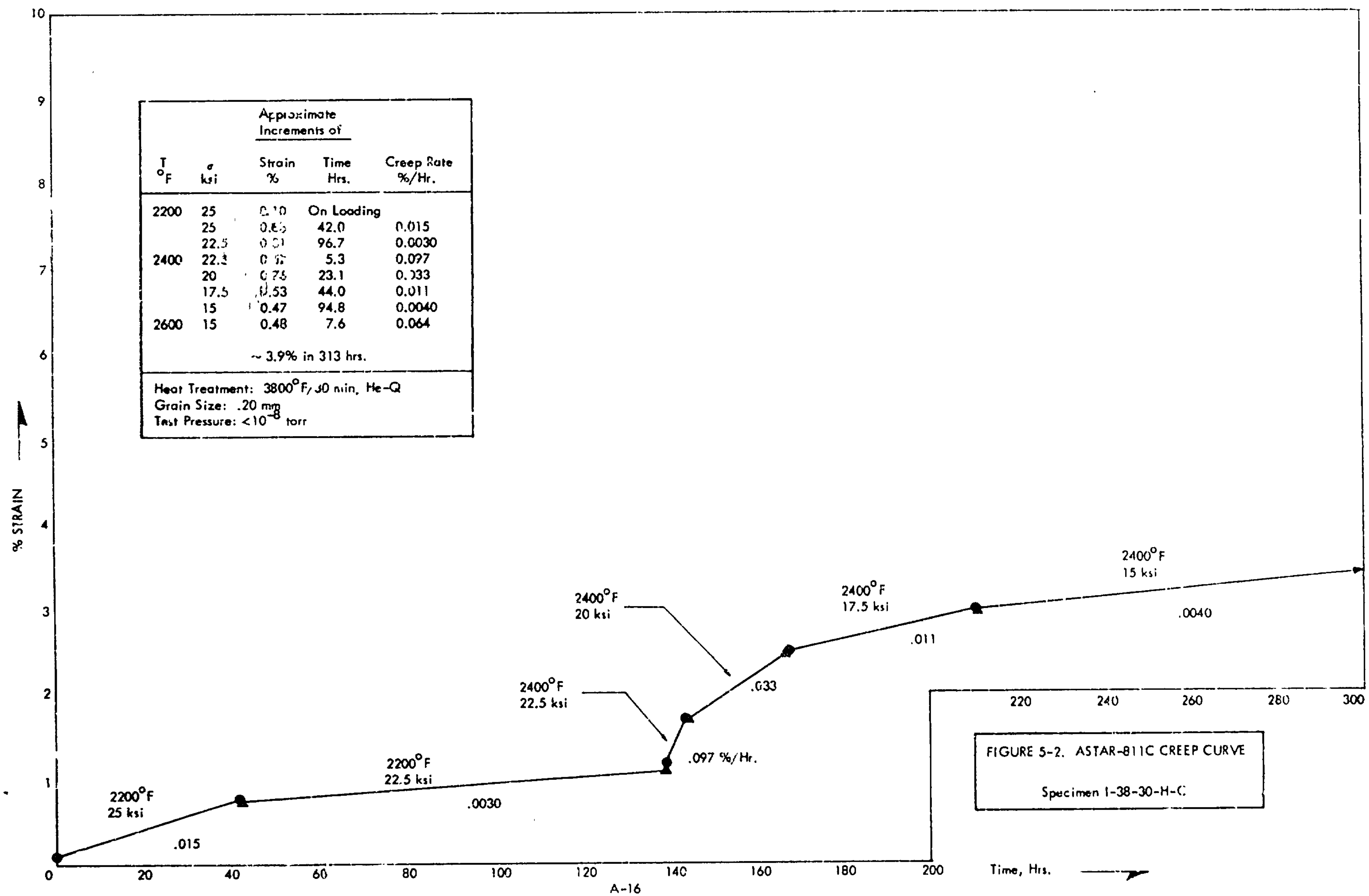
Approximate Increments of

T °F	ksi	Strain %	Time Hrs	Creep Rate %/hr
2200	25	0.14	On loading	
	25	0.43	24.7	0.0174
	22.5	0.45	96.5	0.00477
2400	22.5	0.48	3.7	0.13
	20	0.70	20.3	0.0345
	17.5	0.35	28.5	0.0185
	15	0.25	43.1	0.0086
2600	15	0.32	3.6	0.0089
	12.5	2.72	74.4	0.0366

6.4% in 313 hrs

Heat Treatment: 3800°F/5 min, He-Q
Grain Size: 0.1 mm
Test Pressure 10^{-8} torr

Figure 5-1. ASTAR-811C Creep Curve
Specimen 1-38-5-H-C



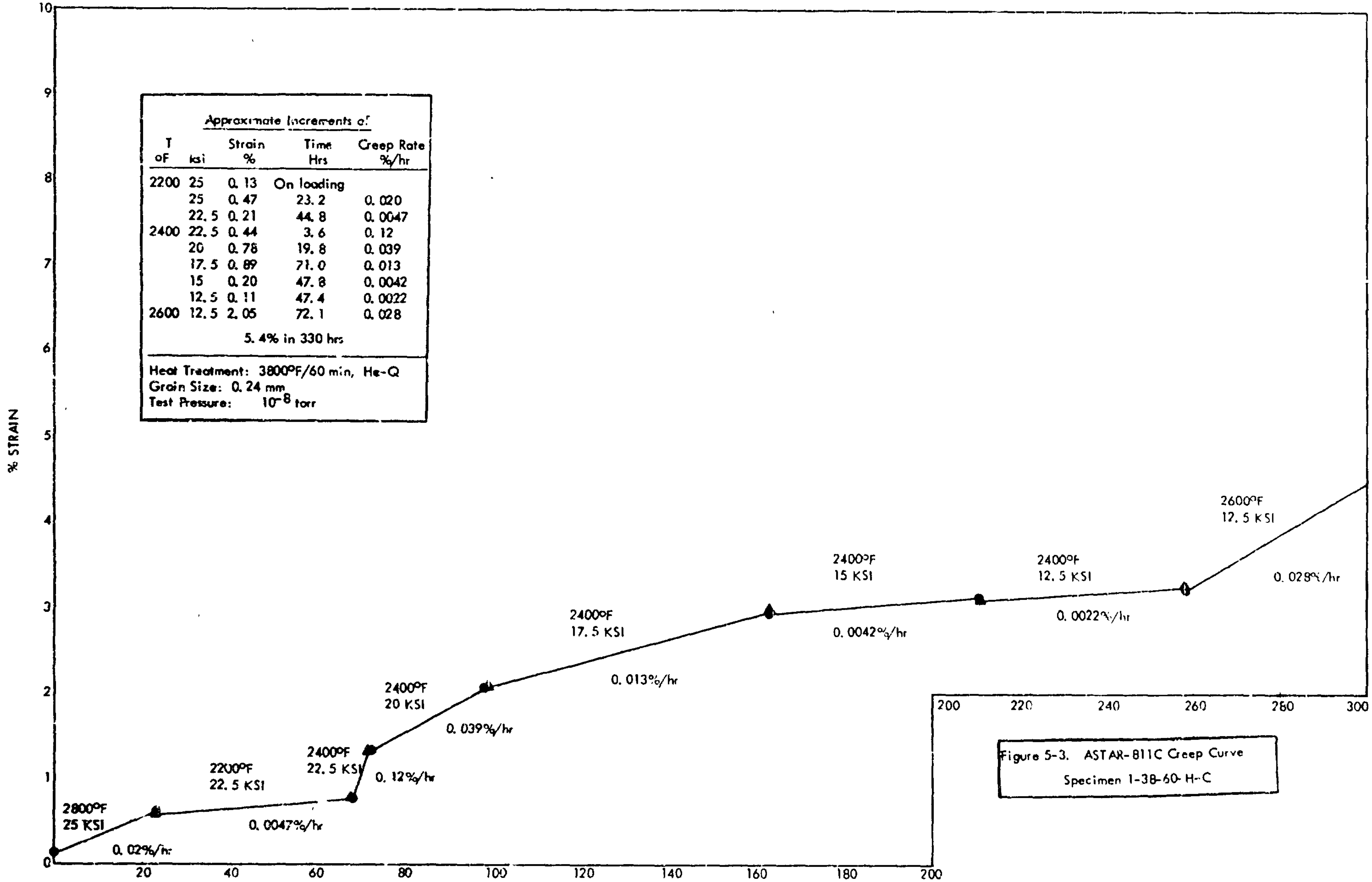
T °F	σ ksi	Approximate Increments of Strain %	Approximate Increments of Time Hrs.	Creep Rate %/Hr.
2200	25	[illegible]	On Loading	
	25	[illegible]	42.0	0.015
	22.5	[illegible]	96.7	0.0030
2400	22.5	[illegible]	5.3	0.097
	20	0.75	23.1	0.033
	17.5	0.53	44.0	0.011
	15	0.47	94.8	0.0040
2600	15	0.48	7.6	0.064

~ 3.9% in 313 hrs.

Heat Treatment: 3800°F/30 min, He-Q
Grain Size: .20 mm
Test Pressure: $<10^{-8}$ torr

FIGURE 5-2. ASTAR-811C CREEP CURVE

Specimen 1-38-30-H-C



T °F	ksi	Strain %	Time Hrs	Creep Rate %/hr
2200	25	0.13	On loading	
	25	0.47	23.2	0.020
	22.5	0.21	44.8	0.0047
2400	22.5	0.44	3.6	0.12
	20	0.78	19.8	0.039
	17.5	0.89	71.0	0.013
	15	0.20	47.8	0.0042
	12.5	0.11	47.4	0.0022
2600	12.5	2.05	72.1	0.028

5.4% in 330 hrs

Heat Treatment: 3800°F/60 min, He-Q
Grain Size: 0.24 mm
Test Pressure: 10^{-8} torr

Figure 5-3. ASTAR-811C Creep Curve
Specimen 1-38-60-H-C