

STRUCTURAL AND MECHANICAL EFFECTS
OF INTERSTITIAL SINKS

by

Mark J. Klein

January 1968

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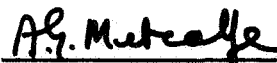
ABSTRACT

The effects of a titanium interstitial sink on the interstitial concentration and the recrystallization of TZM and T222 were studied in the temperature range 2000 F to 2700 F. The grain structure of these alloys was not significantly affected by the action of the titanium sink. The carbon concentration of the TZM, however, was reduced to less than one-half of its initial concentration when this alloy was vacuum annealed with or without titanium. The carbon concentration of the T222 was not reduced by the same annealing treatments.

The activation energy for creep of D43 was determined from 1600 F to 3200 F. Above about 2200 F the activation energy for creep is equal to that for self-diffusion and is independent of stress and temperature. Below about 2200 F preliminary results indicate that the activation energy for creep is greater than that for self-diffusion.

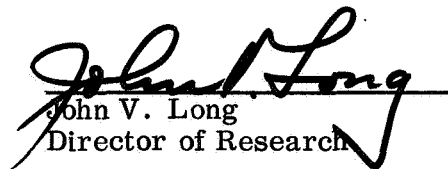


M. J. Klein
Senior Staff Engineer



A. G. Metcalfe,
Associate Director of Research

Approved:



John V. Long
Director of Research

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I. INTRODUCTION

This is the fifth quarterly progress report for Contract NAS-7-469, "Structural Effects of Interstitial Sinks," covering the period September 8 through December 8, 1967.

In many practical applications it is necessary to use a refractory metal at elevated temperatures in contact with a more reactive metal. Under these conditions the interstitial elements in the refractory metal tend to concentrate in the more reactive metal (interstitial sink) causing a serious decrease in the elevated temperature strength of the refractory metal. This investigation is a study of the changes in structure and mechanical properties of columbium base alloys and other refractory metal alloys induced by the action of a titanium interstitial sink. The objective of this study is to obtain a better understanding of the relationships between the changes in interstitial concentration, the structure and the elevated temperature strength of refractory metals when they are placed in contact with an interstitial sink.

The progress during the preceding period is reported in the quarterly report for June 8 through September 8, 1967. During the preceding period the effect of the chemical potential of an interstitial sink on the residual carbon concentration and the creep rate of D43 were studied. The creep rate was found to be inversely proportional to the carbon concentration remaining in the D43 after this alloy had been subjected to an interstitial sink, and the activation energy for creep at 2200 F was found to be approximately equal to that for self-diffusion.

During this period, the activation energy for creep of as-received D43 was determined over the temperature range 1600 to 3200 F. In addition, the effects of a titanium sink on the structure and interstitial concentration of TZM and T222 were studied.

II. RESULTS AND DISCUSSION

The experimental procedures used in this investigation are presented in the Interim Technical Report for the period June 8, 1966 through June 8, 1967. The D43 alloy used for creep studies this period was that processed by Fansteel in the duplex condition. The T222 (Ta-9W-2.3Hf-0.02C) and TZM (Mo-0.5Ti-0.1Zr-0.02C) alloy sheets used for the interstitial sink work were obtained from Universal Cyclops and Fansteel Companies respectively.

In the first sub-section, data are presented showing the variation in the activation energy for creep of D43 as a function of the creep temperature. In the second sub-section the results of a study of the effects of a titanium interstitial sink on the interstitial concentration and annealing behavior of T222 and TZM are presented.

2.1 THE ACTIVATION ENERGY FOR CREEP OF D43 AS A FUNCTION OF TEMPERATURE

Activation energies were determined, as previously described, by measuring the changes in strain rates induced by abrupt shifts in temperature during constant stress creep tests. The variation in the activation energies for creep as a function of the creep temperature is shown in Figure 1. A solid line is drawn through the average activation energy values, and the approximate activation energy for self-diffusion in this alloy is indicated in the center of the diagram.

Above about 2200 F ($\sim 0.5 T_M$) the activation energy for creep is about equal to that for self diffusion (within a scatter range of about ± 10 K cal/mole). As previously discussed, this agreement is consistent with recovery creep where the creep rate is controlled by a dislocation climb mechanism. During these tests it was observed that the activation energy above 2200 F is independent of the stress within the range of stress values tested (3000 psi to 15,000 psi). This stress independence of the activation energy is also a characteristic of recovery creep where the creep rate is controlled by a dislocation climb mechanism.

Additional work will be required to reduce the scatter in data points below 2200 F. However, the results thus far indicate that for a range of temperatures below 2200F, the activation energy for creep is greater than that for self-diffusion. This is

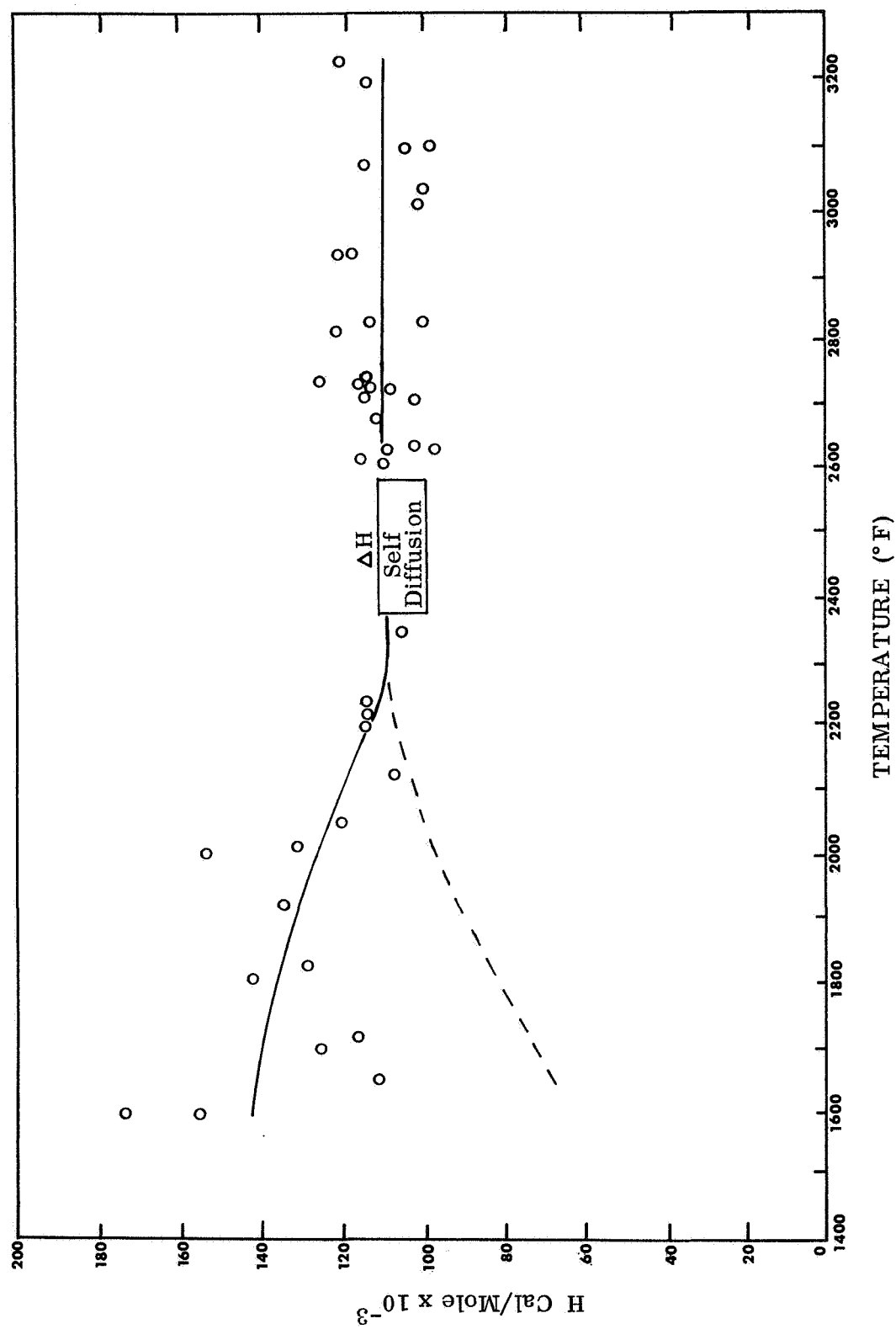


FIGURE 1. THE ACTIVATION ENERGY FOR CREEP OF D-43 AS A FUNCTION OF TEMPERATURE

surprising since below about $0.5 T_M$ the measured activation energy for creep is expected to decrease. The dashed line in Figure 1 shows the decrease expected based on results for face-centered cubic metals. In this temperature region the measured activation energy, $H^\#$ is believed to be stress dependent and to be defined by an equation of the form

$$H^\# = H^\circ - v \sigma^\# \quad (1)$$

where $H^\#$ is the measured or apparent activation energy, v is a coefficient of proportionality between stress and energy and $\sigma^\#$ is the stress effective in overcoming the energy barrier, H° . Activation energy measurements over a range of temperatures are ordinarily made at low stresses at high temperatures and high stresses at low temperatures so that the strain rate is maintained approximately constant, and the tests can be completed in a reasonable length of time (a procedure followed in obtaining the activation energy values shown in Figure 1). According to Eq.(1), $H^\#$ will thus decrease with decreasing temperatures. This is in contrast with the experimental results shown in Figure 1 where $H^\#$ increases with decreasing temperature despite an increase in stress of from 15,000 psi at 2200 F to 44,000 psi at 1600 F.

The increase in $H^\#$ below 2200 F could be accounted for if solute atoms interact with dislocations in this temperature region and affect their mobility. The activation energy for creep deformation may then reflect the energy required for a dislocation to overcome its atmosphere of solute atoms in addition to the normal barriers to dislocation movement(1).

The conditions of temperature and strain rate under which an interaction between solute atoms and moving dislocations can be expected has been analyzed by McLean(2) and Cottrell (3). Their derivations are based on equating the mobility of the solute atoms (the first term in the Eq. 2) to the velocity of the dislocations (the second term in Eq. 2) so that

$$\frac{\sigma b^2 D}{10kT} = \frac{\epsilon^\circ}{\rho b} \quad (2)$$

In this equation σ is the stress, b is the Burgers vector, k is the Boltzmann constant, T is the temperature, ϵ° is the strain rate, ρ is the dislocation density and D is the diffusion coefficient.

Where $\rho = 10^9$, $b = 2.5 \times 10^{-8}$ cm, $\sigma = 10 \times 10^8$ dynes/cm² (about 15,000 psi) and $T = 1365^\circ\text{K}$ ($\sim 2000^\circ\text{F}$),

$$D = 1 \times 10^{-7} \epsilon^\circ. \quad (3)$$

Since H/kT in the diffusion equation is approximately equal to $17T/T_M$ and the frequency factor is about unity,

$$\frac{T}{T_M} = \frac{17}{16 - \ln \epsilon^\circ}. \quad (4)$$

Equation (3) and (4) thus express very approximately the conditions of strain rate and temperature where interaction between substitutional solute atoms and moving dislocations could lead to an increase in the activation energy for creep as previously described.

For D43, where $\epsilon^\circ = 7 \times 10^{-6}$ /sec (the approximate ϵ° used in these tests) and interaction between substitutional elements and dislocations would be expected to occur according to Eq.(4) at $0.6 T_M$ or about 2650°F . In this regard, Eq.(4) is not a sensitive function of the assumed parameters, eg., an increase in ρ to 10^{10} decreases T/T_M to 0.56. Thus for reasonable values of the parameters the temperature derived from Eq.(4) is considerably higher than the temperature indicated in Figure 1 (about 1800°F or $0.44 T_M$) at which the activation energy appears to rise above that for self-diffusion. The increase in activation energy is not likely to be caused by interstitial interactions with dislocations because of the high mobility of interstitials in this temperature range. The apparent diffusivity of the element responsible for the increase in activation energy below 2200°F in D43 can be calculated according to Eq.(3) for the strain rate used in these tests. Where ϵ° is 7×10^{-6} /sec, D is approximately 10^{-12} cm²/sec. The interaction is probably not due to the diffusion of Zr that may be in solution in D43 since its diffusivity at 1800°F in Cb is reported to be $\sim 10^{-17}$ cm²/sec.⁽⁴⁾ The diffusion of W in D43 will probably be even slower than Zr so that it is not likely to account for the increase in the activation energies. Therefore, some more complex diffusing species must account for the observed results. In this regard, where clustering of solutes affects their mobility or where solutes interact with each other, as well as with dislocations, the temperature range of the interaction may be shifted or broadened according to the mobility of the diffusing species.

2.2 EFFECT OF TITANIUM ON THE STRUCTURE AND INTERSTITIAL CONCENTRATION OF TZM AND T222.

Diffusion couples were prepared by diffusion bonding 0.002 inch titanium foil to nominally 0.030 inch T222 and TZM sheet specimens 0.3 inch wide and 2.5 inches long. The bonding conditions used were 15 seconds at 2200 F with a bonding pressure of 8,500 psi. The titanium foil (Ti-70A) was bonded to both surfaces of flat alloy specimens making Ti/alloy/Ti sandwiches.

All of the diffusion couples were then vacuum annealed to induce interstitial partition to the titanium sink. The structure and interstitial concentration of part of the specimens were then compared with similarly heat treated specimens that had not been bonded to a titanium sink. These results are discussed in the first section below, Change in the As-Received Structures and Interstitial Concentrations Induced by Sink Annealing.

The remaining T222 and TZM diffusion couples that had been heat treated to induce interstitial partition to the titanium were cold rolled to 75 percent reduction in area and heat treated again at various temperatures to induce recrystallization. These structures were then compared with the structures of specimens that had not been bonded to titanium but had received the same thermal and mechanical treatments. These results are discussed below in the section, Recrystallization Studies after Sink Annealing and Deformation.

2.2.1 Changes in the As-Received Structures and Interstitial Concentrations Induced by Sink Annealing.

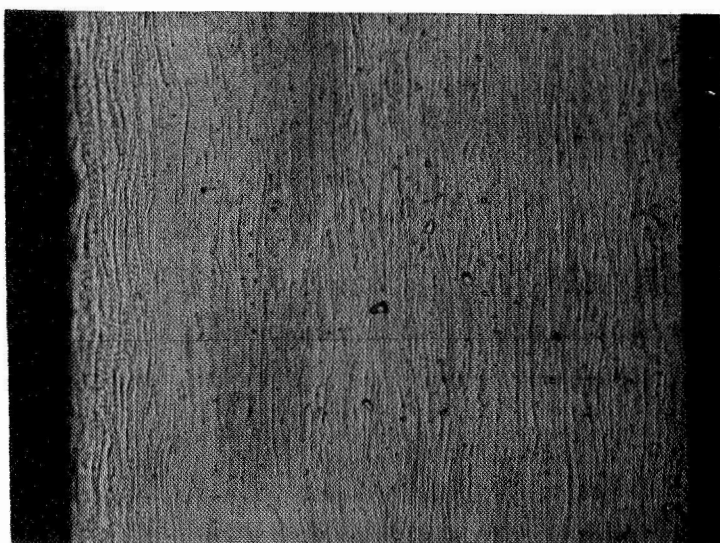
Figure 2 shows the Ti/alloy structures after diffusion bonding. The structure of the T222 and TZM are not influenced by the titanium beyond a narrow diffusion zone between them. The grains in the TZM are deformed and elongated in the direction of rolling, whereas the grains in the T222 are relatively equiaxed.

The structures of the as-received TZM alloy after heat treatments with and without titanium foil are shown in Figures 3 and 4. Heating the TZM (Figure 3) at 2000 F, both with and without titanium, induces a coarsening of the cold-worked structure without evident recrystallization. After the 2500 F heat treatments (Figure 4) the TZM is fully recrystallized for specimens heated with and without a titanium sink. The grain structure after heating one hour at 2500 F without titanium appears the same as that after heating one hour at 2500 F with titanium; however, grain



TZM

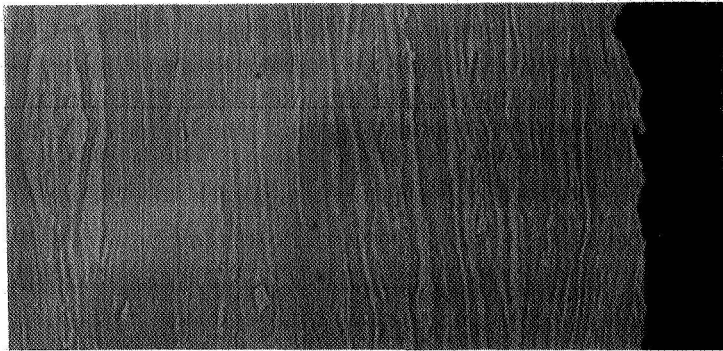
Ti



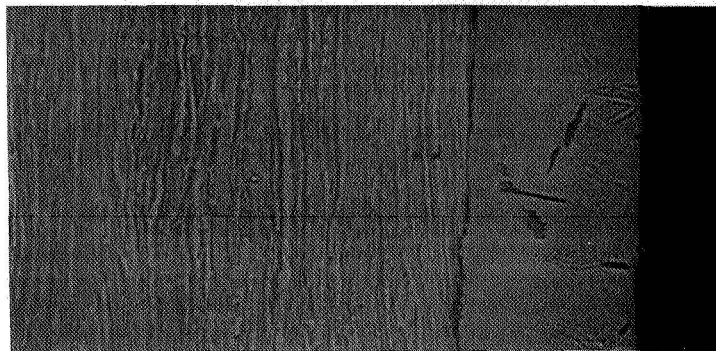
T222

Ti

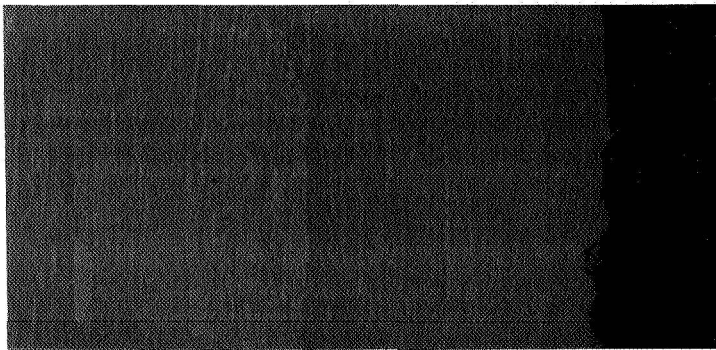
FIGURE 2. As Bonded TZM/Ti and T222/Ti: 500X Magnification



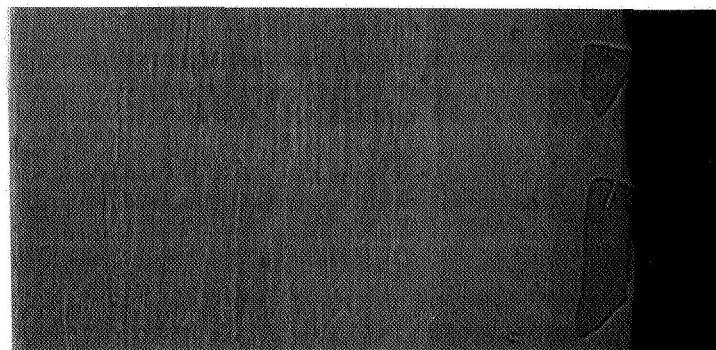
1 Hr/2000 F



1 Hr/2000 F
Plus Ti



16 Hrs/2000 F



16 Hrs/2000 F
Plus Ti

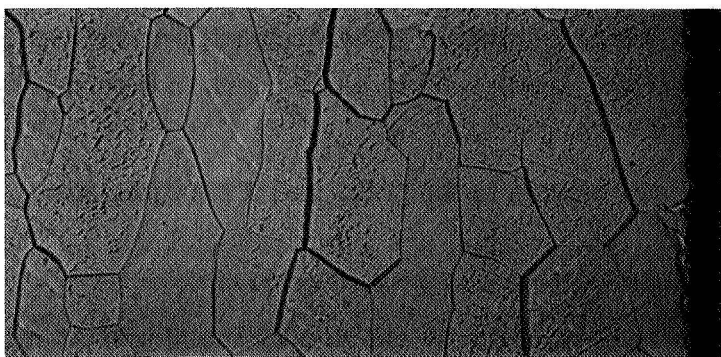
FIGURE 3. Structure of TZM Annealed at 2000 F With and Without A Titanium Sink: 500 X Magnification.



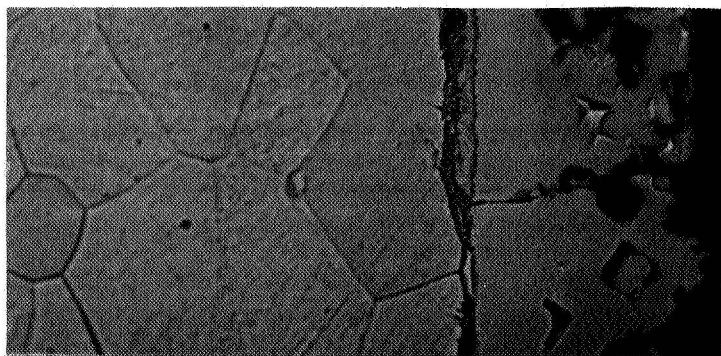
1 Hr/2500 F



1 Hr/2500 F
Plus Ti



16 Hrs/2500 F



16 Hrs/2500 F
Plus Ti

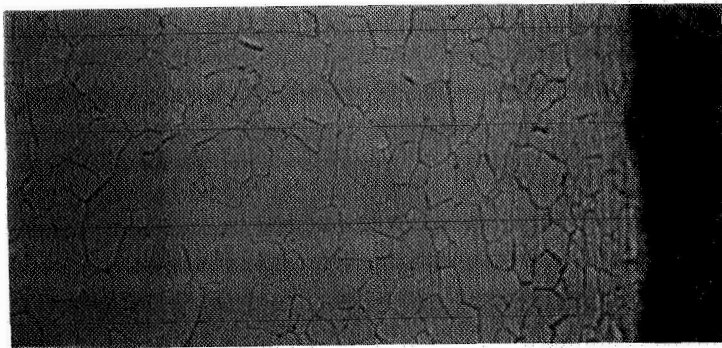
Figure 4. Structure of TZM Annealed at 2500 F With and Without A Titanium Sink: 500X Magnification

in the TZM heated with titanium for 16 hours at 2500 F are somewhat more symmetrical than those in the TZM heated without titanium. This series of tests show that titanium, a strong interstitial sink, has a relatively minor effect on the grain structure induced by the recrystallization of the as-received TZM.

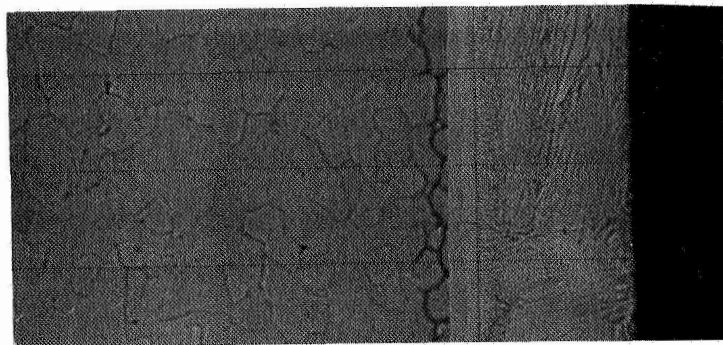
As shown in Figures 3 and 4, a second phase is formed in the titanium foil adjacent to the TZM after both the 2000 F and 2500 F heat treatments. This phase is usually found toward the outside of the titanium foils; although in some cases a phase is evident toward the center of the foils. Additional work will be required to determine if this phase is an interstitial precipitate caused by the partition of interstitials (probably carbon) from the TZM to the titanium or by some more complex reaction or contamination effect.

The structure of the as-received T222 after annealing at 2000 and 2500 F with and without titanium is shown in Figures 5 and 6. The grain structure of the T222 is relatively unchanged by these annealing treatments. The redistribution of interstitials between the alloy and the titanium (if any) is evidently insufficient to alter the grain structure of this alloy. A minor phase is again evident in the titanium after 16 hours at 2000 F and after the 2500 F heat treatments. The composition of these particles will be analyzed using the electron microprobe in future work to determine if they are an interstitial precipitate.

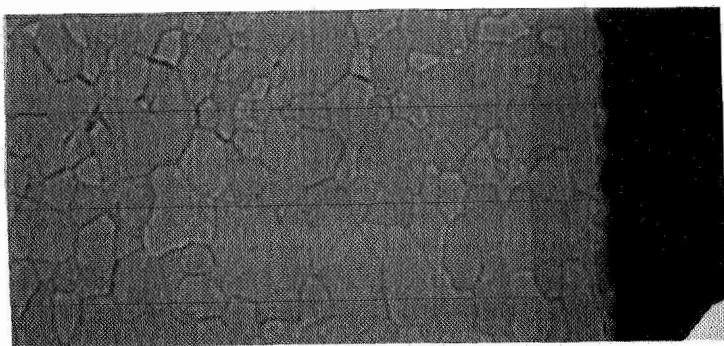
The concentrations of carbon and oxygen in the T222 and TZM after the various annealing treatments with and without titanium are compared in Table I. The analyses shown as "Plus Ti" are for portions of the heat treated Ti/alloy/Ti diffusion couples analyzed after the titanium had been removed by chemically thinning the couple to 0.026 inch. This center section which is well below the zone of titanium diffusion into the alloys was used as a measure of the effectiveness of the titanium as an interstitial sink for TZM and T222. The nitrogen concentrations were also determined but are not reported here because the concentrations were low (<10 ppm), and they did not vary significantly with heat treatments. The oxygen concentration in the TZM appears to be slightly reduced by annealing treatments, both with and without a titanium sink (i.e., from ~40 ppm to ~20 ppm). Although the scatter in analyses is relatively high in this low concentration range, the results are sufficient to show that the titanium used in this study (initial oxygen concentration of ~3000 ppm) is not a strong sink for oxygen in TZM. The carbon concentration is significantly reduced by the 2500 and 2700 F annealing treatment, both with and without titanium. In this regard, the vacuum



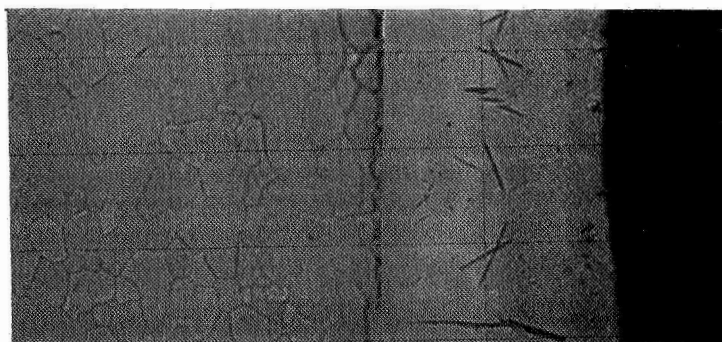
1 Hr/2000 F



1 Hr/2000 F
Plus Ti

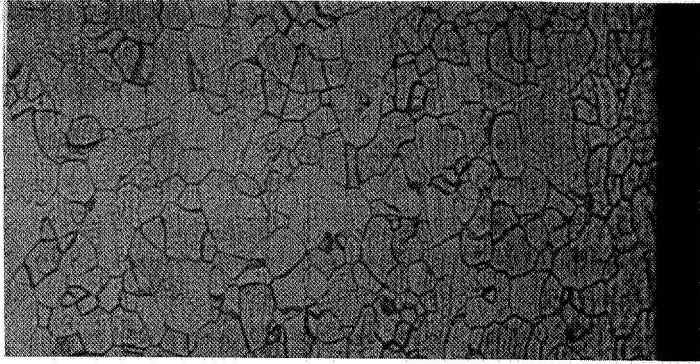


16 Hrs/2000 F

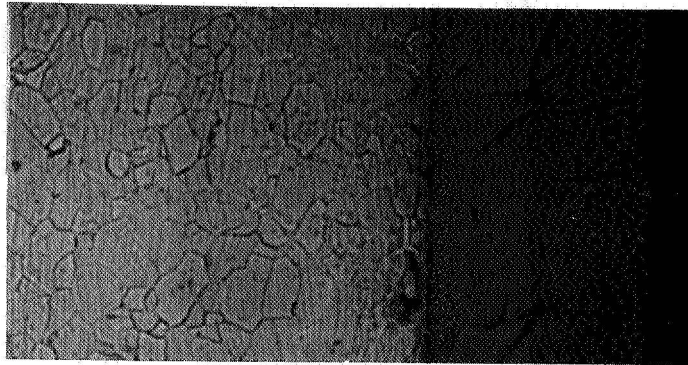


16 Hrs/2000 F
Plus Ti

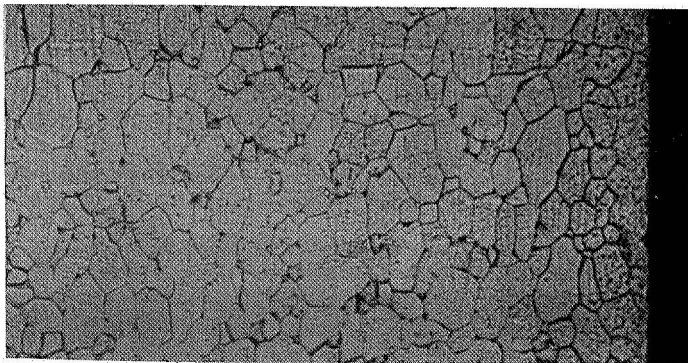
Figure 5. Structure of T222 Annealed at 2000 F With and Without a Titanium Sink: 500X Magnification



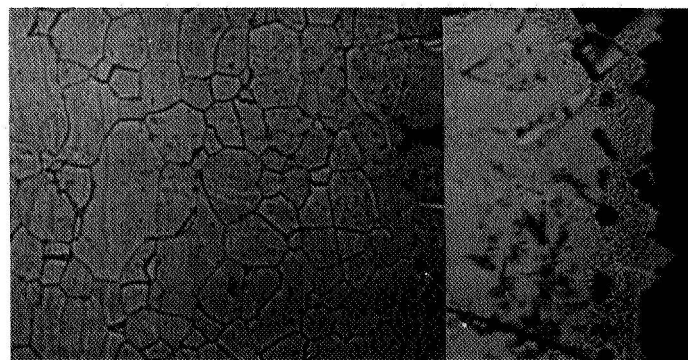
1 Hr/2500 F



1 Hr/2500 F
Plus Ti



16 Hrs/2500 F



16 Hrs/2500 F
Plus Ti

Figure 6. Structure of T222 Annealed at 2500 F With and Without Titanium Sink: 500 X Magnification.

TABLE I

CARBON AND OXYGEN CONCENTRATIONS IN TZM AND T222
ANNEALED WITH AND WITHOUT A TITANIUM SINK

Treatment	TZM		T222	
	Analyses (ppm)		Analyses (ppm)	
	O	C	O	C
As Received	40	260	21	185
1 hr/2000° F No Ti	21	220	61	150
Plus Ti	13	200	25	157
16 hrs/2000° F No Ti	26	222	22	168
Plus Ti	25	220	28	152
1 hr/2500° F No Ti	31	177	72	183
Plus Ti	22	210	22	150
16 hrs/2500° F No Ti	23	100	98	200
Plus Ti	25	90	46	197
70 hrs/2500° F No Ti	7	75	52	320
Plus Ti	19	85	—	275
16 hrs/2700° F No Ti	25	80	60	450
Plus Ti	15	70	40	175

annealing treatments alone appear to be about as effective as the annealing treatment with the titanium sink in removing interstitials from the TZM.

The effects of the annealing treatments on the carbon concentration in the TZM are shown plotted in Figure 7. The carbon concentration in the alloy is only slightly reduced at 2000 F but is reduced to less than half of its initial concentration by the longer heat treatments at 2500 F (or the 16 hour heat treatment at 2700 F).

As shown in Table I, the oxygen concentration in the T222 remains about the same or is slightly increased by the annealing treatments, both with and without titanium. The oxygen increase, however, is somewhat greater when the alloy is annealed without titanium bonded to its surfaces. Although the scatter in the analyses for oxygen in this low concentration range is relatively high ($\pm \sim 10$ ppm) the results are sufficiently accurate to show that titanium is not likely to be an effective interstitial sink for the oxygen that is in T222 at temperatures up to 2700 F.

The carbon concentration in T222 is not significantly reduced by annealing this alloy at 2000 F, 2500 F, or 2700 F, with or without titanium; rather, the results indicate that the carbon concentration may be increased by annealing. This is evident in Table I and in Figure 8 where the analyses for carbon in T222 are plotted versus annealing time at 2000 and 2500 F. These analyses are certainly sufficient to show that the carbon concentrations of T222 is not reduced by the action of titanium or of a vacuum for temperatures up to 2700 F. In addition, two heat treatments, 70 hour - 2500 F and 16 hour - 2700 F, show an increase in carbon concentration of the T222 when heated without titanium and one analysis, 16 hours - 2700 F, shows an increase in carbon concentration of the T222 when heated with titanium. This latter single analysis will be verified in subsequent work.

The equilibrium partition of interstitials between a refractory metal and a sink metal is determined by the difference between the partial molar free energy (μ) of the interstitials in the two metals. As previously discussed, (μ) can be defined by the equation

$$\mu = \Delta F^\circ + RT \ln X/S$$

where F° is the free energy of the interstitial solution for unit interstitial activity (approximated by the free energy of formation of the interstitial compound), X is the concentration of interstitial in solution in the metal, T is the temperature, and S is the saturation solubility. For two metals such as A and B where the partition of

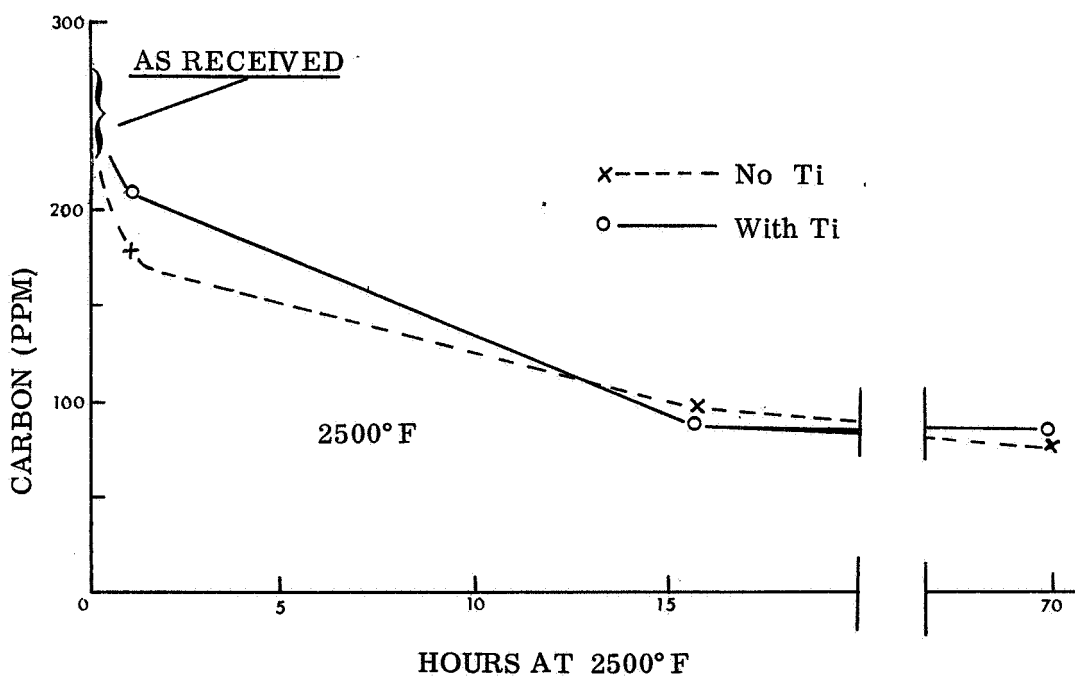
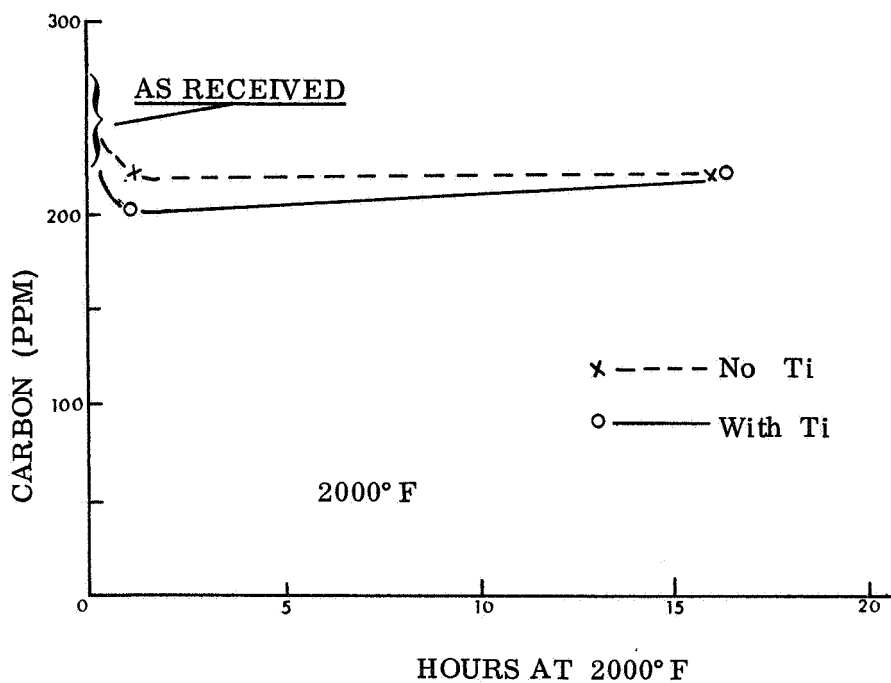


Figure 7. Carbon Concentration in TZM as a Function of Annealing Time at 2000 F and 2500 F

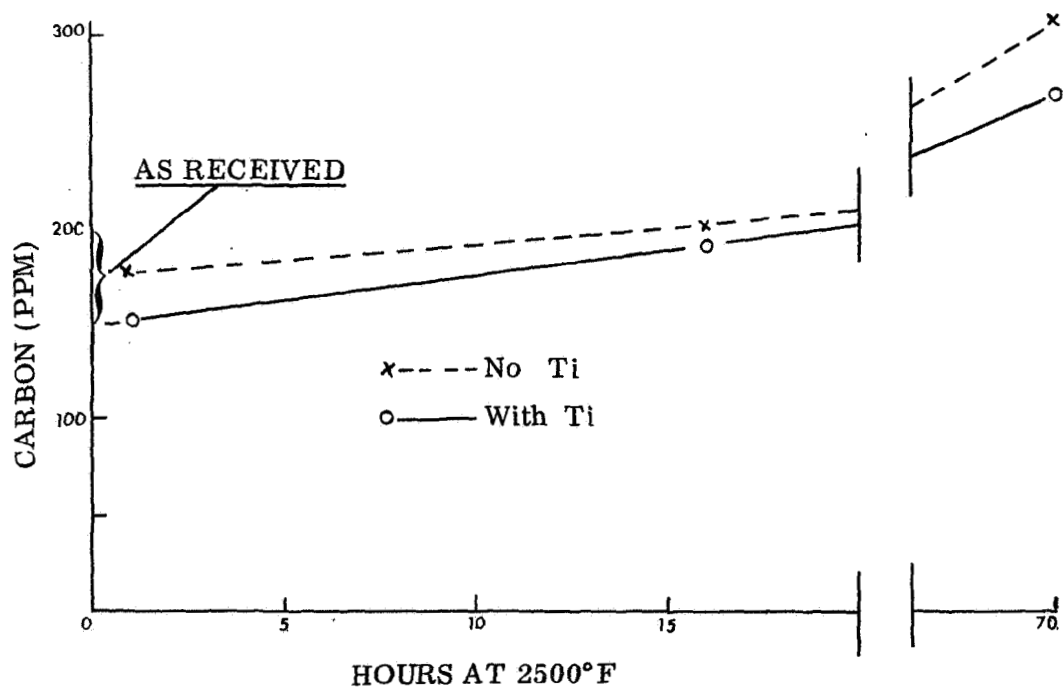
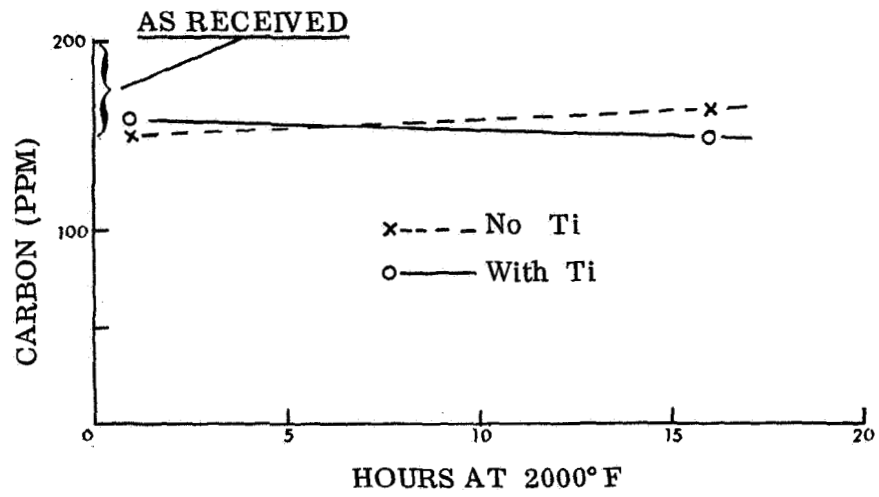


Figure 8. Carbon Concentration in T-222 as a Function of Annealing Time at 2000 F and 2500 F

interstitial, X, has reached equilibrium (i.e., $\mu_{(x \text{ in B})} = \mu_{(x \text{ in A})}$) it has been shown that

$$\Delta F_{AX} - \Delta F_{BX} = RT \ln \frac{S_A}{S_B} \cdot \frac{X_B}{X_A}$$

and

$$X_B/X_A = S_B/S_A \exp (\Delta F_{AX} - \Delta F_{BX})/RT \quad (5)$$

According to Eq. (5) the factors affecting the equilibrium partition, S_B/S_A at constant temperature are the saturation solubility ratio S_B/S_A for the interstitials in the two metals, and the difference in the free energies of formation of the interstitial compounds ($\Delta F_{AX} - \Delta F_{BX}$).

Available thermodynamic data suggest that titanium is qualitatively a strong sink for oxygen in both molybdenum and tantalum (the base metals for TZM and T222 respectively). It is difficult to predict the equilibrium partition of oxygen between titanium and these metals quantitatively because the thermodynamic data are not known with sufficient accuracy to permit an accurate calculation of X_B/X_A from Eq. (5). The equilibrium partition of oxygen between titanium and TZM, O_{Ti}/O_{TZM} , can be approximated, however, from the data in Table I and the initial oxygen concentration in the titanium. The equilibrium or final oxygen concentration in the TZM after annealing the alloy with titanium at 2500 and 2700 F appear to vary only slightly from the initial concentration (~ 40 ppm), and the initial oxygen concentration in the titanium is about 3000 ppm. Therefore, the partition ratio, O_{Ti}/O_{TZM} , is about 100/1. Similarly, if the equilibrium oxygen concentration in the T222 (Table I) is taken to be about 50 ppm, the partition ratio O_{Ti}/O_{T222} is about 60/1. Because of the low initial oxygen concentration in both TZM and T222 (20-40 ppm) and the relatively high initial concentration in the titanium (~ 3000 ppm) the initial and equilibrium partitions between the titanium and the alloys appear to be approximately equal. In this regard, the oxygen analyses in Table I suggest that the titanium may even lose some of its oxygen to the T222 in adjusting the initial to the equilibrium oxygen ratios. The results shown in Table I also suggest that the vacuum may be a sink for oxygen in TZM. Approximate calculations indicate that a vacuum of about 10^{-8} Torr is required to induce a loss of oxygen in TZM through the dissociation of MoO_2 at 2500 F. However, a loss of oxygen from the TZM to the vacuum could occur by the reduction of a more unstable

oxide phase that may form in this alloy. As shown in Table I, the T222 may gain rather than lose oxygen as a result of vacuum annealing without titanium. This can be predicted qualitatively on the basis of the greater stability of tantalum oxide relative to molybdenum oxide (eg., vacuum of 10^{-15} Torr would be required to dissociate Ta_2O_5 at 2500° C).

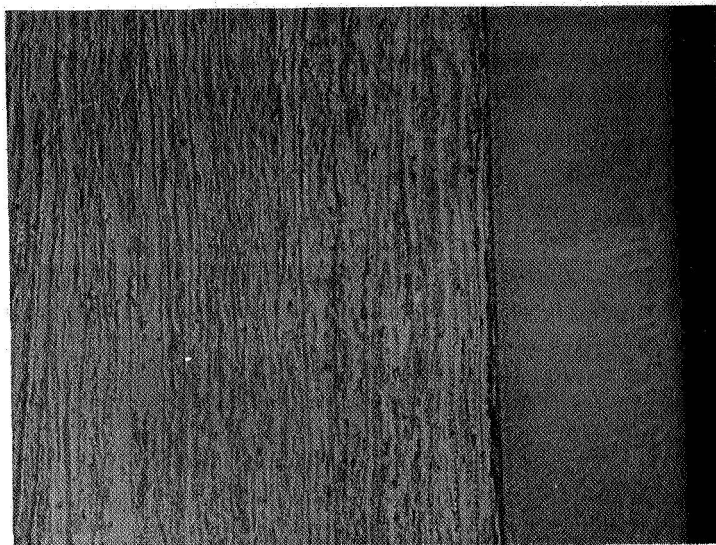
An analysis of thermodynamic data suggests that titanium will be qualitatively a strong sink for carbon in molybdenum and tantalum. However, thermodynamic data do not permit a quantitative determination of the partition of carbon between the titanium and the alloys, TZM and T222. Approximate equilibrium partitions can again be derived from the data in Table I, the initial carbon concentration and the thicknesses of the titanium foil and alloy sheet. For initial and final carbon concentrations in the TZM of 260 and 90 ppm respectively (see Table I) and 500 ppm carbon in the titanium, it can be shown that the final carbon concentration in the titanium must be about 3000 ppm. On this basis the equilibrium carbon ratio C_{Ti}/C_{TZM} is about 30/1.

With the exception of the 70 hour heat treatment at 2500 F, (this analysis is being verified), T222 shows little change in carbon concentration when heated in contact with titanium. For example, after the 16-hour heat treatments at 2500 and 2700 F with titanium, the carbon concentration in the T222 is almost the same as its initial carbon concentration. Therefore, the initial partition of carbon between T222 and titanium is almost equal to the final partition or $C_{Ti}/C_{T222} \approx 500/200 \approx 2.5/1$ (the initial concentration of carbon in the titanium foil is about 500 ppm).

2.2.2 Recrystallization Studies after Sink Annealing and Deformation.

To further investigate the effect of a titanium interstitial sink on the recrystallization kinetics of T222 and TZM the as-received specimens that had been annealed for 16 hours at 2500 F with titanium and specimens that had been annealed without titanium were cold rolled to 75 percent reduction in area and subsequently re-annealed to induce recrystallization. The recrystallization kinetics of the titanium-annealed specimens were then compared with the recrystallization kinetics for specimens that had not been annealed with titanium prior to reduction in area.

The structures of the TZM and T222 after cold rolling are shown in Figure 9, and the structures induced in the alloys by annealing the cold rolled specimens at temperatures from 2000 to 2500 F are shown in Figures 10 and 11. The structures of the alloys after cold rolling (Figure 9) are typical of highly worked metals, and the structures



1. T222

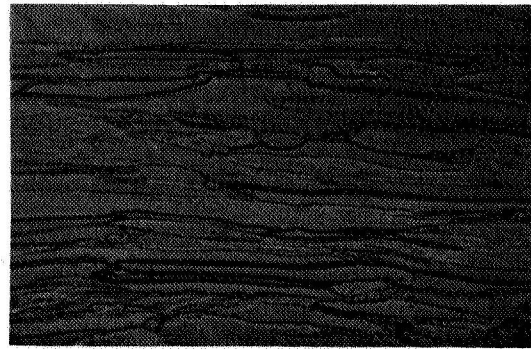
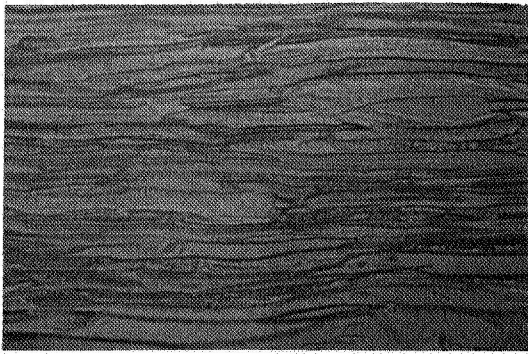


2. T222

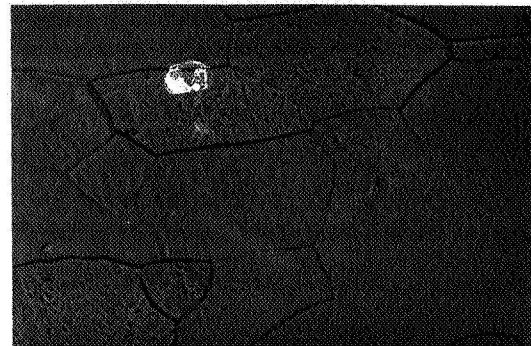
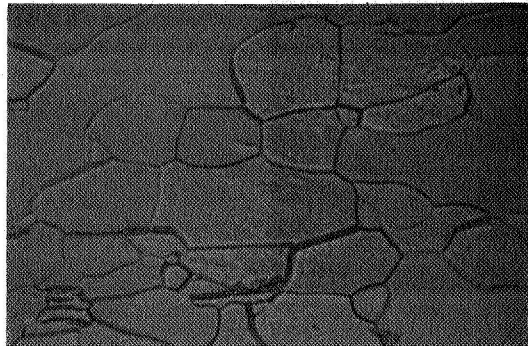
Figure 9. Structures Typical of T222 and T222 After 80 Percent Reduction in Area: 500 X Magnification.

Without Ti

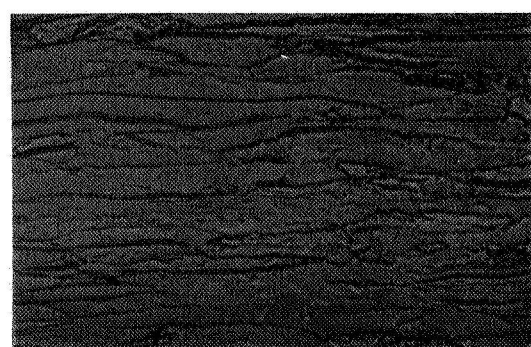
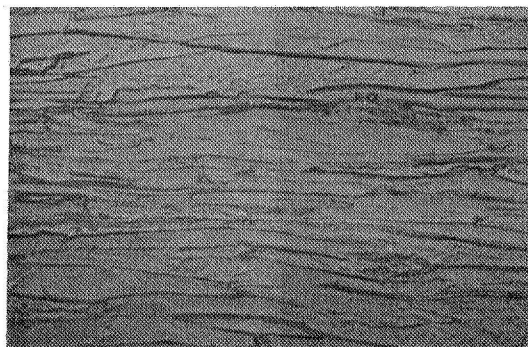
With Ti



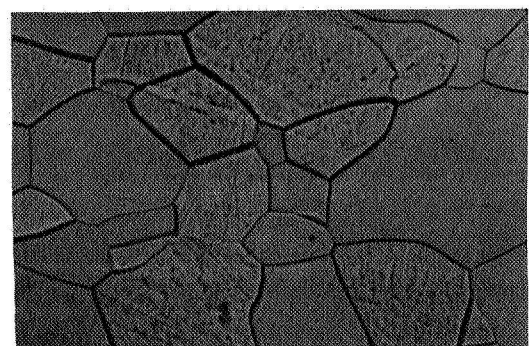
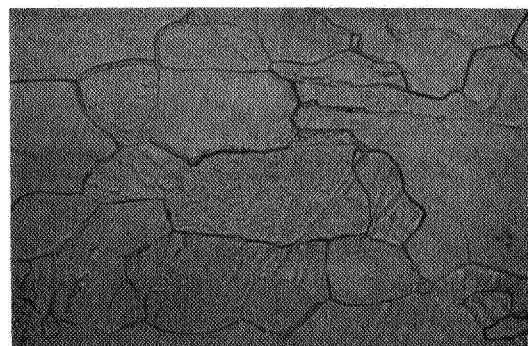
1 Hr/2000



1 Hr/2200



1 Hr/2400

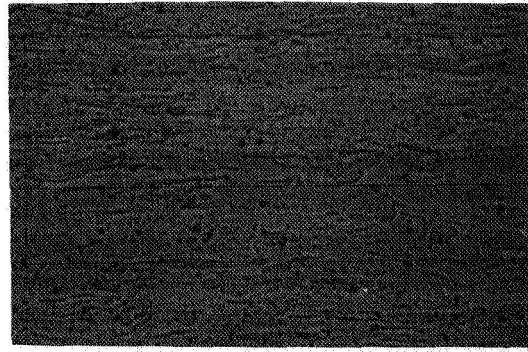


1 Hr/2500

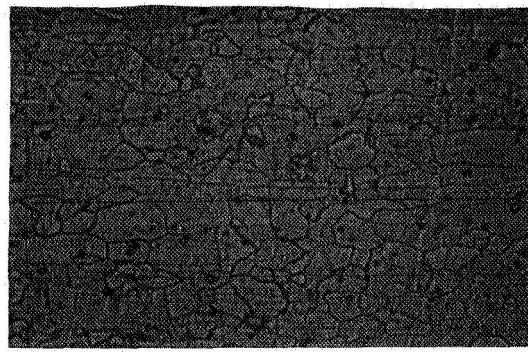
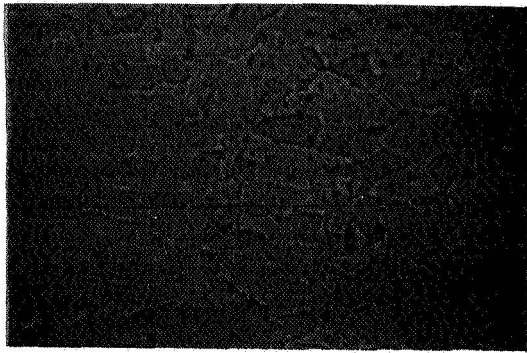
Figure 10. Structure of TZM Annealed 2500 F for 16 Hours With and Without Titanium Sink Prior to Cold Rolling and Annealing at the Temperatures Indicated: 500X Magnification.

Without Ti

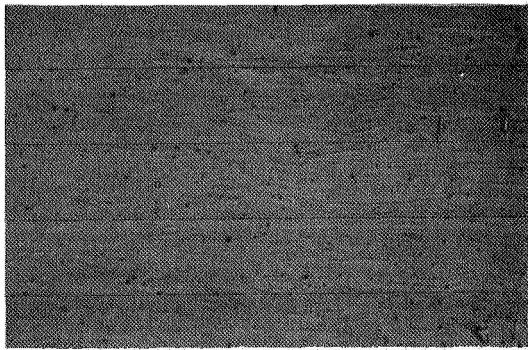
With Ti



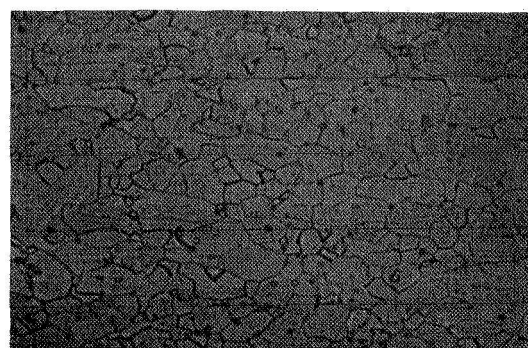
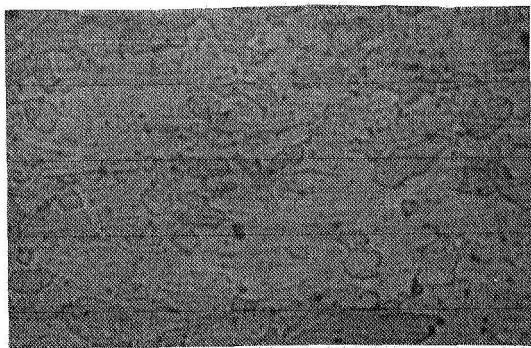
1 Hr/2000 F



1 Hr/2200 F



1 Hr/2400 F



1 Hr/2500 F

Figure 11. Structure of T222 Annealed 2500 F for 16 Hours With and Without a Titanium Sink Prior to Cold Rolling and Annealing at the Temperatures Indicated: 500 X Magnification.

shown in Figures 10 and 11 depict the various stages of recrystallization. The structures in the first column of these figures (labeled without Ti) are those for the as-received alloy annealed for 16 hours at 2500 F prior to a reduction in area of 75 percent and the annealing treatments indicated. The structures in the second column in these figures (labeled with Ti) are those for the Ti/alloy/Ti diffusion couples annealed for 16 hours at 2500 F prior to a reduction in area of 75 percent and the annealing treatments indicated. If interstitial partition to the titanium sink affects the recrystallization kinetics of these alloys relative to vacuum annealing without titanium the grain structures shown in the first columns should be different from those shown in the second columns of Figures 10 and 11. Since the structures are very similar, these results suggest that a titanium sink does not significantly affect the recrystallization kinetics of these alloys.

III. SUMMARY AND CONCLUSIONS

Studies of the creep behavior of D43 were continued to evaluate the high temperature strength properties of this dispersion strengthened alloy. The activation energy for creep of the as-received D43 was determined from about 1600 F to 3200 F. Above about 2200 F the activation energy for creep is approximately equal to that for self-diffusion and is independent of the temperature and stress. These characteristics suggest that the rate controlling creep mechanism in this temperature range involves dislocation climb probably over carbide particles. Below about 2200 F preliminary results indicate that the activation energy for creep increases to values above that for self-diffusion. This increase in the activation energy may be due to a solute-atom dislocation interaction in this temperature range.

The effects of a titanium interstitial sink on the interstitial concentration and the recrystallization behavior of TZM and T222 were studied in the temperature range 2000-2700 F. The recrystallized grain structures of these alloys did not differ significantly for specimens annealed with and without a titanium sink. The carbon concentration in the TZM was reduced to less than half of its initial concentration when the alloy was vacuum heat treated at 2500 and 2700 F. This reduction occurred when the alloy was heat treated with titanium or without titanium; thus vacuum heat treatments were about as effective as titanium in removing carbon from TZM. However, neither the vacuum heat treatments or the action of titanium were effective in removing carbon or oxygen from T222. Results thus far have shown that titanium more effectively removes carbon and oxygen from D43 than from TZM. Titanium does not appear to be an effective sink for the interstitials in T222.

IV. FUTURE WORK

Studies of the effect of an interstitial sink on the creep behavior of D43 will be continued. The effect of stress and temperature on the creep behavior of sink annealed D43 will be compared with the effects of these test variables on the as-received material. This information will be of value in predicting the creep strength of this alloy under different conditions of stress, temperature and interstitial sink environment. The dislocation particle interactions and dislocation structures in D43 after creep testing will be studied using transmission electron microscopy. This study will provide information that is necessary to further evaluate the rate controlling dislocation mechanisms in D43.

The influence of a titanium sink on the structure and interstitial concentration of TZM and T222 will be examined further to better define the effects of an interstitial sink on these alloys.

The equilibrium partition of interstitials between an interstitial sink and a refractory alloy is determined by the chemical potential of the interstitial in the two metals. A limited effort will be expended to determine the chemical potential of oxygen in columbium titanium alloys to provide more accurate data for use in predicting the partition of interstitials in refractory metal/sink metal systems.

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