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STRUCTURAL AND MECHANICAL EFFECTS OF INTERSTITIAL SINKS

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M. J. Klein
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Interim Technical Report
March 8, 1968 through March 8, 1969

April 1969

Prepared under Contract No. NAS 7-469
Solar Division of International Harvester Company

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National Aeronautics and Space Administration
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ABSTRACT

The contribution of solutes in refractory alloys to creep strength is being studied through their influence on the activation energy for creep of H. During this period some of the factors that affect H in columbium and alloys of columbium and tantalum were studied. Both the interstitial concentration and the structure affect H. The results suggest that the effect of interstitials is confined to temperatures below 0.5 - 0.6 T_M and that H approaches a constant value as the structure approaches an equilibrium configuration for the condition of the test. Activation energy - temperature profiles were determined for the columbium-base alloys D43, C103, C129Y, and Cb-1Zr and for the tantalum-base alloy T222, in the temperature range 0.5 to 0.7 T_M. Changes in H for these alloys are being correlated with their creep strengths to determine the significance of the observed variations in H.

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ILLUSTRATIONS

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INTRODUCTION

This is the Third Interim Technical Report for Contract NAS-7-469, "Structural Effects of Interstitial Sinks", covering the period March 8, 1968 through March 8, 1969.

The first part of this program was primarily concerned with the change in structure, strength, and interstitial concentration in a refractory alloy induced by an interstitial sink (a reactive metal in which interstitials concentrate). The loss of carbon from D43 (a columbium-base alloy strengthened with a carbide dispersion) to a titanium sink was correlated with the creep behavior of the D43. The creep rate near 0.5 T_M was found to be inversely proportional to the carbon lost to the interstitial sink.

In subsequent work the scope of the program was broadened to include the effect of interstitial and substitutional solutes on the creep of columbium- and tantalum-base alloys. Since creep is thermally activated, the creep rate and therefore the creep strength, is a sensitive function of the magnitude of the activation energy for creep. Therefore, the influence of solutes on creep strength is being studied through their effect on this parameter. This phase of the investigation should provide information concerning the role that various solutes play in strengthening refractory metals; information of value in selecting solutes to optimize the high-temperature strength of alloys.

During the first part of the period covered by this report, the influence of stress, structure and interstitial concentration on the activation energies for creep of columbium and D43 was studied in some detail. The work on unalloyed columbium was undertaken to provide a basis for comparing the effect of substitutional solutes in columbium-base alloys on their activation energies. The effects of structure and stress were studied because these factors could obscure the influence of solutes on the activation energy. During the latter part of this period activation energy - temperature profiles were determined for some columbium-base alloys (C103, C129Y and Cb-1Zr) and the tantalum-base alloy, T222.

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PAST WORK

In many practical applications it is necessary to use a refractory alloy strengthened with interstitial dispersions in contact with more reactive metals at elevated temperatures. Under these conditions the interstitial elements in the refractory alloy tend to concentrate in the more reactive metal and the alloy is weakened. In the first phase of this program (First Interim Report, June 8, 1966 - June 8, 1967), this effect was studied in D43 (Cb-10W-1Zr-0.1C) in contact with titanium. Changes in the grain structure of D43 were found to correlate with a reduction in the carbon concentration in the alloy and an increase in the carbon concentration in the titanium foil. These structural changes were caused by the dissolution of structure-stabilizing carbides induced by the migration of carbon to the titanium sink. It was shown that the reduction in carbon concentrations induced by the sinks could be predicted from free energy and solubility data.

The effects of a titanium sink on the interstitial concentration and recrystallization behavior of TZM and T222 were also studied (Second Interim Report, June 8, 1967 - March 8, 1968). This work revealed that titanium is an interstitial sink for carbon in TZM but not for carbon in T222. However, hafnium was found to be a sink for interstitials in the latter alloy.

Studies were also initiated during the period covered by the Second Interim Report to determine the effect of an interstitial sink on the creep strength of D43. Near 0.5 T_M (~2200° F) the creep rate of D43 can be expressed empirically by the equation

$$\epsilon^{\circ} = A\sigma^{n}e^{-H/RT}$$

where H is 112-118 K cal/mole (approximately that for self-diffusion), n is 7.7 to 9.5 and A is inversely proportional to the carbon concentration. An interstitial sink reduces the carbon concentration in the D43, thereby increasing the value of A and the creep rate. Electron transmission studies show that this reduction in carbon concentration reduces the number of carbide particles that obstruct dislocation glide. The reduction in the number of these particles correlates with an increase in the creep rate. The values of n and H do not appear to be affected by the action of the interstitial sink.

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EXPERIMENTAL PROCEDURES

Creep tests were made in a vacuum of 10^{-6} Torr under approximately constant tensile stress conditions using a Fullman-type lever arm (Ref. 1). Creep specimens were machined from 0.015- to 0.030-inch sheets of columbium and the various alloys. Specimen extensions over 1-inch gage lengths were continuously recorded using a high-temperature strain gage extensometer.

Differential temperature and stress measurements were used to determine temperature and stress dependencies of the creep rate. Activation energies were calculated from the changes in strain rate induced by abrupt shifts in the temperature during constant stress creep tests. The temperature shifts of 50 to 200°F required 15 to 90 seconds depending upon the temperature at which the shift was made and the magnitude of the shift. The dependence of strain rate on stress was determined by measuring the change in strain rate for incremental stress reductions during constant temperature tests.

The structure of the D43 was studied before and after creep testing using transmission electron microscopy. Thin foils were prepared from the gage lengths of creep specimens cooled from the test temperature under load to retain the elevated temperature dislocation structures.

It has been shown that columbium and columbium-base alloys are susceptible to contamination by gaseous interstitial elements during vacuum heat treatments (Ref. 2). In this regard, the problem of interstitial contamination seems to be less severe for tantalum alloys. However, it is unlikely that any of these alloys can be heat treated without some loss or gain of interstitial elements despite the precautions taken to control the heat treating environment. The results of oxygen and carbon analysis in D43 before and after creep testing illustrate the problem (Table I). The combined nitrogen and hydrogen concentrations which were ordinarily less than 50 ppm did not change in a detectable way in this alloy during creep testing. These analyses, which are the averages of duplicate analyses, show that only minor changes in carbon concentration occurred during creep testing except for specimen 4. This specimen, which was tested at 3100°F, lost a significant amount of its carbon concentration to the vacuum environment. Specimen 1 gained 100 ppm of oxygen, while specimens 2, 3 and 4, which were tested at progressively higher temperatures, lost increasing portions of

TABLE I

CARBON AND OXYGEN ANALYSES (ppm) BEFORE AND AFTER CREEP TESTING

	Specimens							
	Number 1		Number 2		Number 3		Number 4	
Element	Before Creep	After Creep 1700° F	Before Creep	After Creep 2200° F	Before Creep	After Creep 2750° F	Before Creep	After Creep 3100° F
Carbon	1230	1250	1280	1210	1172	1204	1267	400
Oxygen	210	310	240	210	590	380	462	52

their initial oxygen concentrations during testing. These results suggest that the primary contaminant is oxygen and that at higher temperatures, both carbon and oxygen are lost during creep testing. In this regard, other alloys with lower interstitial concentration than D43 might be expected to be more susceptible to interstitial contaminatio

Because of the probable loss or gain of interstitials during creep testing of columbium and columbium-base alloys, the effect of interstitials on the activation energy for creep was carefully investigated. It has been shown that foils placed around the gage length of specimens decrease impingement by gaseous interstitials during vacuum heat treatment, and this reduces contamination of the specimens with interstitials at lower temperatures (Ref. 3-5). This procedure may also reduce the loss of interstitials to the vacuum environment. Therefore, a number of specimens were tested in the wrapped and exposed conditions to determine the effect on the activation energy of changes in interstitial concentration during testing.

3.1 COLUMBIUM

The work on unalloyed columbium was undertaken to provide a base for comparing the effects of solutes in columbium alloys on H. As will be pointed out later, the published values of H for columbium are too inconsistent to be of much use in this study. Some of the factors that could account for the large variation in H for columbium and that could obscure the effects of substitutional solutes on H are considered in this section.

3.1.1 Activation Energy - Temperature Profile and the Effects of Interstitials

The concentration of interstitials in columbium creep specimens was varied by heat treating and testing specimens wrapped with tungsten foil and specimens exposed to the vacuum (10^{-6} Torr). The foil reduces impingement by gaseous interstitials during vacuum heat treatments and thus reduces contamination of the specimen with interstitials (Ref. 2, 4). Therefore, wrapped and unwrapped specimens given the same heat treatment will have the same structure but different interstitial concentrations. The variation in H with temperature for wrapped and unwrapped specimens of columbium is shown In Figure 1 for two lots of columbium. The number of determinations at each temperature for Lot 1 columbium is indicated by the data points. The specimens tested below 2300° F were first annealed for five minutes at 2300° F in the creep furnace to stabilize their structure. Specimens tested above this temperature were given no prior annealing treatment but were held at the test temperature for five minutes before beginning the test.

There are several important points concerning the apparent activation energy for creep, shown by the results for Lot 1 columbium. First, there is an anomalous maximum in H near 1900° F (0.48 T_M). Second, between about 1500 and 2200° F, H is noticeably less for the wrapped specimens of the same columbium than for the unwrapped specimens (compare solid line profiles in Fig. 1 for Lot 1 columbium). This difference is greatest near 1800 to 2000° F, the temperature region of the peak in H values for the specimens exposed to the vacuum. This behavior suggests that interstitial contamination of the specimens during the pre-creep annealing treatment at 2300° F or during subsequent testing increases the activation energy for creep. Although the way that interstitials change the value of H for columbium is not known, the effect is most pronounced near 1900° F. Finally, the values of H at the higher temperatures shown in Figure 1 are approaching the value of the activation energy for self-diffusion in columbium. At 2750° F (0.65 T_M) H is 90 k cal/mole, whereas the activation energy for self-diffusion in columbium is reported to be 96 k cal/mole (Ref. 6). For the rate of increase in H with temperature (shown in Fig. 1) near 2700° F, H would equal the activation energy for self-diffusion at about 0.7 T_M.

The results of some additional tests to investigate the peak in H values near 1900° F are shown in Figure 2 and Table II. In Figure 2 the activation energy at 1850° F is shown as a function of strain for wrapped and unwrapped specimens. It is evident that the wrapped specimen has a significantly lower value of H over the range of strain tested than the specimen exposed to the vacuum. The effects of wrapping the specimens and heat treating them on the activation energy for creep at 1850° F are listed in Table II (values derived from 1800 to 1900° F temperature shifts). Most of the speciment were given a pre-creep heat treatment at 2300° F. The intended purpose of this treatment was to stabilize the grain structure for creep testing at lower temperatures.

Specimens numbered 1 through 7 were obtained from columbium (Lot 1) containing 200 ppm oxygen and 95 ppm nitrogen and specimens 1A and 2A were obtained from columbium (Lot 2) containing 85 ppm oxygen and 110 ppm nitrogen in the as-received conditions. Both lots of columbium contained chromium and copper as the

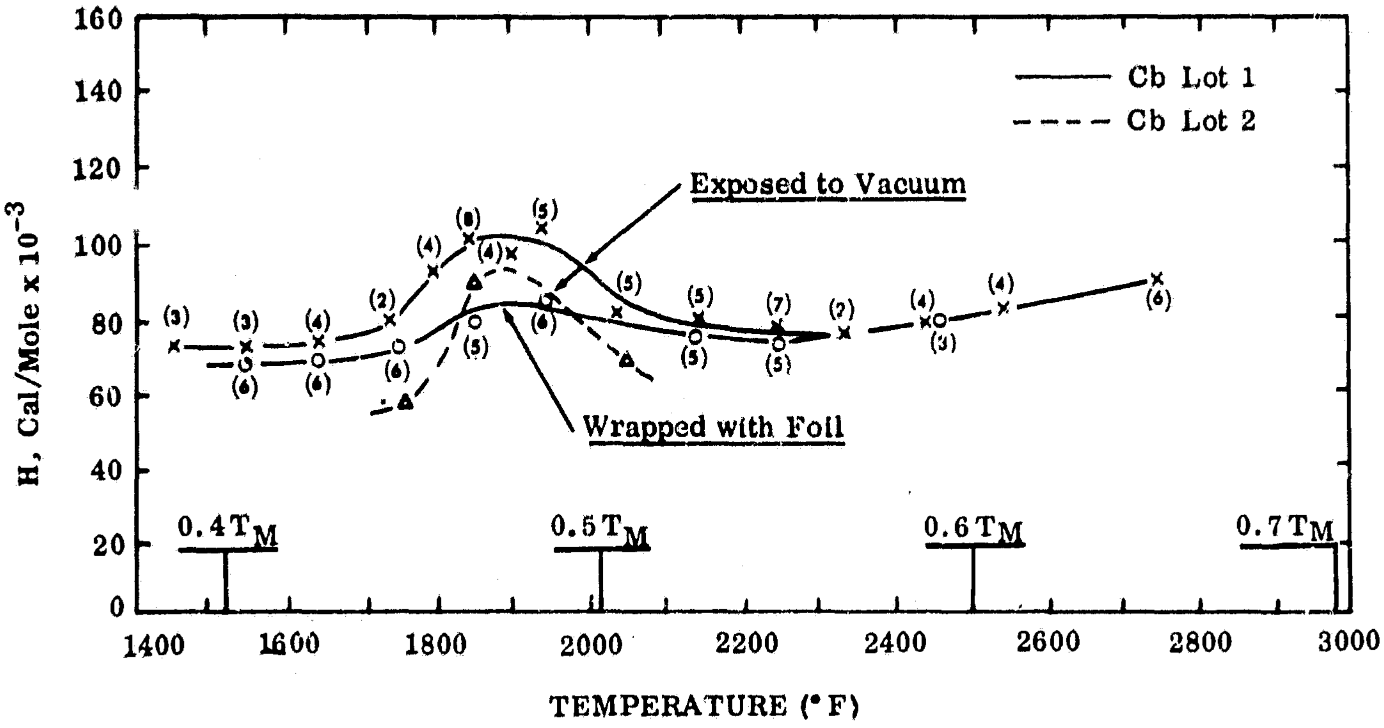


FIGURE 1. ACTIVATION ENERGY FOR CREEP OVER A RANGE OF TEMPERATURE FOR TWO TEST LOTS OF COLUMBIUM

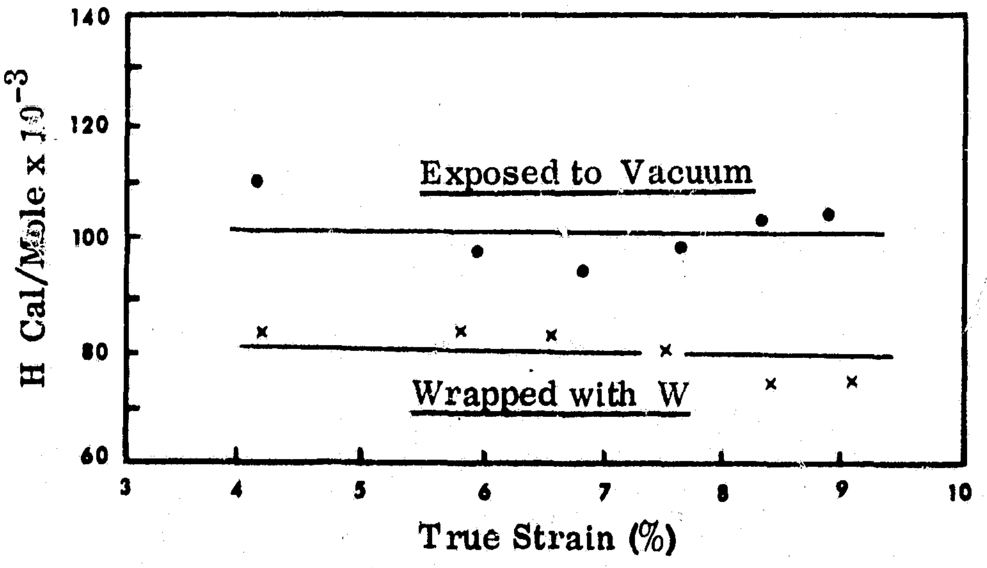


FIGURE 2. ACTIVATION ENERGY FOR CREEP OF COLUMBIUM VERSUS STRAIN AT 1850°F

TABLE II

VARIATION IN H FOR COLUMBIUM AT 1850°F WITH PRE-CREEP TREATMENT AND TEST CONDITIONS

Specimen Number	Pre-Creep Treatment	Test Conditions	H (cal/mole x 10^{-3})
1	2300°F/5 min. (not wrapped)	Not wrapped	102
2	2300°F/5 min. (wrapped)	Wrapped	79
3	2300°F/1 hour (not wrapped)	Not wrapped	150
4	2300°F/1 hour (wrapped)	Wrapped	115
5	As Received	Not wrapped	68
6	As Received	Wrapped	65
7	2300°F/5 min. (wrapped)	Not wrapped Vacuum of 10^{-5} Torr	100
1A	2300°F/5 min. (not wrapped)	Not wrapped	91
2A	2300°F/5 min. (wrapped)	Wrapped	75

major substitutional impurities (in concentrations of 0.1 percent). Past work has shown that the major interstitial contaminants introduced during vacuum heat treatments of columbium and columbium alloys are oxygen and nitrogen (Ref. 2, 4). Analyses of some of the wrapped and unwrapped specimens showed that the wrapped specimens were lower in these interstitials than the unwrapped specimens, but the analyses were not sufficiently accurate to yield quantitative differences in interstitial concentrations.

As shown in Table II, all of the wrapped specimens yield lower values of H than the unwrapped specimens for identical pre-creep treatments. This suggests that interstitial contamination increases the value of H at the peak in activation energy values near 1900°F. The specimens tested directly without a pre-creep heat treatment (5, 6) yield about the same values of H for both wrapped and unwrapped conditions indicating that the interstitial contamination affecting H takes place primarily during the 2300°F heat treatment rather than during creep testing at 1800 to 1900°F. However, the results suggest that sufficient interstitial contamination to increase H may occur at 1800 to 1900°F if the specimens are tested in a vacuum of 10^{-5} Torr (specimen 7).

The activation energies from Table II are shown as a function of the annealing time at 2300°F in Figure 3. Curves connecting the data for the wrapped and unwrapped specimens show the effect of interstitial contamination on H. The effect of testing in a vacuum of 10^{-5} Torr (specimen 7) rather than wrapped in the normal vacuum of 10^{-6} Torr (specimen 2) on the value of H is also indicated in this figure by the arrow.

The sensitivity of H to the interstitial concentration is also suggested from a comparison of the values of H derived from columbium with different initial interstitial concentrations. Specimen 1A and 2A (85 ppm oxygen, 110 ppm nitrogen) yield lower values of H than specimens 1 and 2 (200 ppm oxygen, 95 ppm nitrogen). Additional values of H for the columbium with the lower initial oxygen concentration (Lot 2 columbium) are shown by a dashed line in Figure 1. These data suggest that there is also a peak in the activation energy - temperature profile for this lot of columbium and that it occurs at about the same temperature (1800-2000°F). However, the peak for the columbium lower in initial oxygen (Lot 2) appears to be somewhat larger and to occur at a lower value of H than that for the columbium higher in oxygen (Lot 1, solid line) when both are exposed to the vacuum.

A comparison of the values of H for the wrapped and unwrapped specimens given the same heat treatment certainly suggests that the interstitial concentration affects the value of H near $0.5\ T_M$ for unalloyed columbium. However, evidence was obtained indicating that other factors may also affect H for columbium near this temperature. This is shown by the following test. The interstitial concentration in a specimen was increased to 3000 ppm oxygen and 135 ppm nitrogen. The value of H at 1850°F for this specimen was about the same as that for a specimen with only 135 ppm

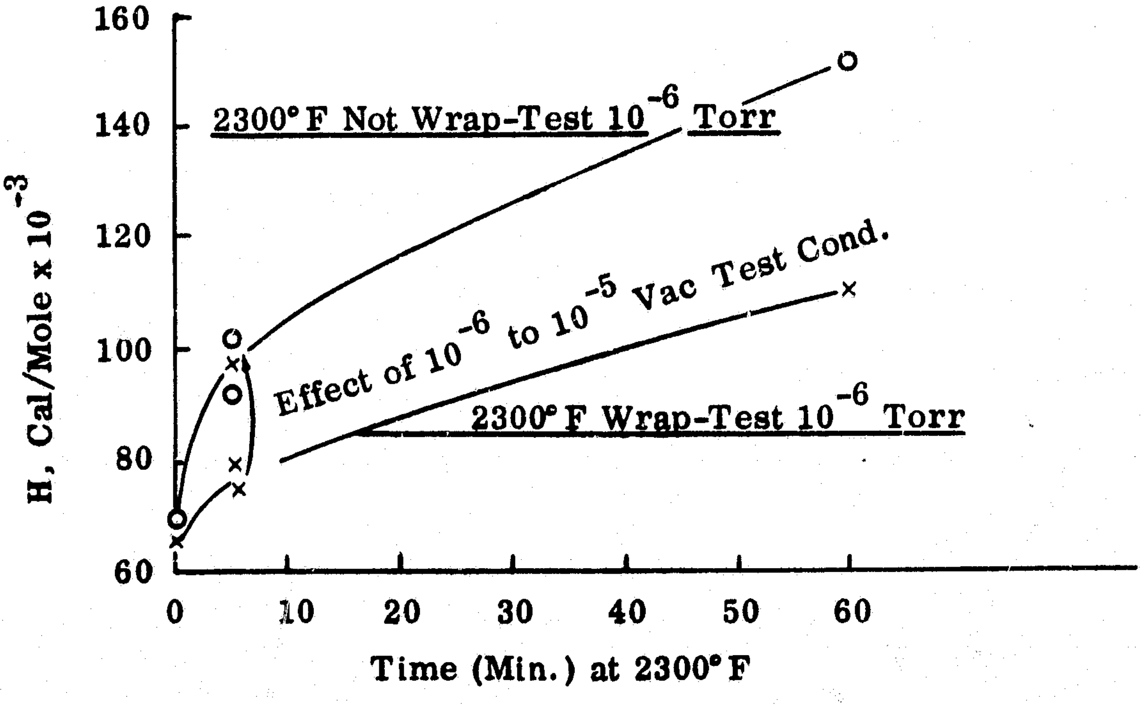
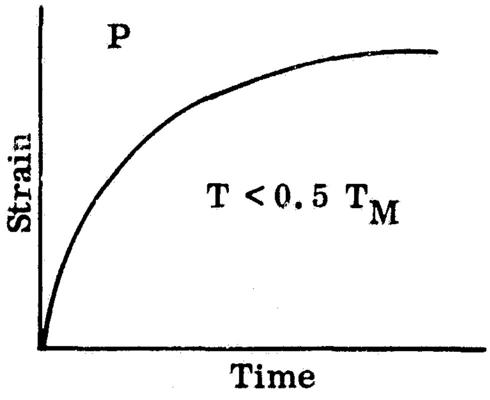


FIGURE 3. DATA OF TABLE I SHOWING EFFECT OF ANNEALING WRAPPED AND UNWRAPPED SPECIMENS AT 2300°F ON THE VALUE OF H AT 1850°F

oxygen and 95 ppm nitrogen (i.e., 100 k cal/mole). Evidently, there is a limit to the increase in H that can be induced simply by increasing the interstitial concentration. Therefore, other factors such as the ratio of interstials to dislocations or to impurity solutes must also affect H if values exceeding 100 k cal/mole are to be accounted for (e.g., specimens 3 and 4, Table I). In the following paragraphs the results of a series of tests performed to sort out the effect of structure on H are presented.

3.1.2 Structure

One way to determine the effect of structure on H is to determine the activation energy as a function of strain during primary creep at a temperature where interstitial contamination is not a problem. During primary creep, the creep rate decreases because the structure becomes progressively more worked. Therefore, if H varies with strain during primary creep, it also varies with structure. The duration of primary creep above about 0.5 T_M is ordinarily too short to determine the activation energy as a function of strain using the temperature shift method. However, below 0.5 T_M primary creep (P) becomes more extensive until it can comprise the entire creep curve, and in this event the creep rate will continue to decrease until creep can no longer be detected (see adjacent sketch).



H is shown as a function of strain in Figure 4 for specimens tested at three different temperatures after they were first annealed at 2300° F. Activation energies were determined for the specimen tested at 1850 and 1550° F during approximately steady-state creep conditions (i.e., constant structure), whereas primary creep continued throughout most of the test at 1275° F, implying a continuous variation of structure. The test at 1275° F shows that H is a function of structure since the structure is the only variable during this test. H is initially very high at low strain when the annealed specimen is tested at 1275° F, and H decreases as the specimen becomes more worked with increasing creep strain. Apparently the value of H decreases until the structure attains an equilibrium configuration for the test conditions at about 14 percent strain. The data at 1550° F also show decreasing values of H with increasing strain whereas the data at 1850° F do not show this trend.

Another example of the effect of structure on H is shown in Figure 5. The specimen when first tested at 1500 to 1600° F yielded values of H of 70 to 80 k cal/mole. On retesting at 1250 to 1300° F, H increased to values well over 200 k cal/mole before decreasing to lower values as the structure became more worked with increasing

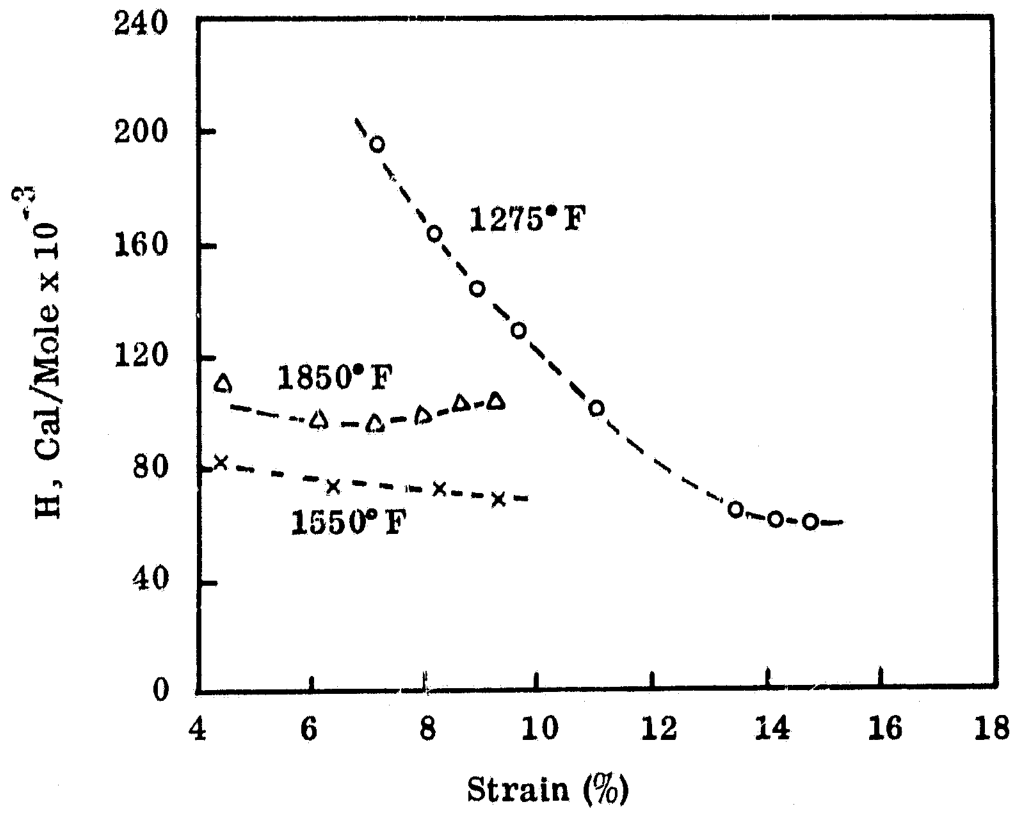


FIGURE 4. ACTIVATION ENERGY FOR CREEP VERSUS STRAIN AT 1850, 1550 AND 1275°F; Pre-creep Treatment 2300°F For Five Minutes

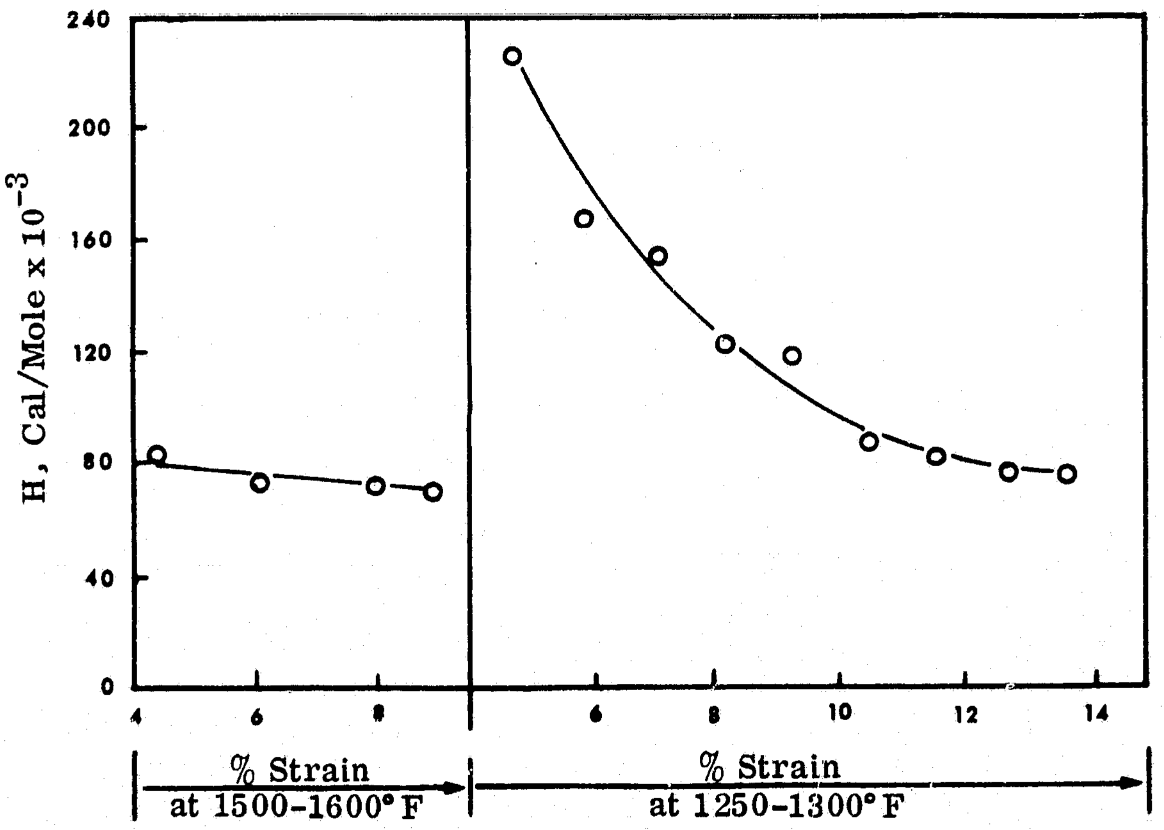


FIGURE 5. ACTIVATION ENERGY FOR CREEP VERSUS STRAIN FOR A SPECIMEN TESTED FIRST AT 1500 TO 1600°F AND AGAIN AT 1250 TO 1300°F

deformation at the lower temperatures. The results of both of these tests suggest that H approaches a value typical of the test temperature when the structure reaches an equilibrium configuration.

Previous results show that interstitial contamination during creep testing is not sufficient to affect H at 1850° F in a vacuum of 10^{-6} Torr. Therefore, interstitial contamination during creep testing is not likely to be a factor affecting the activation energy values derived from these tests at lower temperatures.

As shown in Figure 6, the activation energy for creep at 2150° F is also a sensitive function of the thermal and mechanical history of the columbium specimens. The activation energy for creep for specimen A was measured at 2150° F after it was first annealed for five minutes at 2300° F. Specimen B was crept to ten percent strain in the temperature region 2500 to 2600° F and then H was measured at 2150° F. These different pre-creep treatments have drastically altered the value of H at 2150° F. The activation energy for creep for specimen B is about twice that for specimen A at the same strain. However, the values of H for both specimens could approach the same value if creep could be continued to sufficiently high strains. The structure of specimen B may require extensive creep at 2150° F before it attains an equilibrium structure for this test condition.

3.1.3 Stress

In a number of metals it has been found that H becomes stress dependent below the temperature range where it is equal to the activation energy for self-diffusion. These results show that H decreases with decreasing temperature when activation energy measurements are made using higher stresses at lower temperatures (Ref. 7-9). For these tests (Fig. 1) the stress was increased from 1 ksi to 7 ksi between 2750° F (0.65 T_M) and 1500° F (0.4 T_M) so that the tests could be completed in a reasonable length of time. With the exception of the anomalous peak near 1900° F, H gradually decreases with decreasing temperature from 90 k cal/mole at 1750° F to about 70 k cal/mole at 1500° F. This decrease can be accounted for if H is somewhat stress dependent in the temperature region investigated. However, these results, as well as measurements of H made at different stresses during a single test (Fig. 7), suggest that the stress dependence of H, if any, is small.

3.1.4 Summary, Activation Energy for Creep of Columbium

The activation energy for creep, H, was determined for columbium specimens whose gage lengths were sheathed with foil to minimize interstitial contamination during testing. H was also measured for unsheathed columbium specimens. The results of these tests show that the activation energy for creep of columbium near 0.5 T_M is a function of its interstitial concentration (probably oxygen or nitrogen). The interstitials

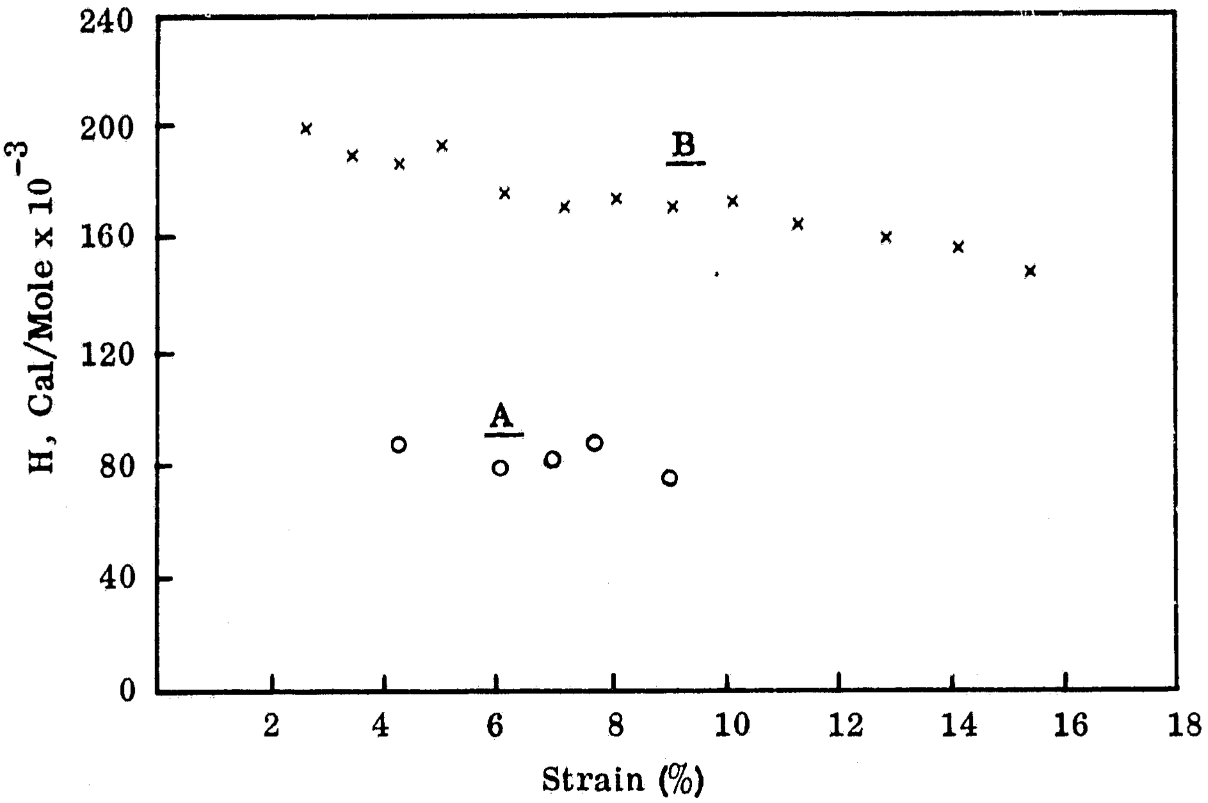


FIGURE 6. ACTIVATION ENERGY AS A FUNCTION OF STRAIN AT 2150°F FOR SPECIMENS GIVEN DIFFERENT PRE-CREEP TREATMENTS; Specimen A, 2300°F For Five Minutes; Specimen B, Crept 2500-2600°F To Ten Percent Strain

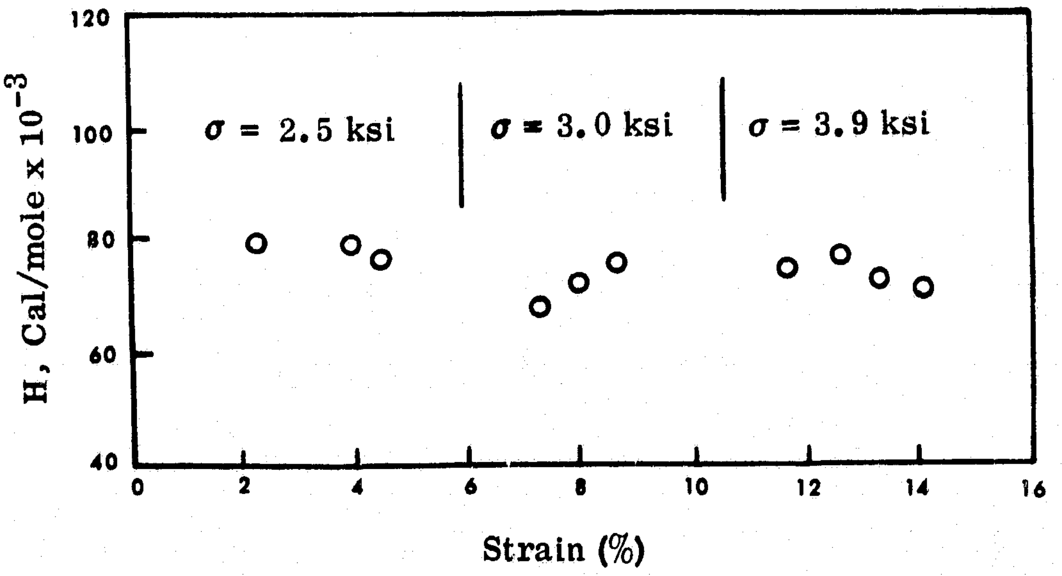


FIGURE 7. ACTIVATION ENERGY FOR CREEP OF COLUMBIUM AT 1850°F AT DIFFERENT STRESSES

in columbium appear to contribute to an activation energy peak between 1800 and 2000° F when H is determined as a function of temperature. One mechanism thought to account for activation energy peaks in other metals is an interaction between solutes (or solutes in combination) and moving dislocations through a dynamic strain aging effect. The mobility of interstitials at the temperature range of the peak is probably too great to account for high values of H through an interstitials-dislocation interaction. However, the mobility of interstitials combined with other impurity solutes in columbium may be of the correct order to account for this peak.

H for columbium near 0.5 T_M is also a function of the structure of columbium (e.g., grain or subgrain size, dislocation density or configuration) near 0.5 T_M. This was shown by the dependence of H on pre-creep structure and structural changes that occur during primary creep. However, the results indicate that H approaches a value typical of the test temperature when an equilibrium structure is attained.

Over the range of temperature tested, the results indicate that if there is an effect of stress on H, it is relatively minor. In this regard, it is found that H for most metals is not noticeably stress dependent above about 0.5 T_M.

A comparison of the present activation energy determinations for columbium, together with those from previous work (assembled by Begley, et al, Ref. 10) are shown in Figure 8. The effects of interstitial on H (i.e., the results for unsheathed columbium) determined in this program are shown by the pronounced peak between 0.4 and 0.5 T_M. It is evident that there is a very large amount of scatter in the published activation energy values for columbium, especially in the region between 0.4 and 0.5 T_M. The results of the present study suggest that some of the scatter in activation energy determinations in past work can be accounted for by the effects of structure and interstitial concentration on H.

3.2 CREEP OF D43

The apparent activation energy for creep, H, was derived from creep curves similar to that shown in Figure 9. Steady-state creep was rapidly attained at the beginning of the test and with each change in temperature. This behavior suggests that the alloy rapidly attains a stable structure with each shift in temperature or that the structure is constant throughout the test. Since the dispersion will tend to stabilize the structure, the latter is probably the case. The activation energy was found to be independent of the direction of the temperature shift and the magnitude of the shift (50 or 100° F). Although H was approximately independent of the strain, there was a tendency for it to be somewhat greater and more varied at lower strains than at higher strains. The activation energy is shown as a function of strain for one of these tests in Figure 10. The average value of H for the first nine percent strain is 119,500 cal/ more and the average value for the last nine percent is 118,000 cal/mole.

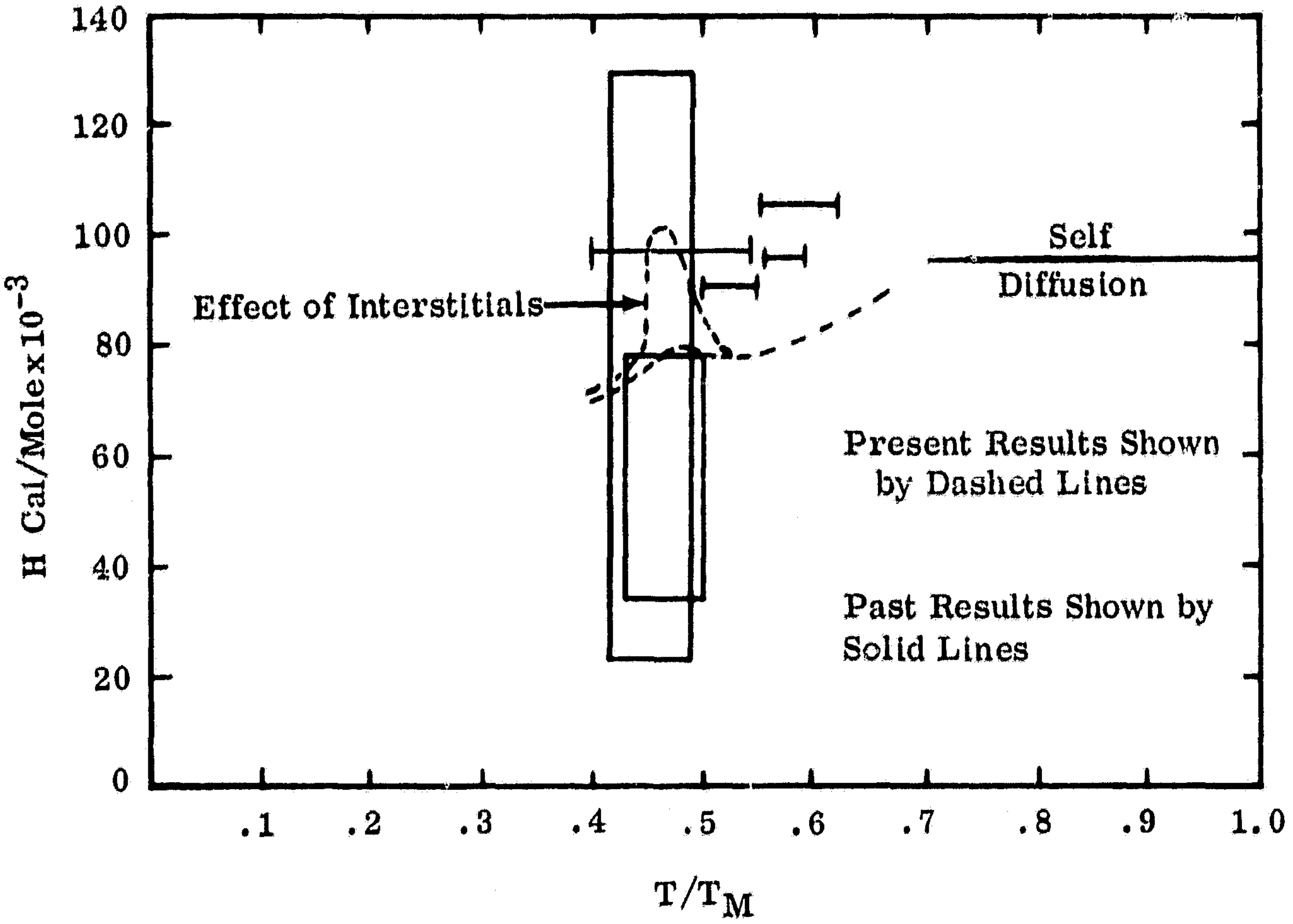


FIGURE 8. COMPARISON OF PRESENT AND PAST DETERMINATIONS OF THE ACTIVATION ENERGY FOR CREEP OF COLUMBIUM; Past Results Shown by Solid Lines are Data Assembled by Begley, Harrod and Gold (Ref. 10)

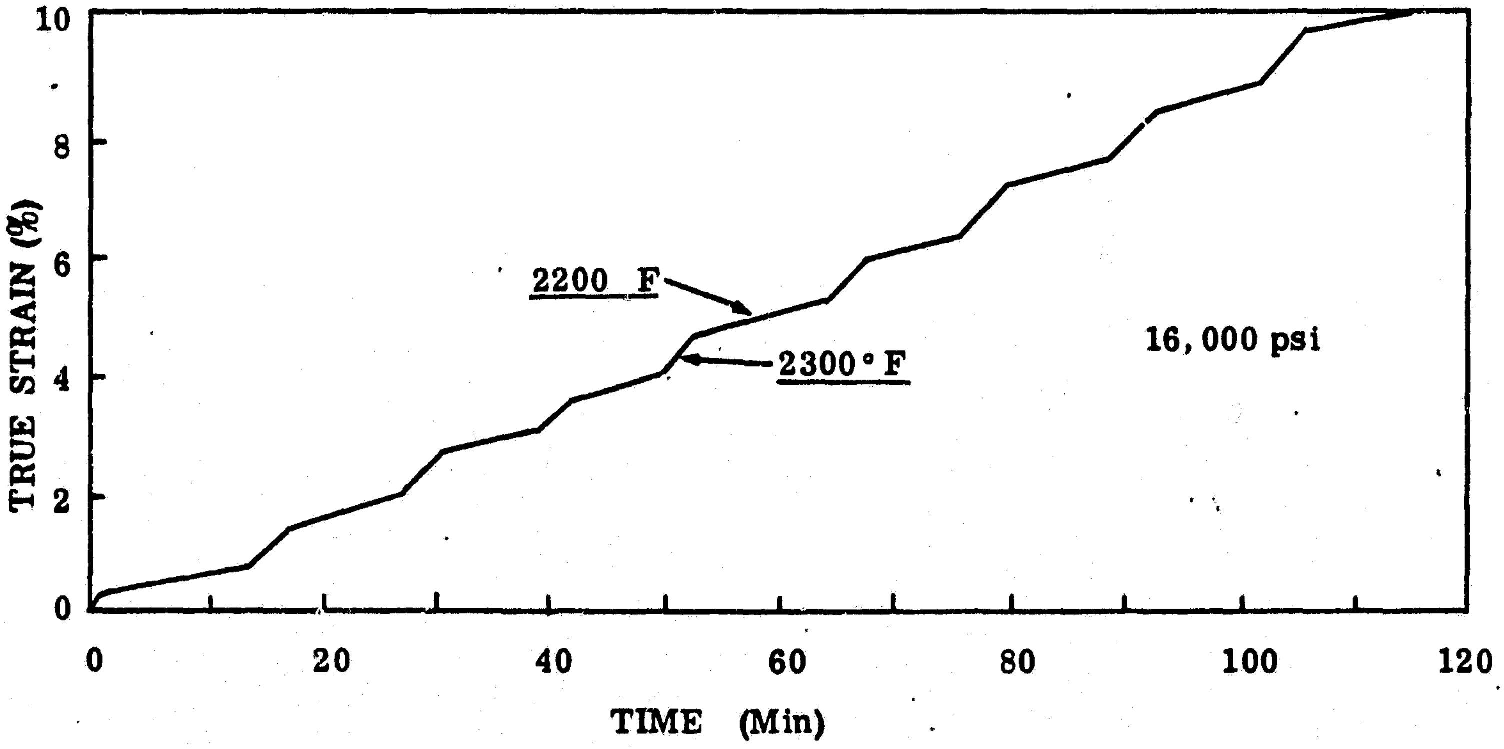


FIGURE 9. CREEP CURVE FOR ALTERNATING 2200 - 2300°F TEMPERATURE SHIFTS

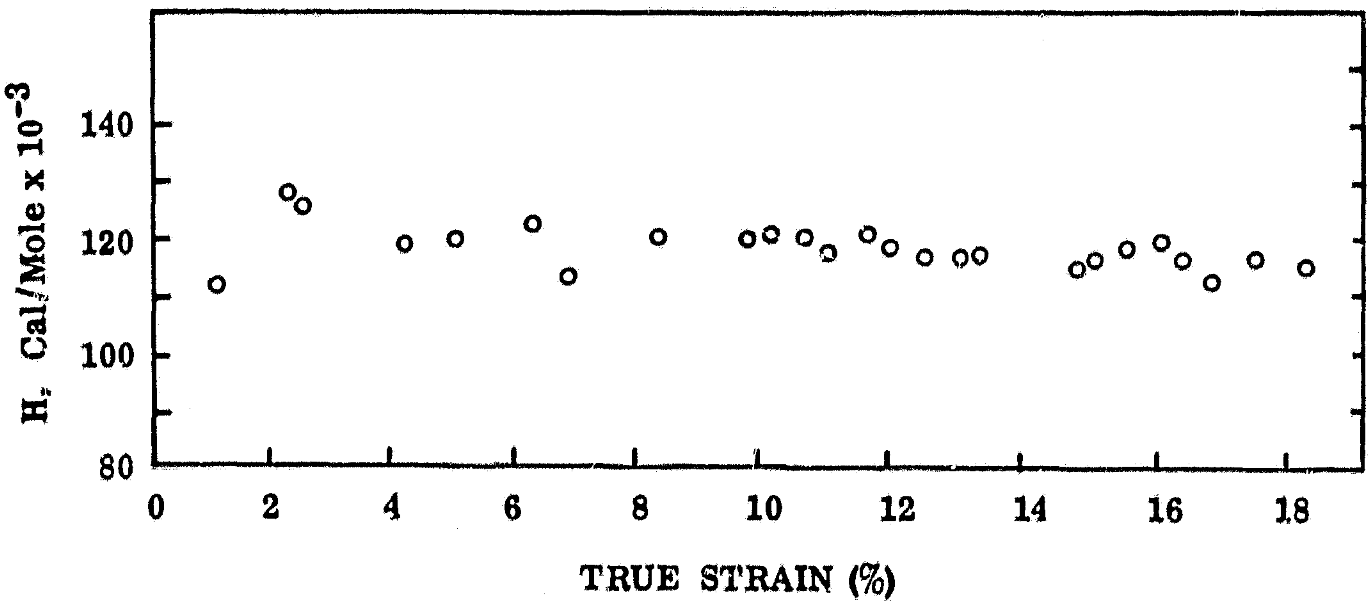


FIGURE 10. ACTIVATION ENERGY FOR CREEP OF D43 VERSUS STRAIN AT 2000°F

3.2.1 Activation Energy - Temperature Profile

The variation in H for D43 over the temperature range 1650 to 3200°F is shown in Figure 11. Multiple values of H at one temperature are plotted to show the average value, the range of values, and the number of determinations. Multiple determinations were made at the lower temperatures where initial results indicated that H might increase with a decrease in temperature. As shown by the solid line in Figure 11, H is approximately equal to 110,000 cal/mole above 2400°F (0.55 T_M). Below this temperature the results suggest that H exceeds 110,000 cal/mole, as indicated by the dashed line.

At temperatures greater than about one-half the absolute temperature of melting the activation energy for creep in metals is independent of stress and is approximately equal to the activation energy for self-diffusion. This behavior is taken as evidence that diffusion-controlled dislocation movement is the rate controlling mechanism for high-temperature creep. Since the activation energies for self-diffusion of the constituent elements in D43 are not known, that for the host element columbium, will be used as a qualitative basis for comparison with the values of H derived in this study.

The activation energy for self-diffusion in columbium is reported to be 96,000 cal/mole (Ref. 6). Since the melting temperature of D43 exceeds that for columbium by about 230°F, activation energies greater than 96,000 cal/mole would be expected for self-diffusion of columbium and the dominant solute element, tungsten. Another factor to be considered in comparing creep and self-diffusion activation energies above about 0.5 T_M is the effect on H of a variation in the elastic modulus with temperature (Ref. 11). In the present case this correction cannot be quantitatively

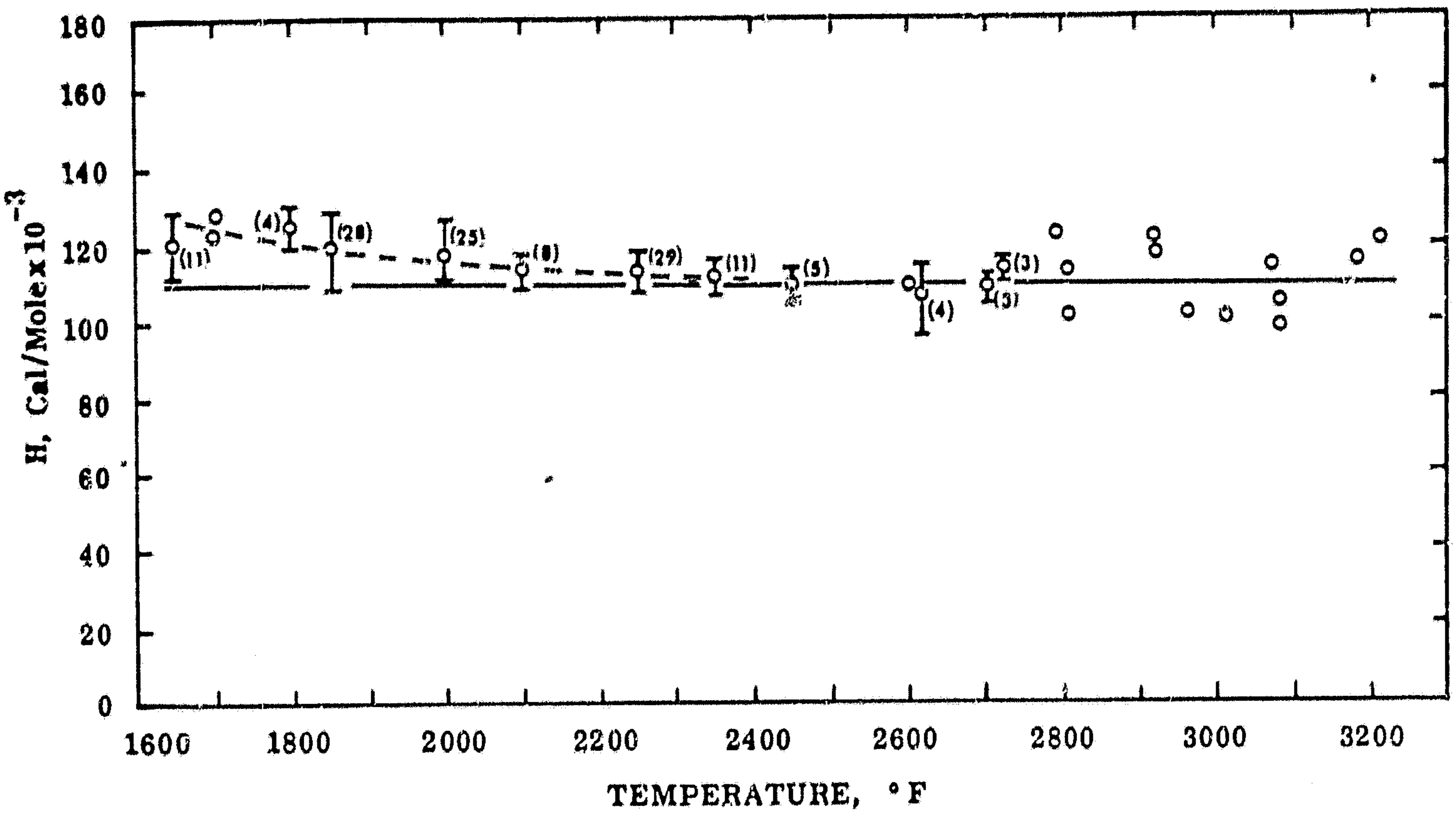


FIGURE 11. ACTIVATION ENERGY FOR CREEP OF D43 FROM 1650°F TO 3200°F

applied because the creep equation and the temperature dependence of the elastic modulus are not defined for D43. According to this concept, however, the corrected value of H would be a few percent less than the 110,000 cal/mole, derived experimentally. Therefore, the value of H above 2400°F of 110,000 cal/mole is about that expected for a creep rate controlled by self-diffusion in D43.

Below ~2400°F, H increases to values likely to be above the activation energy for self-diffusion in this alloy. As previously pointed out, H decreases below about 0.5 T_M for a number of metals when activation energy measurements are made using increasing values of stress with decreasing temperature (to preserve similar creep rates). This is in contrast with the results shown in Figure 11 since H increases slightly with decreasing temperature despite an increase in stress of from 15,000 psi at 2400°F to 44,000 psi at 1650°F. Therefore, effect of stress on H is not likely to account for values of H exceeding 110,000 cal/mole for D43. In view of the sensitivity of H for columbium to interstitials in this temperature range (Fig. 1), it was thought that interstitials in D43 might account for high values of H at the lower temperatures.

3.2.2 Interstitials

The effect on creep of wrapping the specimens to reduce possible interstitial contamination during testing was carefully investigated. Specimens were tested with their gage lengths wrapped with foil and the same specimens were tested again at the same temperature and stress with their gage lengths exposed to the vacuum. In addition, different wrapped and unwrapped specimens were tested for comparison under the same condition of stress and temperature. The activation energies derived from some of these tests are shown in Figure 12. The results show that the activation energy is not significantly affected by protecting the gage length of the specimen with foil during testing. These results, together with the interstitial analyses cited previously (Table I) suggest that interstitial contamination is probably not a significant factor affecting the activation energy values for this alloy.

However, some evidence was obtained concerning the effect of interstitials on H by testing D43 specimens with reduced interstitial concentrations in the temperature region where H is greater than 110,000 cal/mole. In Figure 13, H is shown as a function of strain at 1850° F for specimens whose combined carbon and oxygen concentrations were less than 100 ppm before testing. The interstitial concentrations were reduced by heat treating the specimens (2200° F/60 hours) in contact with titanium (defined in earlier work as the interstitial sink effect). The interstitials concentrated in the titanium which was removed from the specimen before creep testing. After this heat treatment, the concentration of the elements in the specimens was carefully checked using the electron microprobe to ensure that the zone of Ti/D43 interdiffusion has also been removed.

There are several significant points revealed by these tests. First, the wrapped specimen yields lower values of H than the specimen exposed to the vacuum. Evidently, restoration of interstitials during testing at 1850° F increases the activation energy for creep for specimens initially low in interstitials. Second, these tests show a dependence of H on strain (or structure) at 1850° F, whereas H is independent of strain for specimens with a higher interstitial concentration (Fig. 12). In addition, the specimens low in interstitials attain a lower value of H at 1850° F than those high in interstitials (compare Fig. 12 and Fig. 13). Finally, additional tests showed that the effect on H of wrapping the specimen and the dependence of H on strain shown in Figure 13 are no longer present when the tests are conducted at 2200° F.

In summation, interstitial contamination during testing of the as-received D43 does not seem to affect H. However, the tests on D43 with reduced interstitial concentration suggests that interstitials already present in the as-received alloy could contribute to the rise in H to values above 110,000 cal/mole. The specific way in which interstitials affect H is not known. However, in general, interstitials may influence H directly through dynamic strain aging, or indirectly through the effect of the dispersed interstitial phase on structure.

If strain aging is contributing to the increase of H in D43 at lower temperatures, H may include the temperature sensitivity of the equilibrium concentration of solutes near dislocations, as well as the regular temperature sensitivity of creep. As pointed out in the previous section on columium, consideration of atomic mobilities in columbium and columbium alloys suggests that interstitials or solute atoms alone are not likely to give rise to interactions with dislocations in D43 during creep at 1650 to

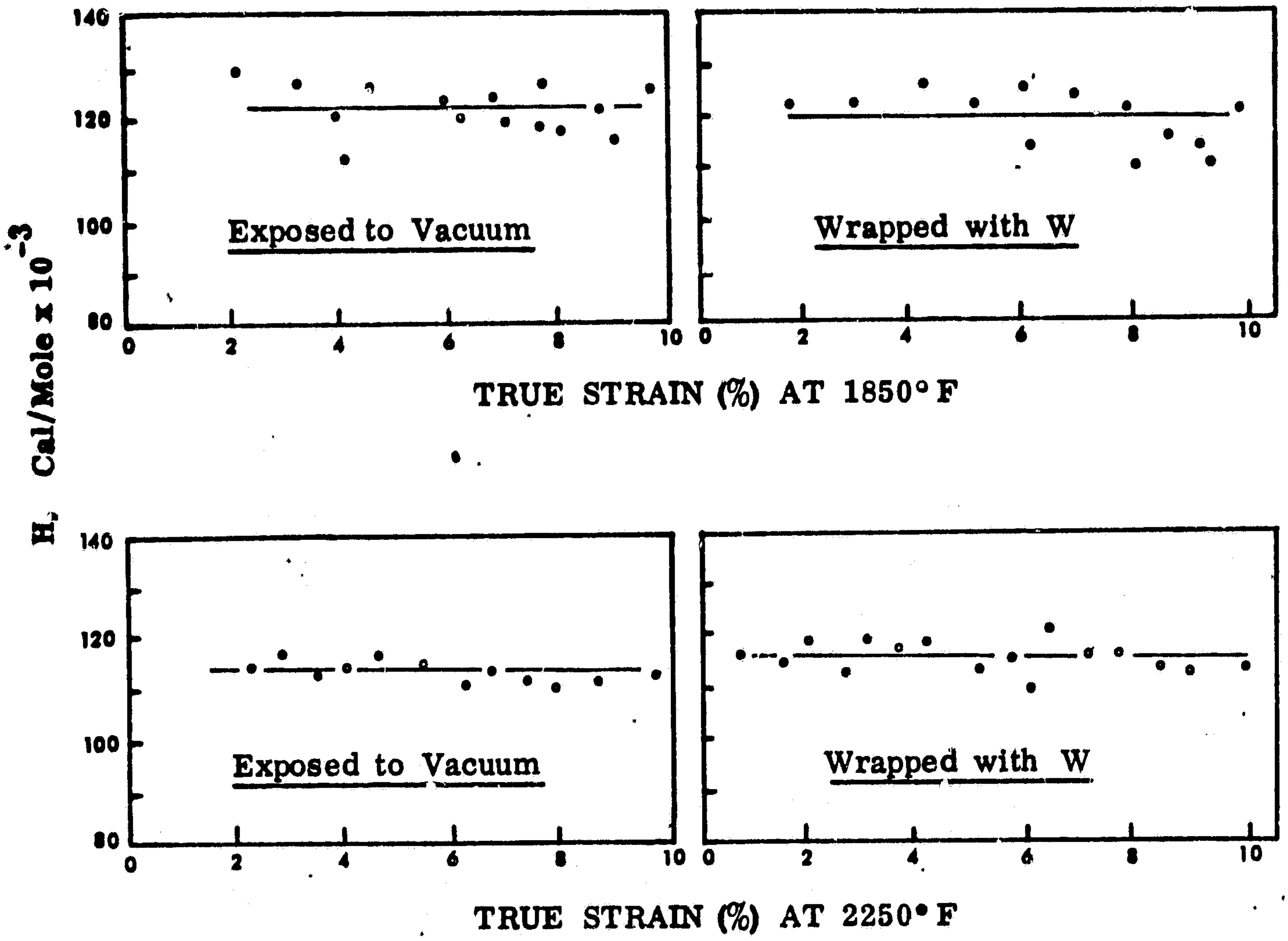


FIGURE 12. ACTIVATION ENERGY FOR CREEP AT 1850 AND 2250°F FOR SPECIMENS TESTED IN VACUUM OF 10^{-6} TORR WRAPPED WITH TUNGSTEN FOIL AND EXPOSED TO THE VACUUM

2400°F (Ref. 12, 13). However, where solutes interact with each other, the temperature range of the dislocation-solute interactions may be shifted or broadened and thus could give rise to high values of H in this temperature range.

3.2.3 Stress

The duration of the tests from which the activation energies were derived was controlled by varying the stress; at low temperatures a high stress was used, and at high temperatures a low stress was used. Between 2400 and 3300°F the stress was reduced from 15,000 to 3000 psi. The activation energy is approximately constant over this range of temperature (Fig. 11) so that it is necessary to determine if this constancy arises from opposing effects of stress and temperature. The dependence of the creep rate on stress was determined by measuring the change in strain rate before and after sudden reductions in stress during constant temperature creep tests. The results of a typical differential stress test are shown in Figure 14, and the stresses together with the strain rates derived from this test are listed in Table III. In general,

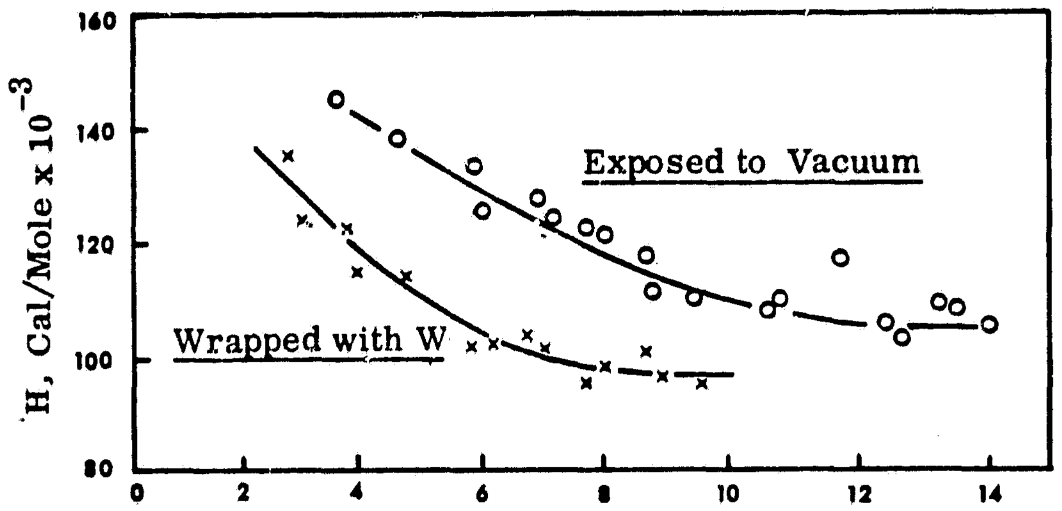


FIGURE 13. ACTIVATION ENERGY FOR CREEP AT 1850° F FOR SPECIMENS WITH LOW INTERSTITIAL CONCENTRATION; Specimens Tested in a Vacuum of 10^{-6} Torr Wrapped With Tungsten Foil and Exposed to the Vacuum

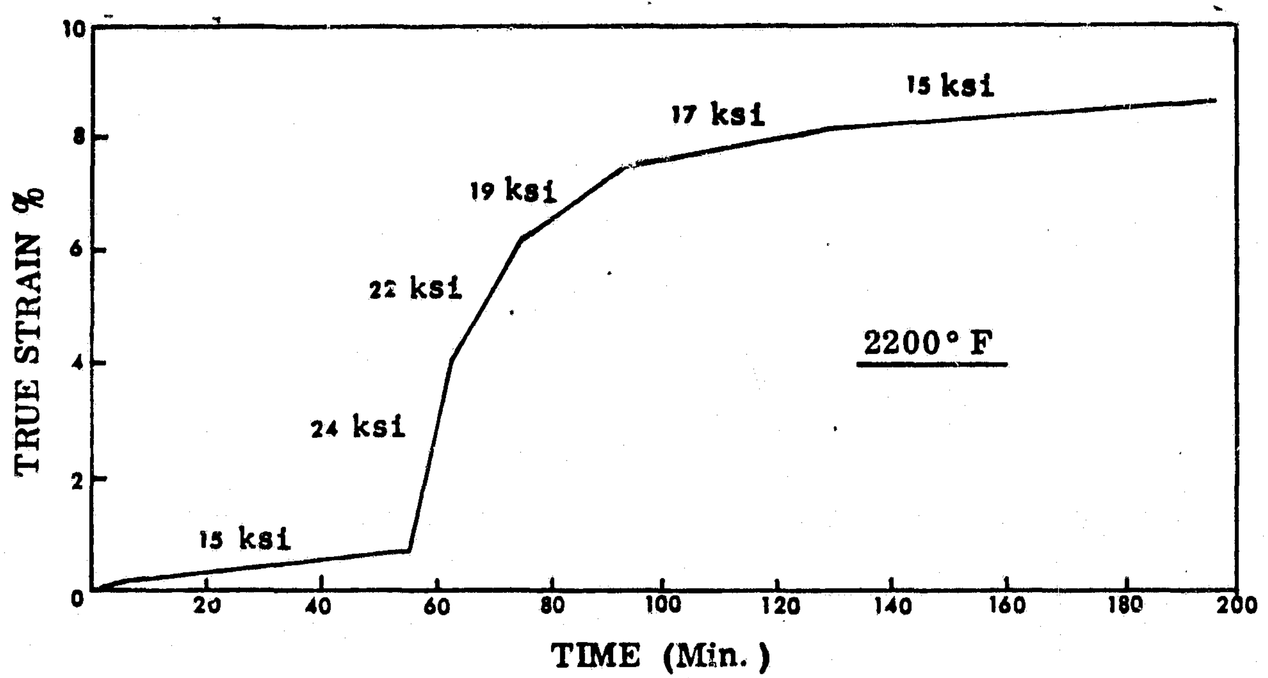


FIGURE 14. TYPICAL SEQUENCE FOR DIFFERENTIAL STRESS TESTS

TABLE III

CHANGE IN CREEP RATE OF D43 WITH STRESS FOR DIFFERENTIAL STRESS TESTS AT 2200°F

σ (ksi)	15	24	22	19	17	15
ϵ (percent)	0 - 0.7	0.7 - 4.0	4.0 - 6.5	6.5 - 7.7	7.7 - 8.5	8.5 - 9.0
$\epsilon^{\circ}/\text{min} \times 10^3$	0.15	6.9	3.4	0.93	0.37	0.14

it was found that the stress dependence of the creep rate for this alloy is independent of its stress and strain history. For example, the strain rate for 15 ksi in Table III is reproducible within experimental error at the beginning and end of the creep test. The differential stress curves, as well as the differential temperature curves, show a sharp transition between steady-state creep regions.

The dependence of the strain rate, ϵ°, on the stress, σ, at 2800, 2200 and 1750°F is shown in Figure 15 for the exponential law $\epsilon^{\circ} \propto e^{B\sigma}$ and in Figure 16 for the power law, $\epsilon^{\circ} \propto \sigma^n$. The exponential relationship (Fig. 15) is approximately linear for all of these temperatures, whereas the power relationship (Fig. 16) is nonlinear at 2800°F. In addition, B is approximately constant at the three temperatures while n varies from 3.5 to 30. Therefore, the proportionality of $\epsilon^{\circ} \propto e^{B\sigma}$ is probably a reasonable one to use in correlating stress variations over the range of temperature tested. In this regard, if B is independent of the stress, n must vary with stress as shown in Figure 16. The slopes of the curves in Figure 16 are $d \ln \epsilon^{\circ}/d \ln \sigma$ or n and for the data conforming to the equation $\epsilon^{\circ} = Ae^{B\sigma}$ where A and B are constants $d \ln \epsilon^{\circ}/d \ln \sigma = \sigma B$. Therefore,

$$n = \sigma B \qquad (1)$$

Thus, where B is $\sim 0.8 \times 10^{-3}$ psi^{-1} over a range of stress variations of 4 to 40 ksi, n will have values varying from 3 to 30 in agreement with the values of n shown in Figure 16.

In many metals, an exponential stress law will fit the creep data at low temperatures and high stresses while at high temperatures and low stresses a power stress law is better. However, for D43 a simple exponential stress law adequately describes the stress dependence of the creep rate over the range of temperature and stress tested. In the following discussion the power stress law will be referred to because n fo D43 can then be compared with published values of n for other metals. In addition, as shown in Figure 16, the σ^n relation will describe the stress dependence of D43 over a narrow range of stress variations.

When the stress dependence of the creep rate for pure metals is expressed by the power law the stress exponent, n, is found to be about 5 (Ref. 14, 15). Data have

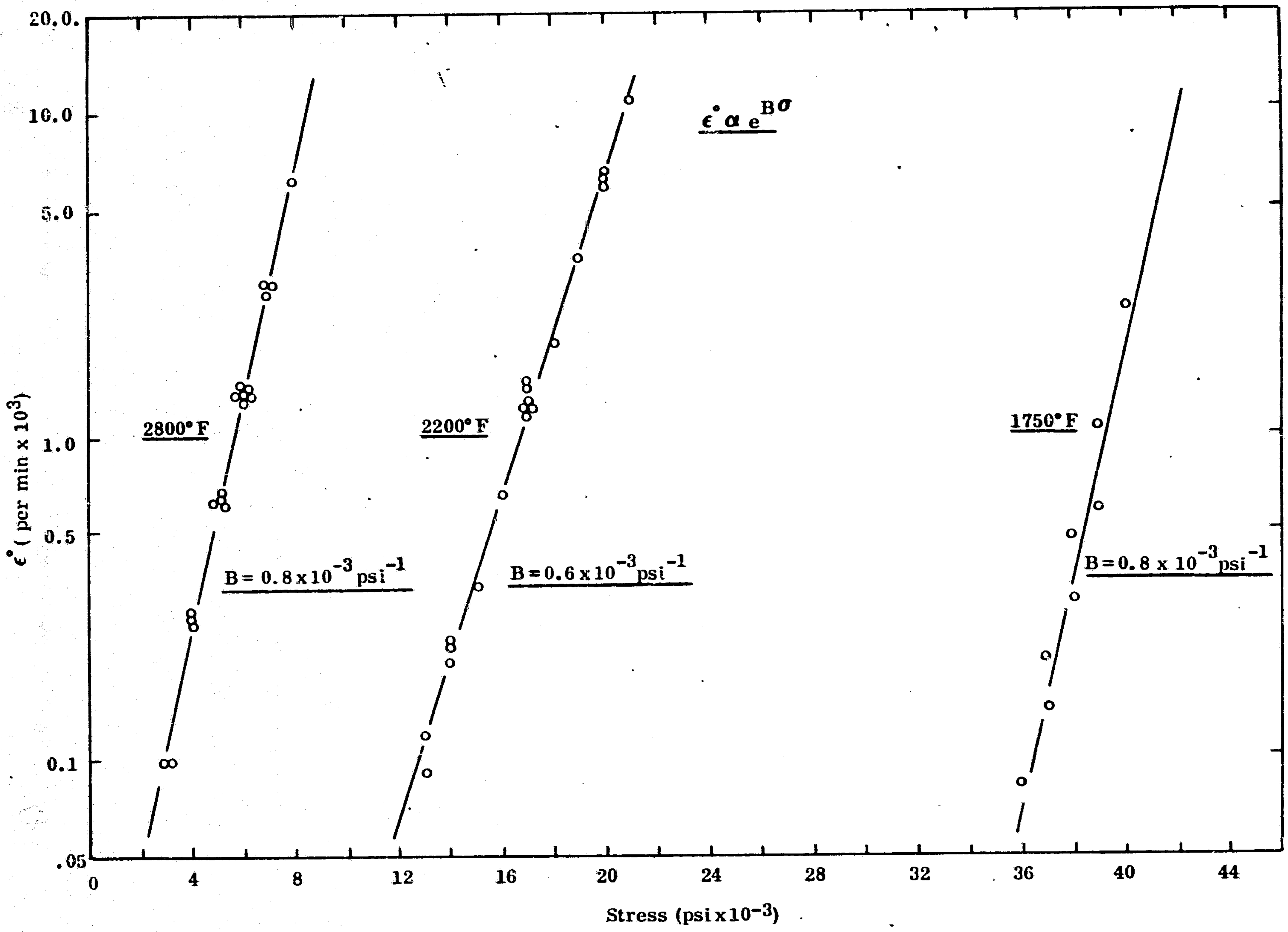


FIGURE 15. STRESS DEPENDENCE OF CREEP RATE FOR $\dot{\epsilon} \propto e^{B\sigma}$

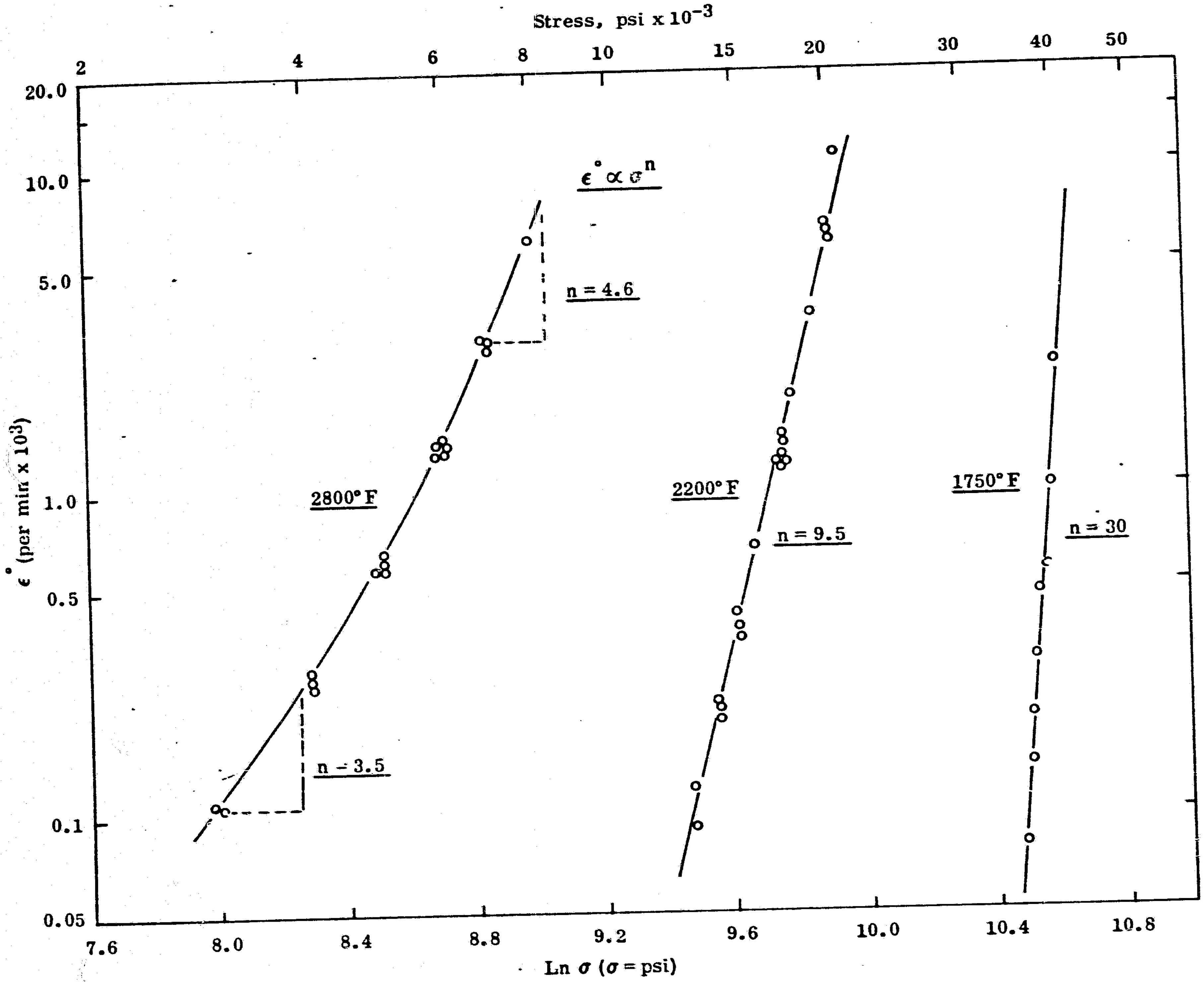


FIGURE 16. STRESS DEPENDENCE OF CREEP RATE FOR $\epsilon^\circ \propto \sigma^n$

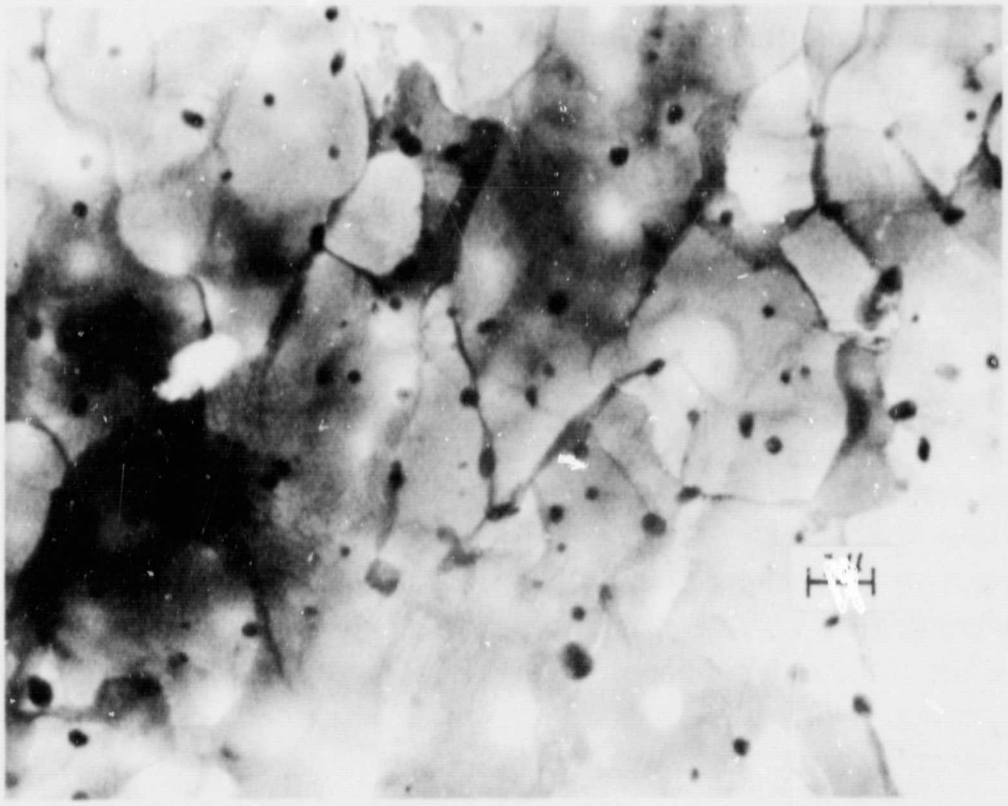
been reported showing a tendency for solute additions to decrease n (Ref. 16) and for a dispersed phase to increase n (Refs. 16 thru 20). Although the data available are meager, most of the dispersion strengthened alloys investigated show stress exponents in the range of 6 to 8. However, higher values of n (as high as 40) have been reported for several dispersion strengthened metals after work hardening (Ref. 18, 19). The stress exponent for D43, therefore, spans the entire range of values found in other systems. At low temperatures where a more worked dispersion strengthened structure would be anticipated, the value of n is as high as 30. At high temperatures where the dispersion would tend to be ineffective because of solutioning or agglomerating effects or because of the loss of interstitials, the value of n is in the range 3 to 5. As previously pointed out (Ref. 19), high values of n cannot be rationalized by present theories of creep of dispersion strengthened metals (Ref. 16, 21). However, the results of this study are in general agreement with other work showing a tendency toward higher values of n for more worked structures. This has been expressed quantitatively by Barrett (Ref. 22) by relating the stress dependence of the mobile dislocation density to the stress dependence of the creep rate. High values of n in dispersion strengthened alloys are attributed to a greater internal resistance to dislocation movement.

3.2.4 Structure

The structure in the creep specimens was studied before and after creep testing using transmission electron microscopy. The structures in different regions of the alloy before creep testing are shown in Figure 17 and those after steady-state creep to about seven percent strain at different temperatures are shown in Figure 18.

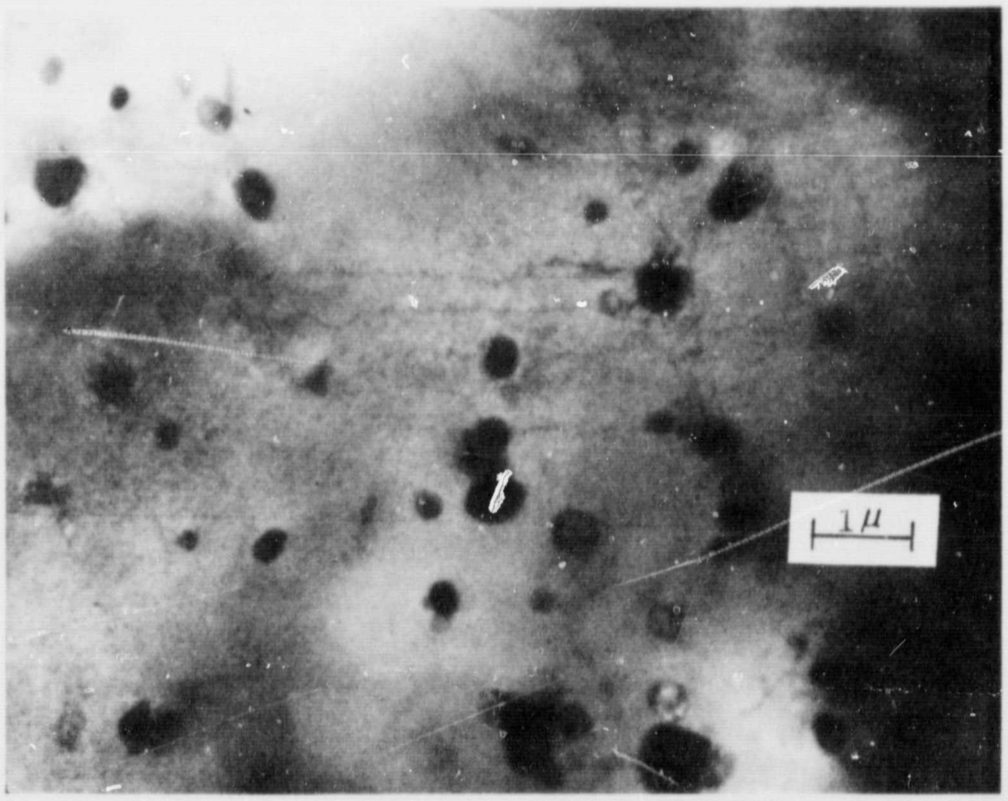
It is difficult to assign a typical structure to the D43 before creep testing because of the variations in structural features observed in different regions of the specimen. However, some of the structures frequently observed in the alloy before before creep testing are shown in Figure 17. Some regions of the alloy contained fine subgrains (Fig. 17A) while other regions were almost devoid of subgrains (Fig. 17B). A fine dispersed phase identified as (Zr, Cb)C (Ref. 23) was observed in all of the regions examined. The particles were often coincident with subgrain boundaries or dislocations. However, the dispersion was also observed in regions that were free of both boundaries and dislocations.

After creep at 1650°F (Fig. 18A) the dislocation density was much greater than that in the alloy prior to creep. In some regions dislocation networks stabilized by particles were arranged in cell structures with their centers almost devoid of dislocations. These configurations appear to be barriers to moving dislocations. In addition, dislocation tangles were observed both away from particles and centered on particles. These observations suggest that dislocations interact with each other to form networks and tangles, as well as with second-phase particles. Thus, the dispersed phase is only one of several structural features strengthening D43 during creep at 1650°F.



A

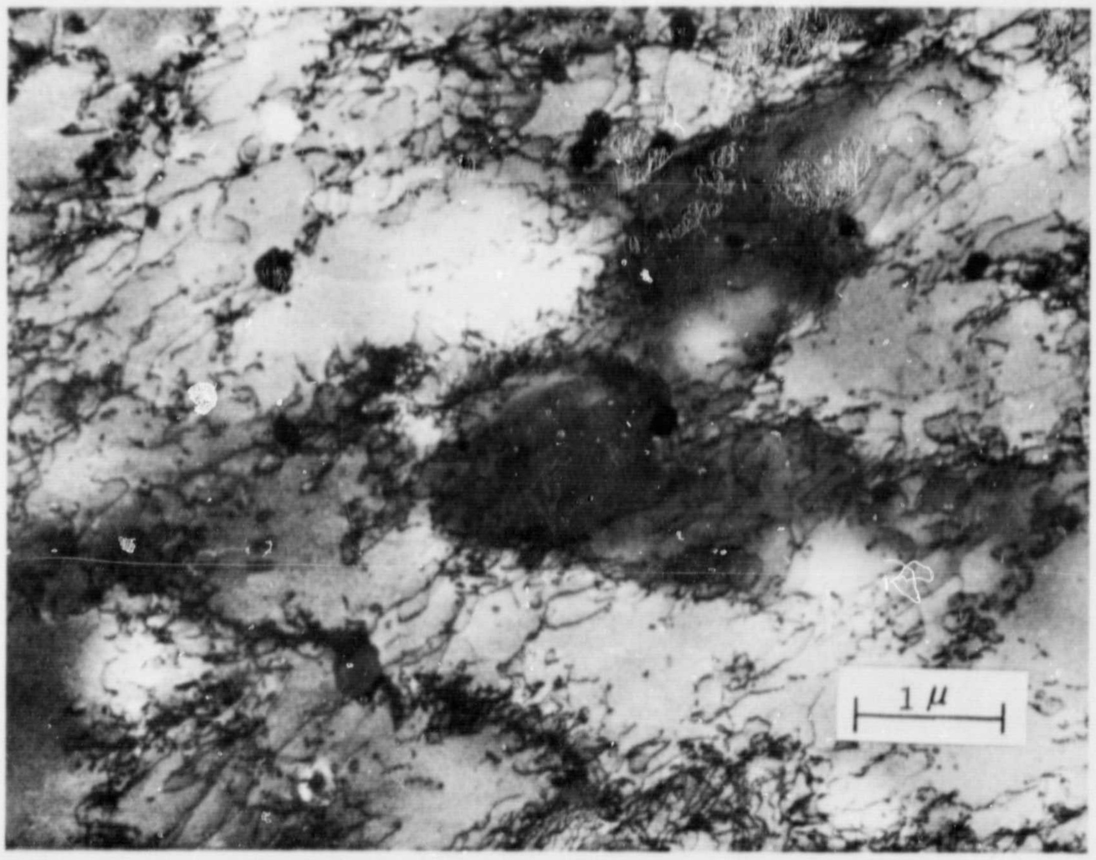
With Subgrains



B

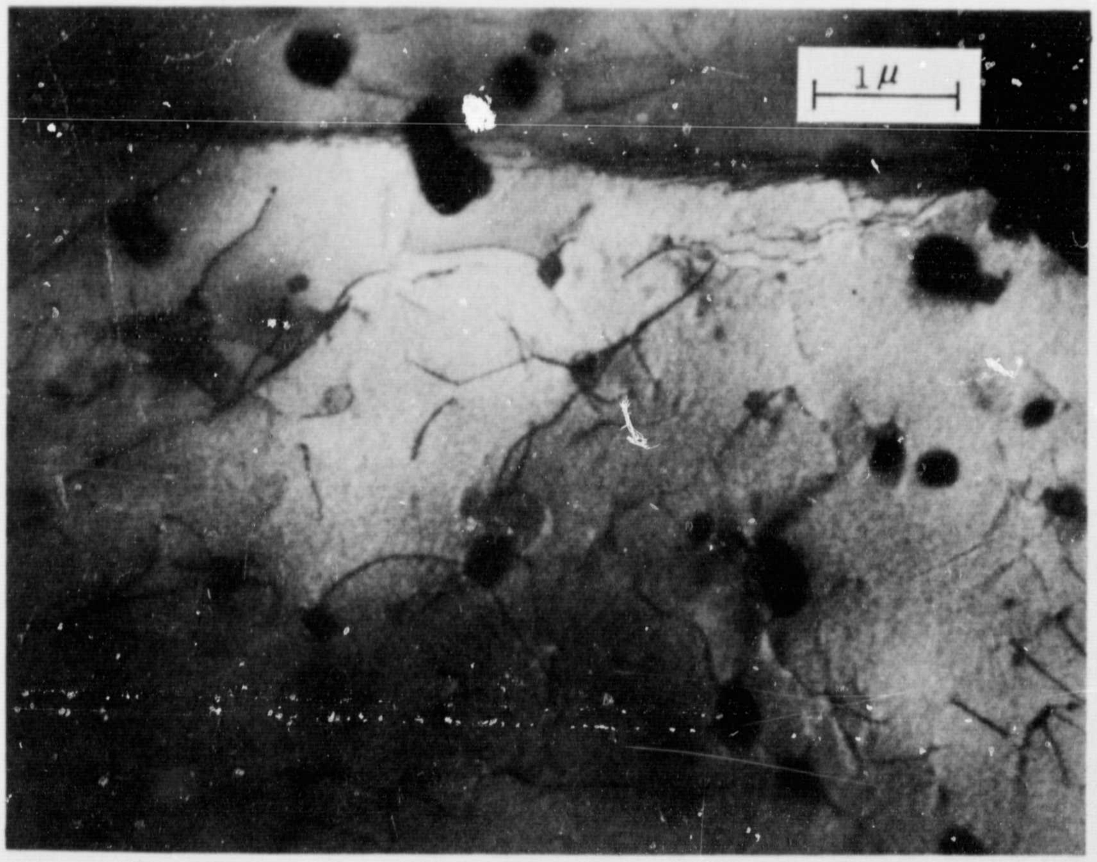
Without Subgrains

FIGURE 17. STRUCTURES FREQUENTLY OBSERVED IN D43 PRIOR TO CREEP TESTING SHOWING REGIONS WITH AND WITHOUT SUBGRAINS



A

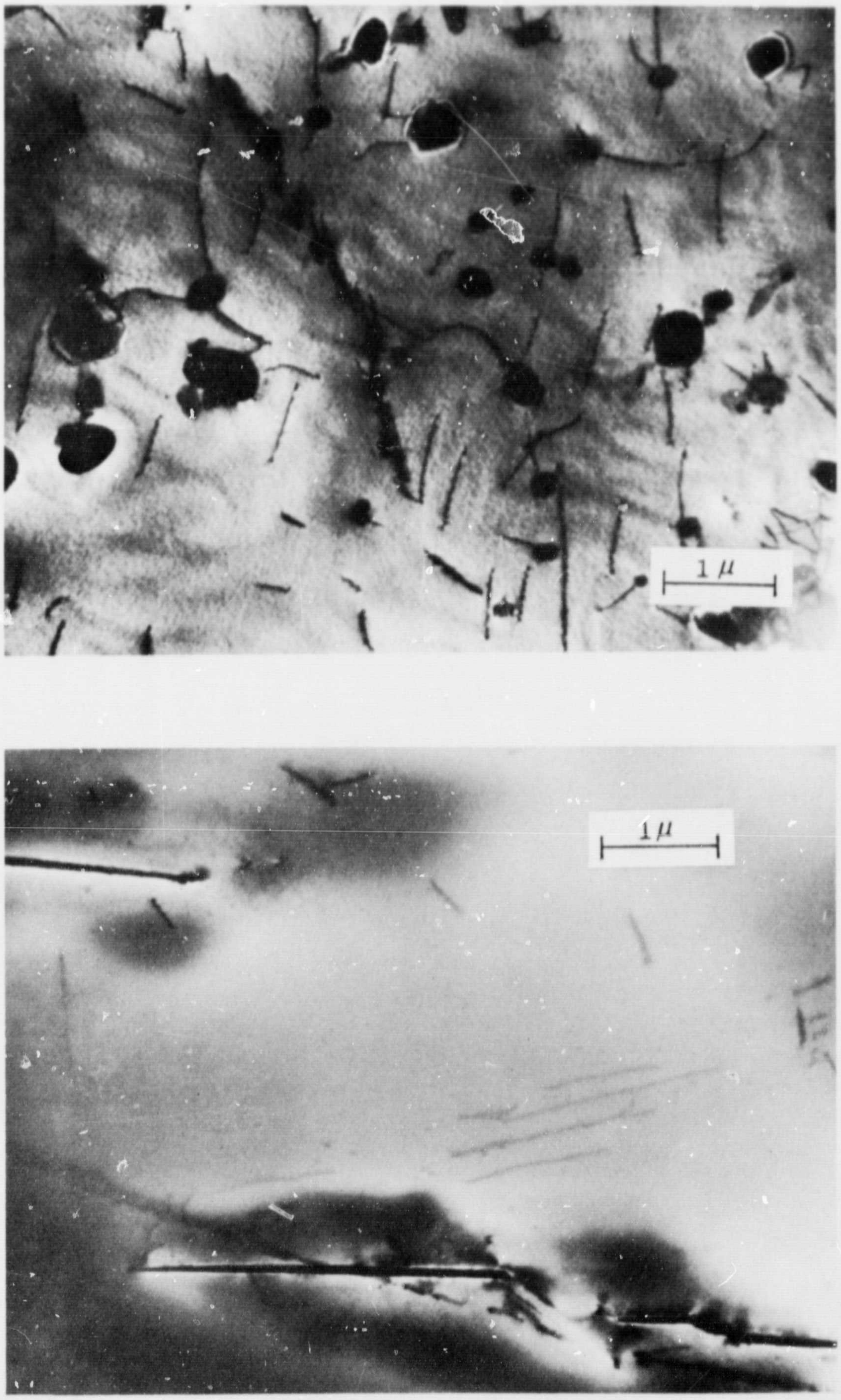
1650° F



B

2200° F

FIGURE 18. STRUCTURES AFTER CREEP AT VARIOUS TEMPERATURES
(Sheet 1 of 2)



C

2700° F

D

3100° F

FIGURE 18. STRUCTURES AFTER CREEP AT VARIOUS TEMPERATURES
(Sheet 2 of 2)

The dislocation density is lower in the specimens crept at 2200°F (Fig. 18B) than in specimens crept at 1650°F to the same strain. In addition, the dislocations are more uniformly distributed, and they nearly all lie along particles. Many instances of dislocations bowing out between adjacent particles were observed and some dislocation networks stabilized by particles were detected. However, the subgrains seen in the specimen before creep were only rarely found after creep at 2200°F. The substructure in the pre-creep specimens is apparently unstable at 2200°F. These observations suggest that interaction of dislocations with the dispersed phase is a major structural feature contributing to the strength of this alloy at 2200°F. At this temperature the movement of edge dislocations is likely to be more restricted by particles than is that of screw dislocations since the latter can bypass particles by cross-slip. Therefore, the rate controlling process for creep at this temperature is believed to be the climb of edge dislocations over particles.

As shown in Figure 18C, the structure of specimens crept at 2700°F is similar to that observed in specimens crept at 2200°F. The dislocations form a network connecting the particles throughout the matrix, and in many instances dislocations appear to bow out between adjacent particles. The structure is almost free of subgrains that characterize the alloy before creep, and no evidence was found for the formation of substructure through dislocation or dislocation-particle interactions during creep. These observations suggest that the dispersed phase is probably the important structural feature strengthening D43 during creep at 2700°F as well as at 2200°F.

After creep at 3100°F the alloy is almost free of the fine dispersion that characterized the structure of specimens crept at lower temperatures (Fig. 18D). The reduced amount of the dispersed phase is in agreement with the analyses showing a loss of both carbon and oxygen after creep at this temperature (Table I). Large acicular particles were observed in a few of the regions examined, but these particles were not generally associated with dislocations. This phase is probably Cb_2C formed on cooling from the test temperature (Ref. 23). The dislocation density was low and dislocation arrays were only rarely observed in these specimens. In general, few structural features were detected that could contribute to the elevated temperature strength of D43 during creep at 3100°F. These results suggest that this alloy is not strengthened significantly by the dispersed phase or by dislocation arrays at this temperature.

3.2.5 Creep Equation

It is often of interest to determine an empirical equation that may be useful in predicting the creep behavior of a metal. High-temperature creep data spanning a range of stress and temperature variations can often be correlated by an equation of the form

$$\epsilon^{\circ} \, e^{H/RT} = F(\sigma) \qquad (2)$$

where T is the absolute temperature and R is the gas constant. A linear plot of Equation (2) will reveal $F(\sigma)$ and the empirical equation governing creep.

The creep data over the range of stress and temperature tested are shown in Figure 19 (solid line) for a logarithmic plot of Equation (2) where $F(\sigma) = Ae^{B\sigma}$ with $H = 110,000$ cal/mole. The data in the high-temperature and low stress region are approximately linear with $B = 0.78 \times 10^{-3}$ psi^{-1}. This value of B is in reasonable agreement with the value of B determined from isothermal tests (Fig. 15). However, the data for low temperatures and high stresses show a large departure from linearity. This can be at least partly accounted for by the increase in H above 110,000 cal/mole in the low-temperature, high-stress region. The values of H required to align the low-temperature, high-stress data with the dashed line in Figure 19 were determined and are shown plotted in Figure 11 (dashed line). The experimental values of H are seen to be in rough agreement with the values derived from Figure 19 with the exception of the data for H at 1650° F. Therefore, the creep data over almost the entire range of stress and temperature can be expressed by the equation

$$\dot{\epsilon} = Ae^{B\sigma}e^{-H/RT} \tag{3}$$

where B is 0.78×10^{-3} psi^{-1}, A is a constant and H has the values taken from Figure 11.

3.2.6 Summary

In this study of the creep of D43, H, the apparent activation energy for creep, was determined in the temperature range 1650 to 3200° F. Above ~2400° F (0.55 T_M) H is 110,000 cal/mole, independent of stress. This value of H is in reasonable agreement with the range of activation energies estimated for self-diffusion in this alloy, indicating that the creep rate is controlled by a recovery process involving dislocation climb.

Below about 2400° F, H progressively increases with a decrease in temperature. This behavior is somewhat unexpected since in a number of metals H decreases below 0.5 T_M with decreasing temperature. One such mechanism thought to account for values of H exceeding that for self-diffusion in other systems is an interaction between solutes (or solutes in combination) and moving dislocations. The temperature range of the increase in H is probably too high to be caused by an interstitial dislocation interaction. However, the mobility of certain solute combinations may be of the correct order to account for the increase in H determined in this study.

An examination of the structure at 2200 and 2700° F shows that the dispersed phase is the primary structural feature obstructing dislocation movement. In this temperature range the movement of screw dislocations is likely to be less restricted

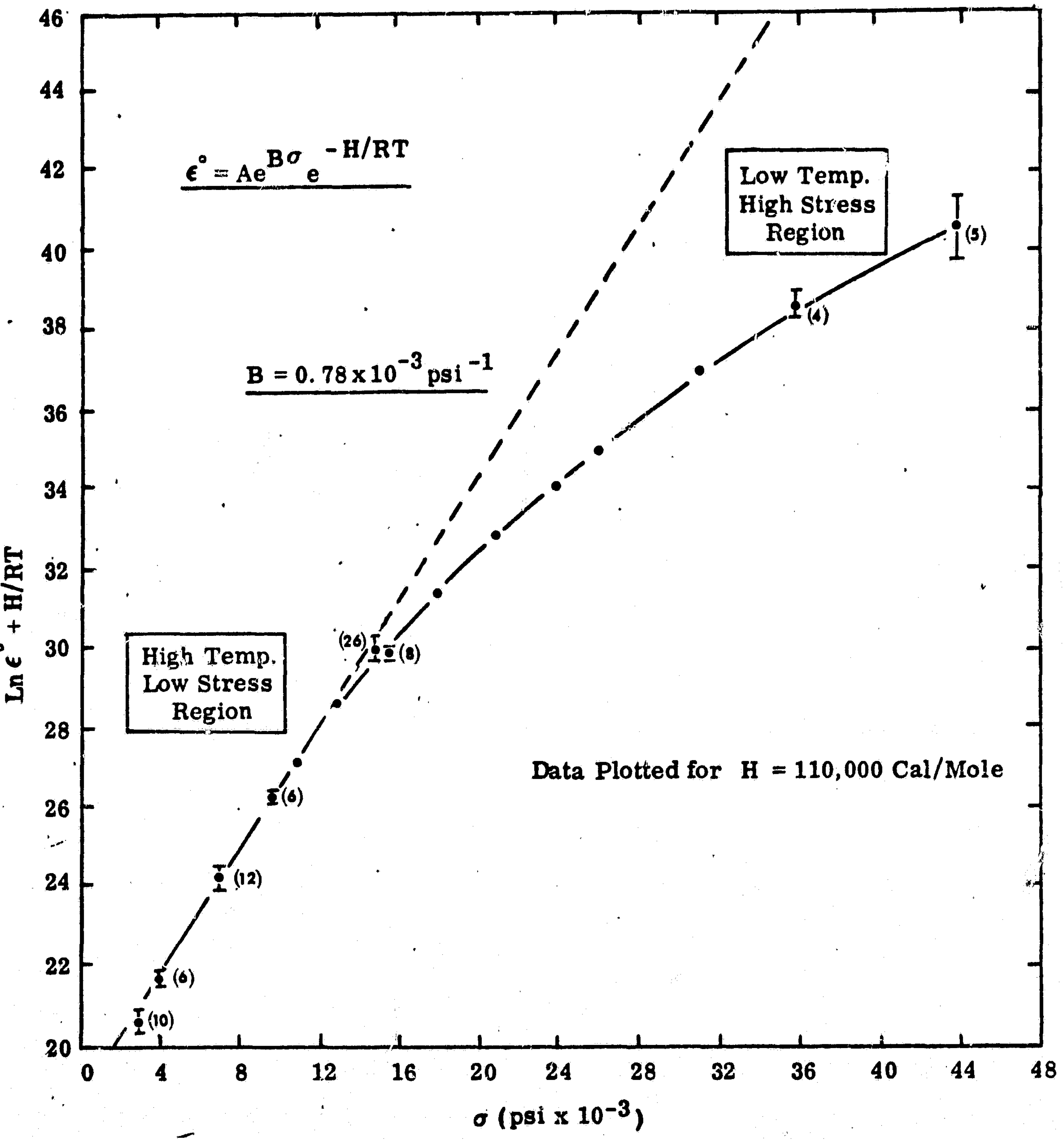


FIGURE 19. TEMPERATURE COMPENSATED CREEP RATE AS A FUNCTION OF APPLIED STRESS

by particles than is the movement of edge dislocations. Therefore, it is likely that the rate controlling creep mechanism in the region where H is near 110,000 cal/mole (2300-2400° F) to temperatures at least as high as 2700° F is the climb of edge dislocations over the dispersed phase. Between 2700 and 3200° F, H remains constant even though there is a drastic change in the equilibrium or steady-state creep structure. At 3100° F the alloy is no longer dispersion strengthened for the conditions of these tests and, in fact, it contains few short range structural features that would require dislocation climb for deformation to continue. Therefore, the rate controlling process may simple be the glide of dislocations in the lattice. For example, it has been proposed that the activation energy for creep at high temperatures is that for the nonconservative movement of jogged screw dislocations, a diffusion controlled process (Ref. 24 thru 26).

The stress dependence of the creep rate was determined from isothermal tests at 1750, 2200 and 2800° F. The creep rate, ϵ°, was found to be proportional to $e^{B\sigma}$ where σ is the stress and B is 0.6 to 0.8 x 10^{-3} psi^{-1}. Over a narrow range of stress variations, the stress dependence of the creep rate also can be described by the power stress law $\epsilon^\circ \alpha \sigma^n$. Over the range of stress tested, n varies from 3 to 30 in accordance with the relation $n = \sigma B$. At the low temperature where the structure of the alloy is more worked, n is near 30. At the high temperature where the alloy tends to be strengthened less by the dispersion, n is near 3.

The creep rate over the range of stress (3,000 to 44,000 psi) and temperature (1650-3200° F) tested can be expressed empirically by the equation

$$\epsilon^\circ = Ae^{B\sigma}e^{-H/RT} \qquad (4)$$

where B is ~0.8 x 10^{-3} psi^{-1}, A is a constant and H has the approximate values shown in Figure 11.

3.3 COLUMBIUM AND TANTALUM ALLOYS

Activation energies were determined as a function of strain and temperature for the three columbium-base alloys Cb-1Zr, C103 (Cb-10Hf-1Ti-0.5Zr), C129Y (Cb-10W-10Hf-0.1Y) and for the tantalum-base alloy T222 (Ta-10W-2Hf). Specimens tested below 2800° F were wrapped with foil, as previously described, to minimize interstitial contamination during testing. Specimens tested at 2800° F and higher were not wrapped because sticking of the foil to the gage length of the specimen interferred with creep measurements. However, the results for D43 suggest that changes in interstitial concentration that may occur during testing at the higher temperatures do not affect H.

Most of the activation energy measurements were made over a range of strain using a constant stress throughout the test. However, in some cases the stress was changed during the test to keep the strain rate at a convenient level. Below about 2900° F, these changes in stress did not noticeably affect the activation energies derived from these tests. Several examples of stress changed made during tests are shown in Figure 20.

At temperatures below about 2900° F, the activation energies were ordinarily approximately independent of creep strain. However, as shown in Figures 21 through 24, some exceptions were noted. At lower temperatures, the activation energy showed a tendency to decrease with increasing strain. This behavior is especially evident for Cb-1Zr at 1850° F (Fig. 21) and for T222 at 2350° F (Fig. 24). In addition, at some of the higher temperatures there was a tendency for the activation energy for creep to increase with increasing strain (e.g., C103 and C129 in Fig. 23). Activation energy measurements for these alloys were ordinarily made in regions of approximately steady-state creep. However, as shown in Figure 25, these alloys did not always attain a true steady-state creep condition over the entire range of strain tested. For example, the creep rates of T222 and C129Y increase at 2350° F during the initial part of the tests indicating that the structure is becoming less creep resistant for these alloys. Changes in structure during creep may be caused by solutioning, precipitation or overaging of interstitial dispersions in the alloys and the associated changes in dislocation or grain structure. In view of the sensitivity of H for columbium to its structure, the variation in H with strain for the alloys may also be associated with structural changes that occur during creep. For T222 steady-state creep and thus a stable structure are not approached until six to ten percent strain has been attained at 2350° F (Fig. 25). This is also the strain at which H for T222 approaches a constant value at this temperature (Fig. 24). This behavior is consistent with that observed for columbium and D43 and suggests that as the structure approaches a stable configuration at high strains or during prolonged testing, H approaches a constant value typical of the equilibrium structure.

The activation energies over a temperature range are shown in Figure 26 for columbium and the columbium alloys and in Figure 27 for T222. The results for D43 are not shown since they have been previously presented and since H for D43 shows no anomolous behavior at high temperatures. The data plotted in these figures are the average values of H for the first ten to 15 percent creep strain except where H is an obvious function of strain (e.g., T222 at 2350° F, Fig. 24). For the latter cases, the values of H at the higher strains were used since these values appear to be closer to those for the equilibrium structure.

The values of H for columbium are generally lower than those for the alloys below about 0.5 to 0.6 T_M. This suggests that alloying increases H in this temperature region. However, as previously pointed out, H for columbium is a sensitive function

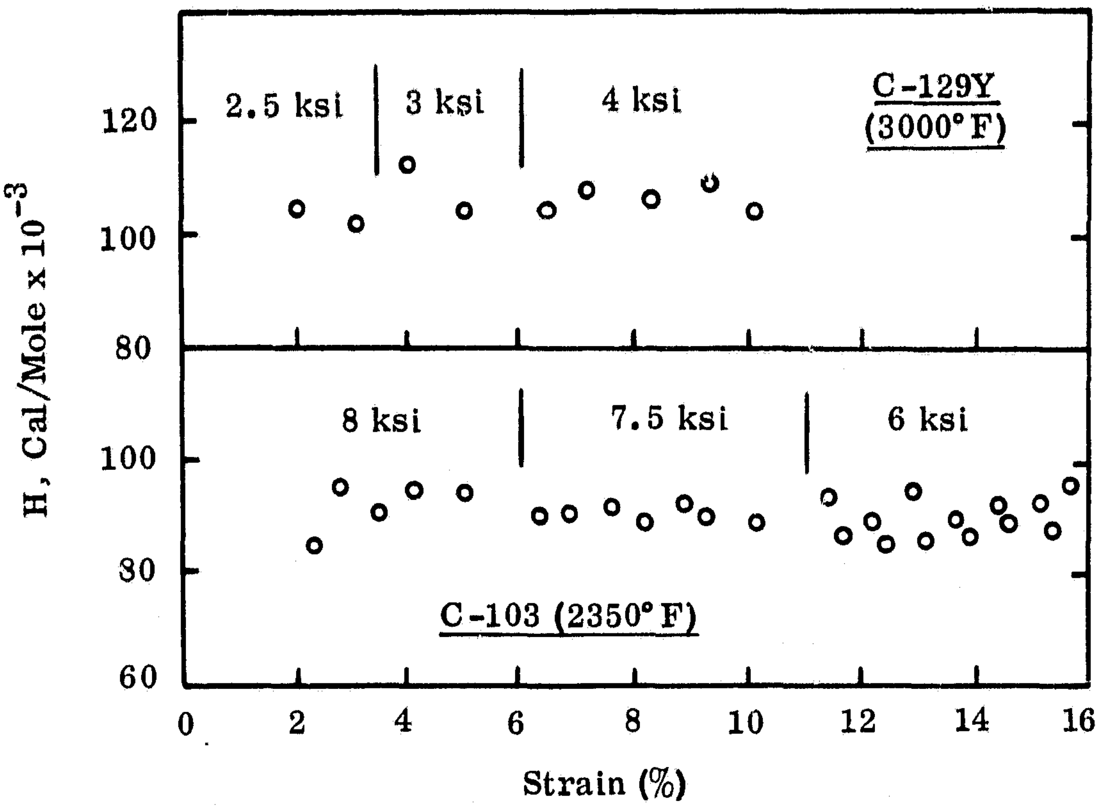


FIGURE 20. ACTIVATION ENERGY FOR C103 AND C129Y AT DIFFERENT STRESSES

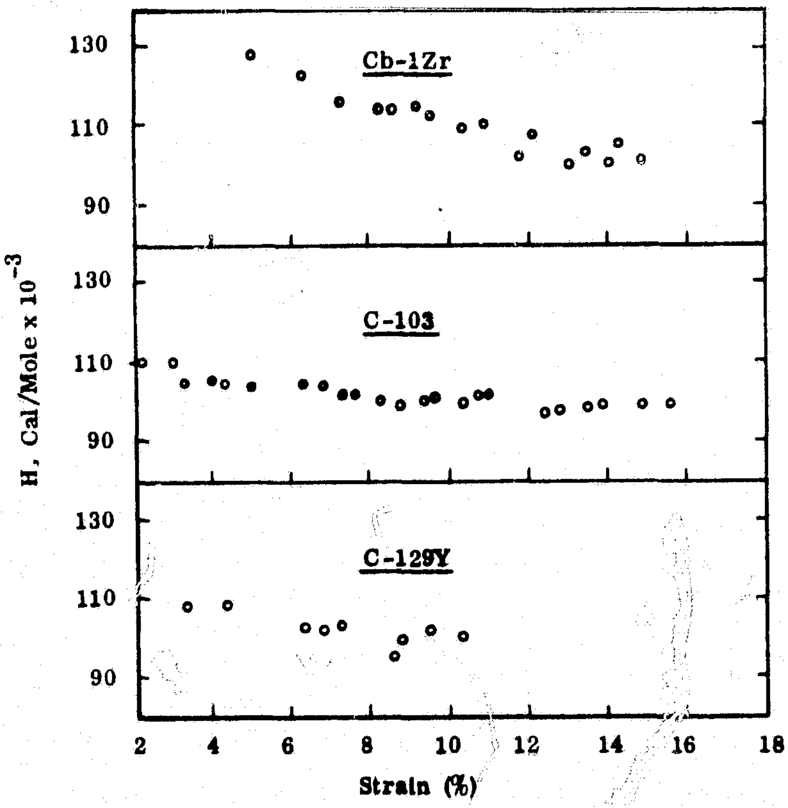


FIGURE 21. ACTIVATION ENERGY AS A FUNCTION OF STRAIN AT 1850°F FOR THREE COLUMBIUM-BASE ALLOYS

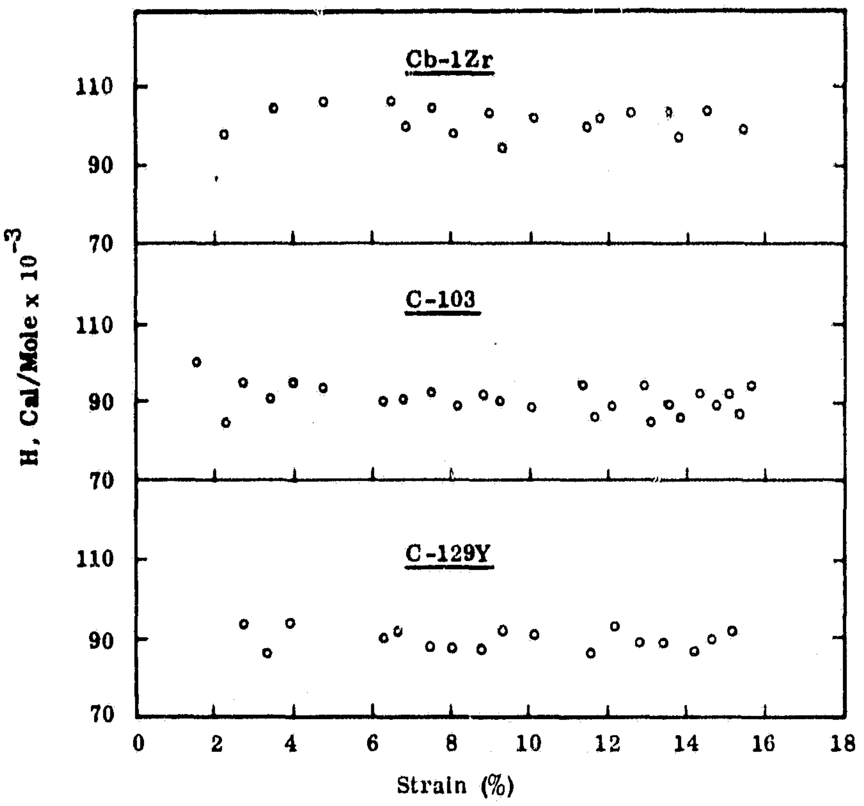


FIGURE 22. ACTIVATION ENERGY AS A FUNCTION OF STRAIN AT 2350°F FOR THREE COLUMBIUM-BASE ALLOYS

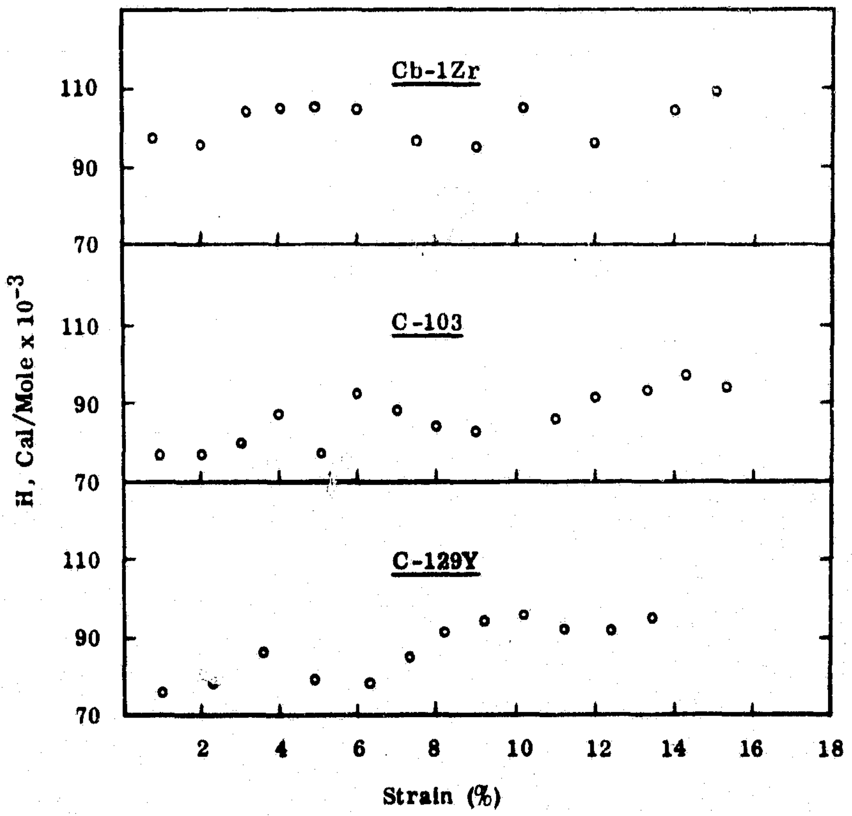


FIGURE 23. ACTIVATION ENERGY AS A FUNCTION OF STRAIN AT 2650°F FOR THREE COLUMBIUM-BASE ALLOYS

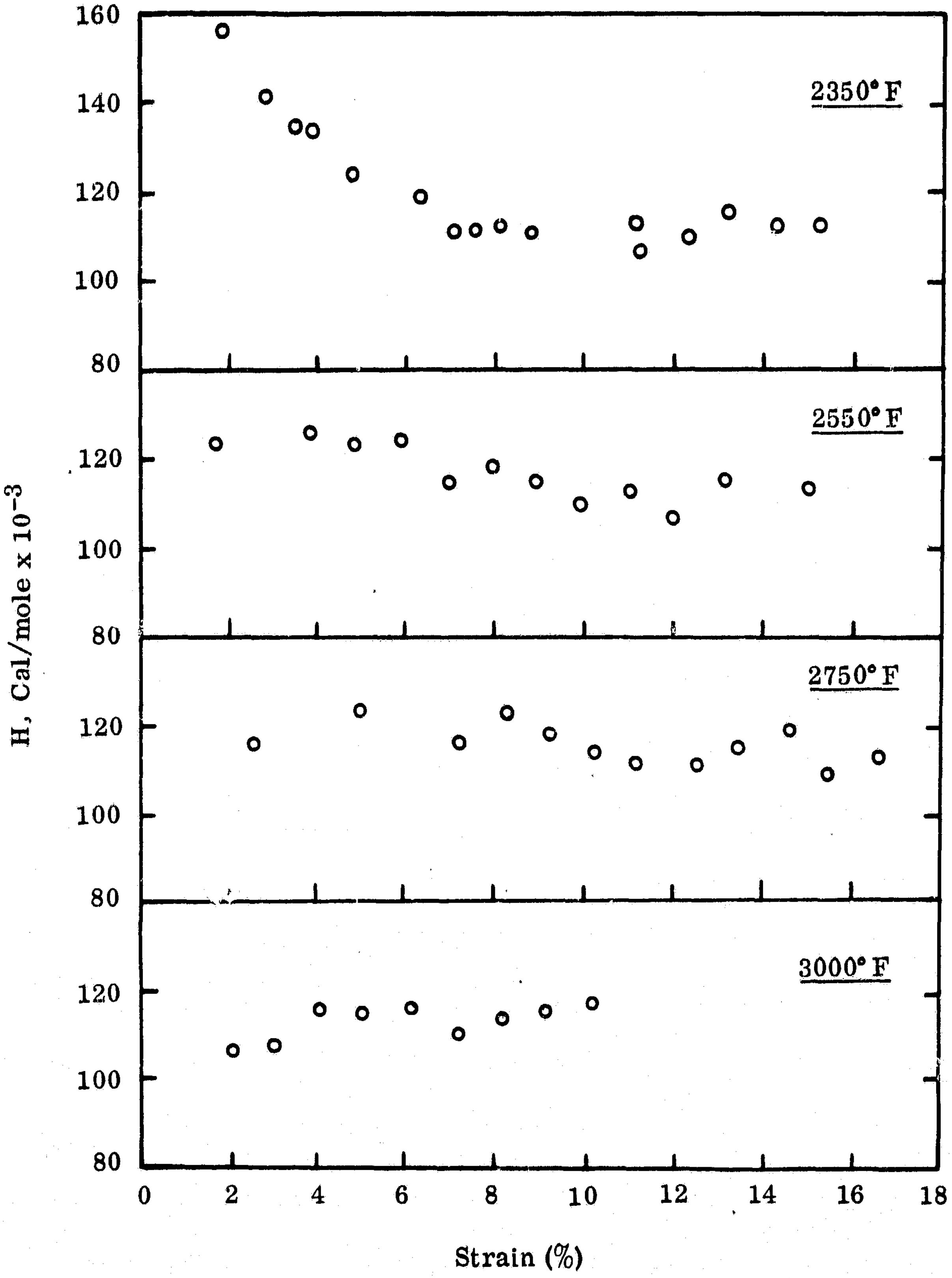


FIGURE 24. ACTIVATION ENERGY AS A FUNCTION OF STRAIN AT DIFFERENT TEMPERATURES FOR T222

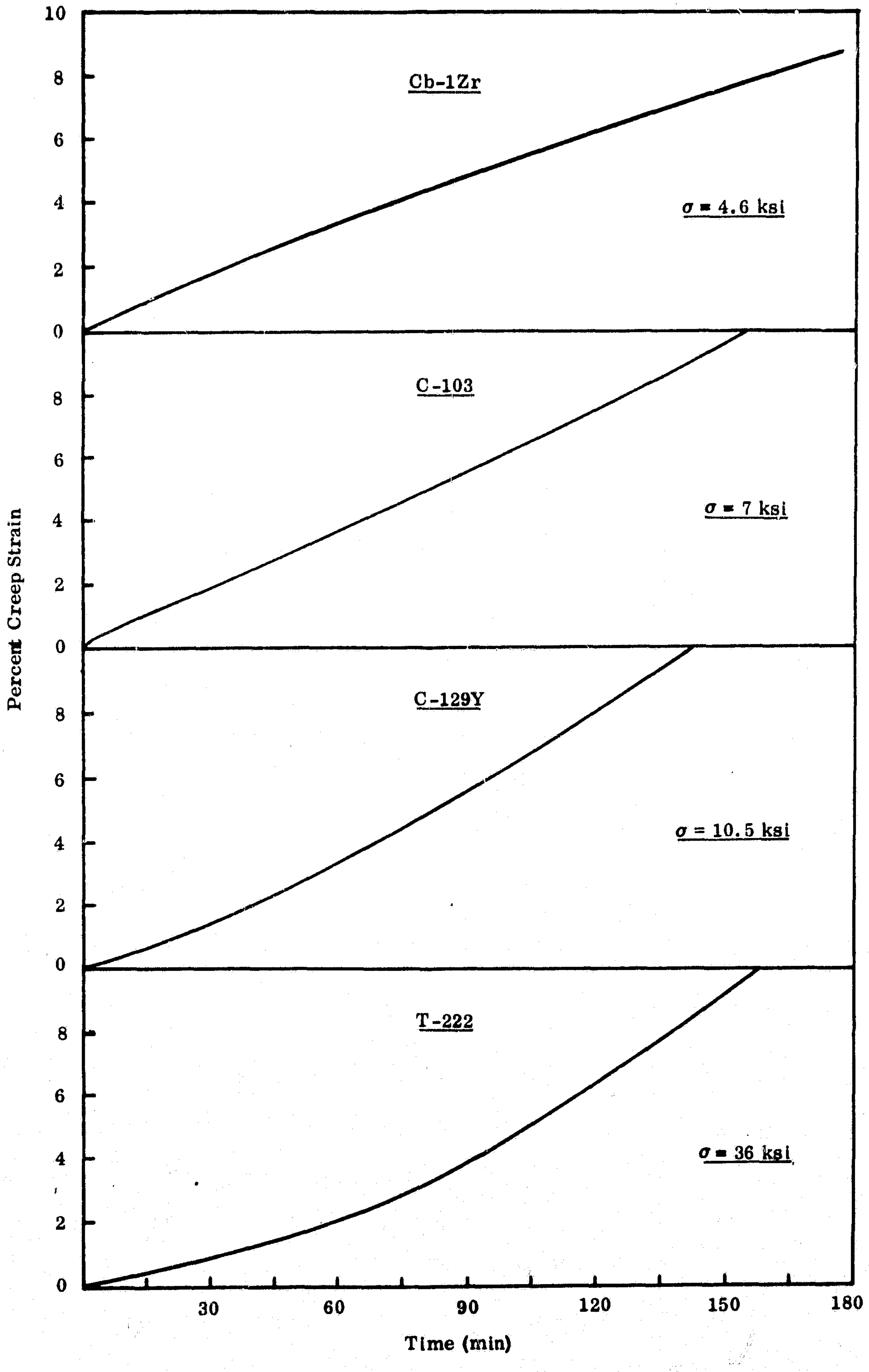


FIGURE 25. CREEP OF COLUMBIUM AND TANTALUM ALLOYS AT 2350°F

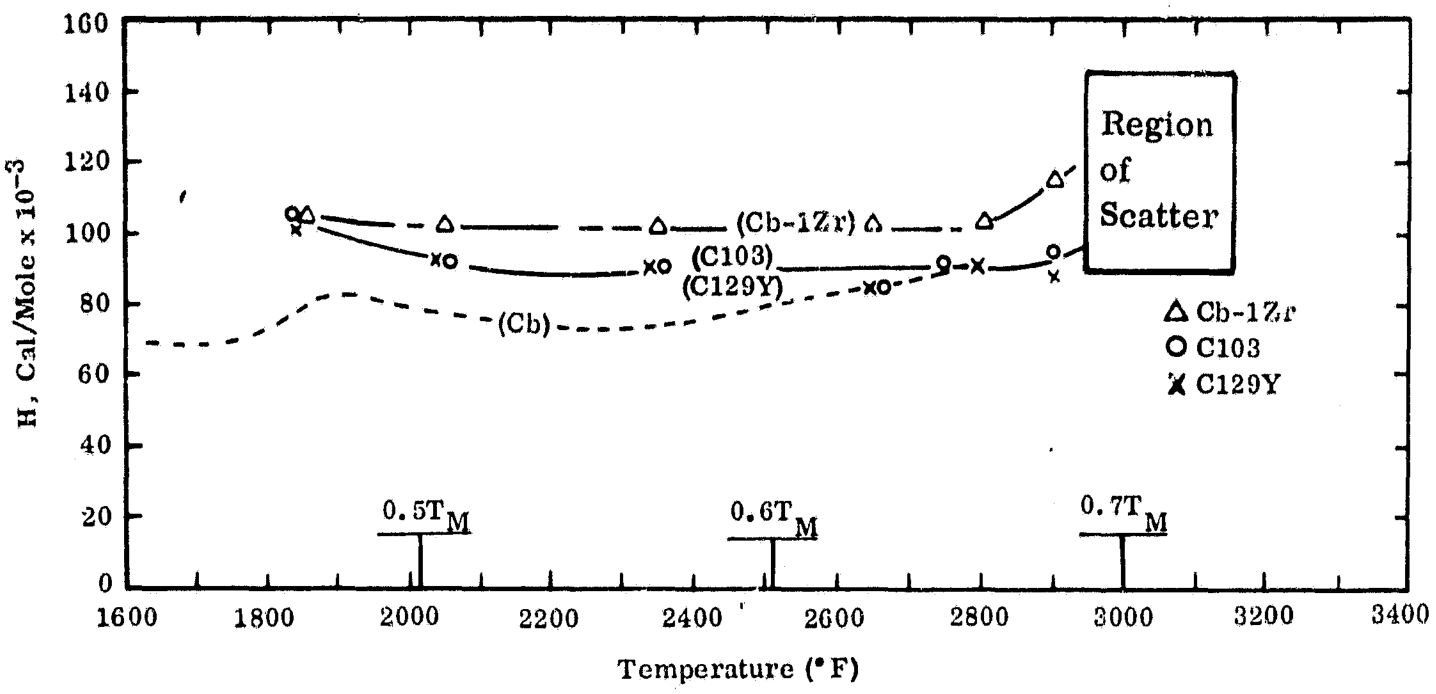


FIGURE 26. ACTIVATION ENERGY FOR CREEP OF COLUMBIUM AND COLUMBIUM ALLOYS OVER A RANGE OF TEMPERATURE

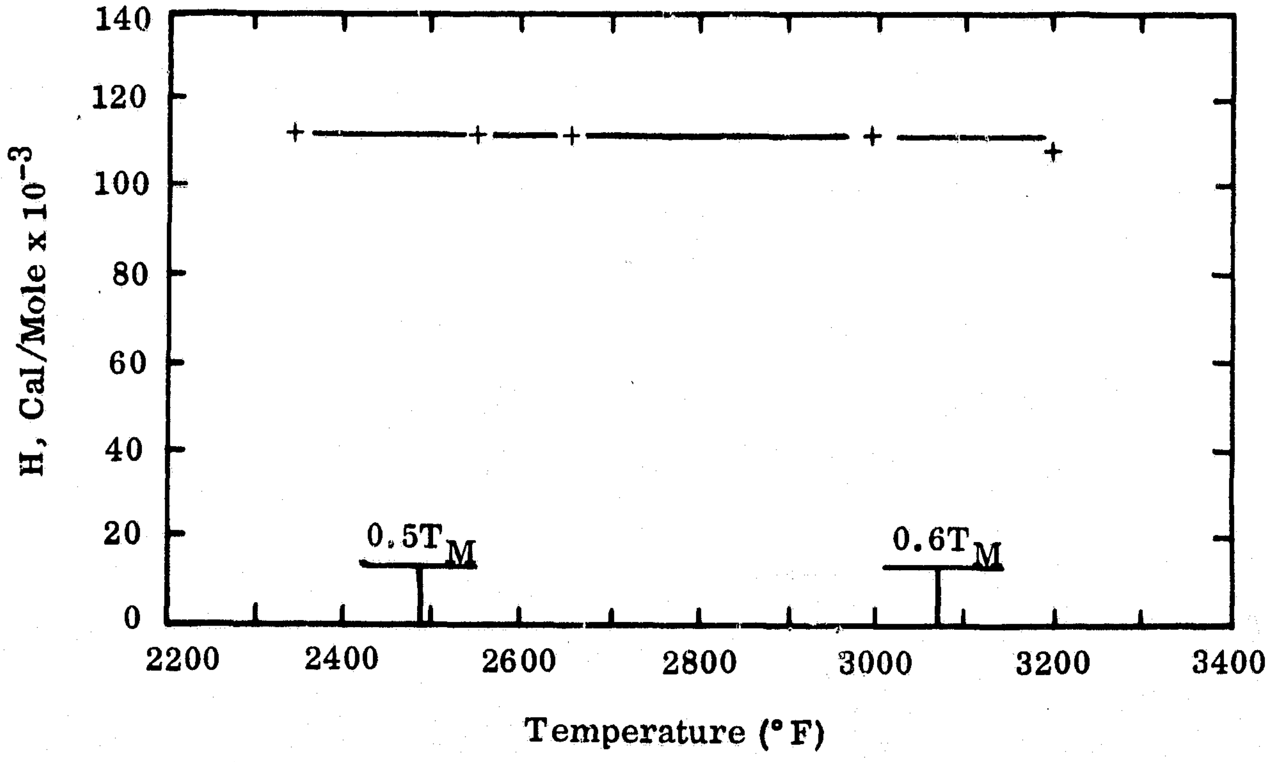


FIGURE 27. ACTIVATION ENERGY FOR CREEP OF T222 OVER A RANGE OF TEMPERATURE

of the interstitial concentration and the structure at the lower temperatures. Therefore, it is difficult to derive quantitative information concerning the effect of solute additions on H in this temperature region. Above about 2200°F, H for columbium increases with increasing temperature and appears to be approaching the activation energy for self-diffusion (96 k cal/mole). Near 2700°F the value of H for columbium is about the same as that for C103 and C129Y. In future work the activation energy temperature profile for columbium will be extended to higher temperatures to use as a basis of comparison with the activation energies for the various alloys.

Towards the end of the period covered by this report, anomalous results were obtained in the temperature range above 2900°F. Insufficient work was performed before the end of the period to permit a plot to be made with some degree of certainty; hence the results are indicated in Figure 26 by the shaded area marked "region of scatter". The trend was for the activation energies to begin to increase to a peak, but the results were found to be stress- and strain-dependent, further complicating analysis of the data. To avoid presentation of data prematurely, it is felt that more extensive investigation of the region should be made before any conclusions are drawn.

As shown in Figure 27, H for T222 is almost constant over the entire range of temperatures for which data have been obtained. However, on the basis of the behavior observed for the columbium alloys, increasing values of H would not be anticipated until temperatures near 0.7 T_M are reached.

4

SUMMARY AND CONCLUSIONS

The influence of solutes on the creep strength of refractory alloys is being studied through their effect on the activation energy for creep, H. This investigation should provide information concerning the role that various solutes play in strengthening refractory metals. During this period, some of the factors that could obscure the effects of solutes on H were studied. In addition, activation energy - temperature profiles were determined for columbium and some columbium and tantalum-base alloys.

The results for columbium and the columbium and tantalum alloys suggest that H is influenced by the structural changes that occur during primary creep and structural changes induced during pre-creep treatments. However, H appears to approach a constant value as the structure approaches an equilibrium configuration for the conditions of the test. The activation energy also appears to be influenced by the interstitials in columbium and the columbium alloys. The results for columbium and D43 suggest that interstitials do not significantly affect H at temperatures above 0.5 - 0.6 T_M. The stress does not seem to be an important factor affecting H above about 0.4 T_M.

The activation energy - temperature profiles were determined for the columbium alloys D43, C103, C129Y, and Cb-1Zr, and for the tantalum-base alloy T222, in the range 0.5 - 7.0 T_M. H for the alloys C103, C129Y and Cb-1Zr show anomalous behavior above about 2900° F.

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FUTURE WORK

The activation energy - temperature profiles for columbium and the commercial columbium and tantalum alloys will be extended to higher temperatures to better define temperature regions where the activation energies may be anomolously high. The creep behavior in these regions will be analyzed to determine if an increase in activation energy is accompanied by an increase in strength.

The commercial alloys are probably too complex to sort out the effect of individual solutes on the activation energy and creep strength. Most of the alloys tested contain muliple solutes and interstitial phases. Differences in structure and composition can affect the creep strength in a complex way by changing the pre-exponential term in the creep rate equation rather than by affecting the activation energy for creep. A better correlation between H and creep strength should be possible for alloys with less component solutes and with more nearly constant structures. Therefore, in future work, binary columbium-base alloys will be arc melted into buttons and rolled into shapes suitable for machining into creep specimens. The binary additions may include those in the commercial columbium alloys being tested as well as those frequently used to strengthen other columbium-base alloys. In this way, it may be possible to sort out the effect of various solutes on the activation energy and creep strength of columbium and to combine solutes to develop more complex creep resistant alloys.

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