TECHNICAL PROGRESS REPORT
DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

1. Internal Friction of Martensite

We have completed an investigation on the low frequency ($\sim 1\text{Hz}$) internal friction behavior of ferrous martensites which contain carbon (1) (for which we had reported preliminary conclusions earlier. (2)). This project has focused mainly on the behaviors and mechanisms of relaxation peaks observed in a variety of ferrous martensites in the temperature range of $\sim 150^\circ\text{C} - 250^\circ\text{C}$ at 1Hz . Most of the experimental work was done on Fe-Ni-C alloys with approximately 14-30 wt. pct. Ni and 0 - 0.96 wt. pct. C, and most of these alloys were "Winchell - Cohen" alloys (3) with Ni and C contents balanced to give an M_s of $\sim -35^\circ\text{C}$. The investigation has included (1) systematic experimental studies of effects of carbon and nickel contents, tempering, and cold-work on the peak height, peak temperature, and peak width; (2) a comprehensive review of the abundant literature on the structure, morphology and internal friction of martensite; (3) detailed correlations of the results of (1) and (2); and (4) the generation of qualitative and quantitative models to explain the results.

In view of the great complexity involved in understanding behavior of martensites, the results and conclusions of this study are remarkably simple and straightforward: In twinned martensites (4), a relaxation peak at $\sim 160^\circ\text{C}$ occurs and is caused by the movement of carbon atoms in concert with twinned boundaries of martensite plates. In dislocated martensites (4), a separate and unique relaxation peak occurs at $\sim 250^\circ\text{C}$ and is caused by the dragging of carbon atoms by moving dislocations which were generated by the martensite transformation (as in the Schoeck mechanism (5)). In mixed martensites containing both twinned and highly dislocated regions, both peaks are observed in proportion to the amount of each substructure present. Figure 1 and Table 1 summarize the findings.

Figure 1A and Table 1 demonstrate that the Fe-Ni-C alloys (#1) which are

characterized by a highly twinned martensite exhibit only a "160°C peak". Alloys such as Fe-0.4%C (#2) and Fe-2.4%Si -0.39%C (#4) which exhibit highly dislocated martensite morphologies exhibit only a "250°C peak" which is analogous to the cold-work peak in α -iron (ferrite). Finally, the Fe-0.8%C alloy in Figure 1A is predominately dislocated but has been refrigerated at 77°K to transform retained austenite to fresh, untempered, and (most importantly) twinned martensite. Correspondingly, both peaks are observed.

Figure 1B summarizes these results by relating the occurrence of each peak to the observed habit planes. Dislocated martensites, as typically observed in quenched alloys #2, 3 and 4 in Figure 1A, tend to have habits along the $\{111\}_\gamma \rightarrow \{225\}_\gamma$ region of the stereographic triangle. For these habits, the transformation strains are accommodated by generation of dislocations and give rise to occurrence of a 250°C (unit) dislocation-carbon interaction peak. On the other hand, martensites such as #1 which have habits in the region $\{259\}_\gamma \rightarrow \{3\ 10\ 15\}_\gamma$ can accommodate most of the transformation strain by twinning. The resulting microstructures have very small dislocation densities, hence exhibit no 250°C peak. However, the carbon atoms can relax anelastically if they can reorient to new (octahedral) sites under the influence of a moving twin interface - the predominant microstructural feature of such martensites. Finally, as before, alloys with habits in the $\{225\}_\gamma \rightarrow \{259\}_\gamma$ region exhibit both peaks because of the tendency of the microstructures to be "mixed". (Actually, even $\{111\}_\gamma \rightarrow \{225\}_\gamma$ alloys can have small amounts of twinned martensites⁽⁴⁾. However, their relatively high M_s temperatures allow auto-tempering, resulting in precipitation of Fe_3C on $\{112\}$ twin planes and "pinning" of the twins which might otherwise contribute to a 160°C peak.

Previously, we had ascribed to the 160°C peak a mechanism dealing with either atomic or dislocation motion at ϵ -carbide particles⁽²⁾. This choice was based on transmission electron microscopy studies⁽⁶⁾ that suggested that

the 160°C peak probably increased and then decreased in height with increasing temperature in the same way that ϵ would be expected to precipitate and subsequently dissolve. However, it should be noted that the Fe-Si-C alloy (#4) in Figure 1A and Table I exhibits no 160°C peak, even though ϵ is known to be stable to temperatures much above the 150°C - 250°C range of interest. Also, in some of our experiments using very fast heating rates (a few seconds) we noted that the 160°C peak in the Fe-Ni-C alloys was present at the first measurement and did not "temper in" as ϵ was precipitated. We now feel the major role of ϵ is to inhibit Fe_3C precipitation on {112} twin planes, thus stabilizing the 160°C peak until Fe_3C does precipitate.

The exact mechanism of the 160°C peak is still uncertain. Thus far, we have mainly considered a modified-Schoeck model, wherein the only difference in mechanism from that for the 250°C peak is that partial (twinning) dislocations constituting the twin interface rather than unit dislocations drag the carbon atoms⁽¹⁾. However, an equally attractive model at this point is one in which a twin interface moving under an applied stress simply reorients carbon atoms to new octahedral positions as it passes by them. This process is analogous to reorientation of interstitials in iron by magnetic domain wall motion. Whatever the detailed mechanism, we are confident that the twinned structure and morphology play the key role in the relaxation mechanism of the 160°C peak. ✓

II. Alloy Hardening and Softening in Niobium and Tantalum Base Interstitial Solid Solutions

During the past year we have continued our program designed to clarify the role of interstitial-interstitial interactions on the mechanical behavior of the Group Va refractory metals. The major portion of this work has involved collection of data on the mechanical properties of niobium doped with controlled amounts of oxygen and nitrogen or oxygen and hydrogen. A corollary study has been carried out on the system Ta-O-N to test the generality

of the results on niobium. The results of these experiments show that the alloy softening reported earlier by Ravi (7-10) is indeed due to interstitial-interstitial interactions as previously proposed. Furthermore, the alloy softening phenomenon produced solely by interstitial impurities is not unique to the niobium base alloys but is a general occurrence in at least the Group Va metals.

Figure 2 shows data taken from three sources for the resolved shear stress as a function of oxygen concentration at 113°K for Nb-0 alloys. The data of Ravi (7) show a dramatic lowering of the yield strength with small additions of oxygen. The data from Loomis and Gerber (11) and that from our current investigations show only hardening with additions of this interstitial. The high purity niobium produced by both Loomis and us is undoubtedly freer of residual interstitials than that of Ravi, as suggested by the higher yield stress of unalloyed niobium in Ravi's work. It should be noted that the proportional limit of Ravi's as-outgassed niobium is as high as the 2% plastic strain flow strength of our material. The data of Ravi show the effect of a reaction between oxygen and residual interstitials left over from the outgassing treatment, probably nitrogen or carbon.

Figure 3 shows the yield strength of niobium as a function of nitrogen concentration as determined by both Taylor and Bowen (12) and us. For nitrogen the hardening rate is higher than for oxygen and there is no softening. Ravi did not make additions of nitrogen in his study so it cannot be determined what the effect would have been on his somewhat less pure starting material. If the residual interstitial species is primarily nitrogen then only hardening would be expected from additions of nitrogen. If, however, carbon is present as a residual solute, then nitrogen additions could very well lead to alloy softening from N-C interaction, as in the case of studies on tantalum discussed later.

Figure 4 shows the effect on the yield strength of adding both nitrogen

and oxygen to high purity niobium, as well as the effect of adding each interstitial separately at 113°K. In this figure we see that oxygen or nitrogen added separately cause only alloy hardening. On the contrary, when oxygen is added to niobium containing a constant amount of nitrogen (either 100 or 300 at. ppm in this case) the yield strength decreases with oxygen additions at first and then increases at higher concentrations. The decrease in yield strength continues with oxygen additions until the oxygen content is roughly equal to the nitrogen content. This behavior demonstrates conclusively that alloy softening can occur from interstitial-interstitial interaction in intentionally produced ternary interstitial alloys, just as it can occur from unintentionally produced ternary alloys⁽⁷⁾.

We have also done experiments in which the yield stress of Nb-O-N alloys is determined as a function of nitrogen content for various constant backgrounds of intentional oxygen contamination. Figure 5 shows such a plot for oxygen concentrations of 170 and 430 at. ppm in tests made at 113°K. In this type of experiment alloy softening is present, but is not as dramatic as in Figure 4 where the nitrogen content was held constant and the oxygen content varied. Nevertheless, the curves for both oxygen contents show a definite reduction in the yield stress when compared to the curve for nitrogen. It should be noted that the data of Figure 5 have much the type of appearance as alloy softening observed in Fe-N single crystals⁽¹³⁾. The extent of alloy softening undoubtedly depends a great deal on the potency with which "background" interstitials harden.

Experiments similar to those described above were performed on the system Ta-N-O. The essential findings of this part of the study are shown in Figure 6. Although these results are cursory, several interesting features are evident. The data for the high purity Ta-O and high purity Ta-N alloys are taken from the work of Smialek⁽¹⁴⁾. The outgassing technique used in the present experiments to prepare unalloyed tantalum was intention-

ally not as efficient as that of Smialek; we did not outgas at as high a temperature. This fact is demonstrated when the yield stresses of the outgassed materials at 113°K are compared, 21.5 kg/mm² in our study as compared to 19 kg/mm² for Smialek's material. When oxygen or nitrogen is added to our "purified" tantalum there is an initial decrease in the yield stress, below that of the as outgassed material, until a minimum is reached. The softening effect is present whether the interstitial impurity added is oxygen or nitrogen. This indicates that at least part of the residual interstitials are some third species, probably carbon. If outgassed specimens are intentionally doped with nitrogen to a constant concentration and the oxygen content is varied, then the tantalum alloys show an initial decrease in yield strength followed by normal alloy hardening, as in the above experiments on niobium alloys.

The data for the tantalum alloys in Figure 6 can be compared with similar data for niobium presented in Figure 7. Figure 7 contains selected data from Figures 2-4. It shows alloy softening with the addition of a single interstitial type to niobium of relatively low purity, but only normal solution hardening from addition of oxygen or nitrogen to a high purity material. Finally, this figure indicates the softening which occurs when both oxygen and nitrogen are added to high purity niobium. The behaviors of the tantalum and niobium alloys are so similar that there is little doubt that the alloy softening is due to the same mechanism in both materials. Furthermore, since alloy softening only occurs when two interstitial species are present, either through interstitial additions or through incomplete outgassing, it is clear that association of interstitials or other similar interactions represent the major cause of this softening.

In order to determine how general the alloy softening phenomenon is in niobium base alloys we have begun a study of the system Nb-O-H. This system is different from the Nb-O-N system in that hydrogen is quite mobile even at

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temperatures as low as liquid nitrogen whereas nitrogen has very low mobility even at room temperature. Figure 8 shows our results to date for Nb-O-H alloys. These data show that alloy softening does occur from interaction between oxygen and hydrogen, but that the concentration of one or both species must be high compared to those involved for the oxygen-nitrogen alloys in order to make the effects of the interaction evident. Figure 8 shows that when hydrogen is added to the 170 at. ppm oxygen alloy there is a high degree of alloy hardening until about 200 at. ppm of hydrogen has been added. Further additions of hydrogen lead to a decrease in the yield stress with no apparent minimum in strength up to at least 550 at. ppm of hydrogen. In the 900 at. ppm oxygen alloy there is only slight hardening with the initial hydrogen addition and softening, once it begins, takes place at a faster rate and reaches a minimum at about 400 at. ppm of hydrogen. These results are consistent with those of Ravi⁽¹⁵⁾ who observed only alloy hardening in slightly impure Nb-H alloys (~ 50-70 at. ppm of O,N and C).

Summarizing, our experiments during the past year have shown that a large part of the alloy softening that occurs in the Group Va metals containing interstitial impurities can be attributed to solute association. Softening can occur either by direct addition of two interstitial species to very high purity base material or by the addition of only one interstitial species to materials of lesser, although still relatively high, initial purity. In the latter case the residual interstitials present after purification are enough to produce alloy softening when they interact with the interstitial species used as the dopant. It must also be pointed out that if the level of residual impurities is too high the softening effect will not be observed because it will be effectively masked by the hardening caused by the unassociated interstitials which are very much in the majority. The qualitative difference in behavior between the Nb-O-H and the Nb-O-N systems can be explained if the high mobility of hydrogen is taken into account. This high

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mobility necessitates a rather higher concentration of either or both species be present so that on a time averaged basis enough hydrogen and oxygen are in configurations to reduce resistance to dislocation passage through the material. Finally, we have obtained no results that suggest that mechanisms other than solute interaction (such as an interstitial-induced increase in mobile dislocation density⁽¹³⁾ or a decrease in the Peierls-Nabarro stress⁽¹⁵⁾) are effective or that they even exist.

III. Anelasticity of BCC Refractory Metals

We have been using Snoek-type anelasticity as one of several tools in attempts to characterize those bcc solid solutions which exhibit the phenomenon of alloy softening. One particular alloy system which has served as a prototype of those which exhibit alloy softening is the Ta-Re system⁽¹⁷⁾. Importantly, Smialek et al.⁽¹⁸⁾ have recently demonstrated that the alloy softening in this system results from effects of solute interaction (and possibly precipitation) between the Re and interstitials either intentionally added or present as residual impurities. Their results on Ta-Re-N alloys and earlier ones by Raffo⁽¹⁷⁾ on impure Ta-Re alloys served as the basis for our investigation of the anelasticity of these materials. Briefly, we anticipated seeing Re-N "interaction peaks"⁽¹⁹⁾ as satellite relaxations to the normal nitrogen Snoek peak in tantalum. Such results have been reported by other investigators in other refractory metals and iron alloys^(20,21).

Initial experiments reported previously⁽²⁾ had demonstrated that both the oxygen and nitrogen Snoek peaks were broadened by addition of ~ 2% Re in solid solution. In the past year, these experiments have been expanded for the Ta-Re-N system. Some alloys of tantalum with 0-4 at. pct. Re were prepared by zone melting of ~ 1/8" rod, swaging to ~ 0.8 mm wires, and outgassing under ultra-high vacuum to reduce interstitials to less than ~ 10 at. ppm of N and O. Nitrogen was readded in amounts to several hundred at. ppm with a Sievert's apparatus. These materials were tested by torsional

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pendulum techniques at ~ 0.8 Hz. Other Ta-Re-N alloys were outgassed in rod form and cut for and tested by piezoelectric resonant bar techniques at 50kHz. All techniques have been used and reported on previously (22,23).

The results on Ta-Re-N alloys are summarized in part in Figure 9, where the effect of additions of Re to Ta with a constant concentration of N is given. The nitrogen Snoek peak at $\sim 340^\circ\text{C}$ is decreased in height and broadened on the high temperature side in a regular fashion by the addition of Re until at 3.8% Re a definite hump is observed at $\sim 380^\circ\text{C}$.

Results such as these have been analyzed by computer techniques to determine if the relaxation spectra can be synthesized into component Debye-like (single relaxation) peaks. The methods for doing this have been described elsewhere (20,24). We have found that the results for all measurements made to date consistently analyze into three component relaxation peaks with values of the relaxation strength Δ and relaxation rate parameters τ_0 and ΔH that are largely insensitive to details of the computational techniques and the assumed accuracies of the raw data and input (trial) parameters. See Table II. A sample computer analysis of a measurement on a Ta-3.8% Re - 0.06% N alloy is given in Figure 10.

The three component peaks can be identified as follows:

1. Process # 1 at $\sim 340^\circ\text{C}$ at ~ 0.8 Hz is the nitrogen Snoek peak in tantalum which measures the amount of free unassociated nitrogen in solid solution.

2. Process # 2 at $\sim 380^\circ\text{C}$ varies linearly in relaxation strength as a function of the nitrogen and rhenium contents of the tantalum. Hence, it most likely involves motion of a single nitrogen atom about a nearest neighbor rhenium atom.

3. Process # 3 at $\sim 425^\circ\text{C}$ appears to vary linearly in relaxation strength with nitrogen content, but faster than linearly with rhenium content. More data will be obtained to determine the exact variation of Δ_3 with these

concentrations. Tentatively we have ascribed this peak to motion of single nitrogen atoms in a cluster with two rhenium atoms, i.e. a Re_2N complex.

The following additional experiments have been conducted or are under way:

1. Effects of thermal history. In their work on alloy softening in Ta-Re-N alloys, Smialek et al. (18) reported that the softening was pronounced only in slowly cooled specimens. In experiments on slowly cooled Ta-Re-N internal friction specimens, we noted an appreciable decrease in the overall level of damping in slow-cooled as opposed to vacuum-quenched specimens, consistent with the results of Smialek et al. Typical results are given in Figure 11. However, we have not yet determined that the decreased damping is not a result of loss of nitrogen from the specimen. These experiments will be continued later.

2. Effects of frequency (and temperature). To determine the relaxation rate parameters of the three peaks in Ta-Re-N alloys accurately and to permit detailed thermodynamic analysis, such as determination of the binding enthalpy between atoms in the small clusters, damping measurements have been made over wide frequency (and hence temperature) ranges. In the torsional pendulum range, measurements are being made at frequencies of $\sim 0.3 - 30$ Hz. These data exhibit decreased relaxation strengths of processes #2 and #3, Δ_2 and Δ_3 , with increasing frequency (temperature), whereas Δ_1 for the Snoek peak does not change or increases slightly with increased frequency. Such results are consistent with "positive" binding enthalpies, suggesting that the clusters are present in more than random numbers.

Measurements have also been made at high frequencies (~ 50 kHz) but mainly on single crystals for studies of the crystallographic orientation dependence of the Δ_i 's discussed below.

3. Orientation dependence of the relaxation strengths. The measurements at ~ 50 kHz designed for determining orientation dependence of the Δ_i 's

(hence the configurations of the clusters) have largely been abandoned in favor of low frequency (~ 30 hz) torsional and flexural excitation on ~ 0.050 " diameter single crystal wires. The former measurements have been plagued by semi-regular breakage of the ceramic adhesive bond between the piezoelectric transducer drive system and the sample in the temperature range of the interaction peaks ($\sim 730^\circ\text{C} - 900^\circ\text{C}$). Initial attempts with the latter experiments have been regularly successful.

All of the above experiments will be continued and largely completed in the next year. We anticipate having a fairly complete characterization of the damping behavior of Ta-Re-N alloys and possibly comparable results on Ta-Re-O alloys. In addition, some similar, but brief experiments will be done on Nb-Mo-O, Nb-O-N, and possible Nb-O-H alloys. These are materials for which we are presently investigating the alloy softening phenomenon.

IV. Alloy Hardening and Softening in Nb-Mo and Nb-Mo-O Alloys

In the past year, we have conducted experiments on the mechanical properties of Nb-Mo and Nb-Mo-O single crystal alloys. To summarize these results briefly, we obtain only alloy hardening (i.e. no alloy softening) when high purity Nb-Mo alloys (relative to interstitial solutes) are prepared by ultra-high vacuum outgassing and are tested at any temperature between 77°K and 450°K . This result is contrary to ones obtained by previous investigators who have not made the same efforts to purify their alloys⁽²⁵⁻²⁷⁾. When controlled additions of oxygen are made to the high purity Nb-Mo alloys or when we intentionally do not purify our alloys as completely as possible, alloy softening is observed. Thus, the overall behavior of the Nb-Mo alloys is the same as that observed in other bcc alloys for which effective arguments have been made that the alloy softening is caused by solute interactions^(7-10,18,24).

Figure 12 shows the compositional dependence of yield stress for a variety of Nb-Mo alloy single crystals tested at 77°K . Alloy softening does not occur if the alloys are relatively pure with respect to the interstitial

solutes, as in curve (A). However, softening is observed when the samples contain a higher residual interstitial concentration as in curve (B) for which the outgassing temperature is much lower. The orientation dependence of the yield stress of the Nb-Mo alloys can be noted by comparing our results with those of other investigators^(25,26). Unfortunately, some of the differences in yield stress may be due to the differences in the residual impurity contents of the alloys.

Results similar to those of Figure 12 are obtained when tests are made at other temperatures, as shown in Figure 13. At all of the temperatures examined so far we have not observed alloy softening in high purity Nb-Mo alloys. In Figure 13, we have compared our results with those of Kan⁽²⁷⁾ which show alloy softening in alloys of a different orientation. We believe the difference in the two investigations probably lies in the interstitial impurity level in the two sets of materials.

We have done other experiments in which controlled additions of oxygen were made to the high purity Nb-Mo alloys described in Figures 12 and 13. Alloy softening was observed when low oxygen concentrations were added and the alloys were tested at low temperatures. When higher oxygen concentrations were added or when tests were made at high temperatures for all concentrations, alloy hardening was observed. Results obtained for a Nb-2.7%Mo base alloy are given in Figure 14. For reference, the hardening caused by additions of Mo and O separately are also given. At 77°K and 113°K, alloy softening is observed as oxygen is added up to ~ 0.017 at. pct.; at higher concentrations the Nb-2.7%Mo alloy hardens at a rate approximately that observed in pure Nb-O alloys (with no Mo). At the higher temperatures of 190°K and 300°K, only alloy hardening was observed. Of course, when either Mo or O is added separately, only alloy hardening is observed at all temperatures and compositions.

From the results of Figure 14, one gets a feel for some of the reasons

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alloy softening is not observed at higher temperatures. Note the large differences in hardening rates of O and Mo at 113°K compared to those at 300°K. Consistent with the results presented in Figures 4, 5 and 8 on Nb-O-N and Nb-O-H alloys, to observe appreciable alloy softening it appears necessary that (1) the two obstacles in solution have widely different hardening rates and (b) the "background" solute must be present in numbers great enough that its hardening is appreciable. In Figure 8 and in Figure 14 for the results at 300°K the former condition does not hold, whereas in Figure 5 the latter condition does not hold.

The results portrayed in Figure 14 have been obtained for other Nb-Mo alloys and are given in part in Figure 15. Again alloy softening is observed in the dilute ternary Nb-Mo-O alloys but not in the Nb-O alloys (data taken from Loomis and Gerber⁽¹¹⁾). In general, the alloy softening becomes less pronounced with increased Mo or O contents. At high O contents, the hardening in all Nb-Mo-O alloys tends to parallel that for the Nb-O alloys. It will be necessary to consider particular models of solute interaction to account in detail for these and the other results presented in earlier sections.

In summary the present work shows the following results:

(1) Impure binary alloys of Nb-Mo exhibit alloy softening at low Mo concentrations and at low temperatures.

(2) High purity Nb-Mo alloys show only alloy hardening at all test temperatures.

(3) Ternary alloys of Nb-Mo-O show alloy softening at low temperatures and low oxygen concentrations.

(4) All of the results may be interpreted in terms of effects that substitutional-interstitial interactions have on localized and thermally activated point defect hardening.

V. Other Programs

Other programs are in progress but are not advanced enough to report on

in any detail at present. These include:

1. Effects of Inhomogeneous Stresses on Dislocation Damping - F. Povolo.
2. Dislocation Damping in High Purity Niobium - F. Povolo.
3. Effects of Irradiation on Point Defect Anelasticity in Austenitic Alloys - J. Slane.
4. Effect of Texture on Snoek Anelasticity - R. Krupitzer.
5. Effect of Hydrogen on the Internal Friction of Martensite - J. Robison and R. Gibala.

The main investigators of the work presented in earlier sections are:

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- III. A.A. Sagues
- IV. A.K. Vasudevan

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TABLE I

COMPARISON OF MARTENSITE HABIT AND INTERNAL FRICTION PEAKS FOR VARIOUS MARTENSITES⁽¹⁾

<u>Alloy</u>	<u>Martensite Morphology</u>	<u>Martensite Habit</u>	<u>1st Temper Precipitate*</u>	<u>160°C Peak</u>	<u>250°C Peak</u>
Fe-Ni-C	twinned	{259} _γ	ε	Yes	No
Fe-29.7% Ni-0.09 to 0.16% C	twinned	{259} _γ	ε	Yes	No
Fe-19% Ni-0.5% C	twinned	{259} _γ	ε	Yes	No
Fe-0.3 to 1.36% C ‡	dislocated/twinned	{111} _γ to {225} _γ	ε or C	Yes	Yes
Fe-1.5 Cr-0.96% C ‡	dislocated/twinned	{225} _γ	ε or C	Yes	Yes
Fe-0.12 to 0.81% C	dislocated	{111} _γ to {225} _γ	ε or C	No	Yes
Fe-0.31% C	dislocated	{111} _γ	ε or C	No	Yes
Fe-0.71% C	dislocated	{225} _γ	ε or C	No	Yes
Fe-0.75% C	dislocated	{225} _γ	ε or C	No	Yes
Fe-0.93% C	dislocated	{225} _γ	ε or C	No	Yes
Fe-2.4% Si-0.39% C	dislocated	{225} _γ	ε	No	Yes

*ε designates ε-carbide and C designates cementite.

‡ refrigerated at 77°K after transformation.

TABLE II

Analysis of Selected Anelasticity Data on Ta-Re-N Alloys into Component Relaxation Peaks (See Figures 9 and 10).

Alloy (<u>X%Re</u> + 0.06%N)	<u>Δ_1</u>	<u>Δ_2</u>	<u>Δ_3</u>	<u>Δ_2/Δ_1</u>
1.3	14.5	3.4	~ 0	0.23
2.1	10.7	4.3	~ 0	0.40
3.8	7.2	5.1	2.8	0.71

$$\Delta H_1 \approx 37400 \text{ cal/mol}$$

$$\Delta H_2 \approx 40000 \text{ cal/mol}$$

$$\Delta H_3 \approx 42600 \text{ cal/mole}$$

$$\tau_0 \approx 8 \times 10^{-15} \text{ sec}$$

(1,2,3)

Process No. 1 = Snoek Relaxation ($T_p \sim 340^\circ\text{C}$ at 0.8Hz)

Process No. 2 = Re-N Relaxation ($T_p \sim 380^\circ\text{C}$ at 0.8Hz)

Process No. 3 = Re_2N Relaxation ($T_p \sim 425^\circ\text{C}$ at 0.8Hz)
(tentative)

Figure 1. (A) Internal Friction at $\sim 1\text{Hz}$ of martensite in (1) Fe-25.5%Ni - 0.32%C, (2) Fe-0.4%C, (3) Fe-0.8%C, and (4) Fe-2.4%Si - 0.39%C alloys.
(B) Stereographic triangle showing the relationship between habit plane and the occurrence of 160 $^{\circ}\text{C}$ and 250 $^{\circ}\text{C}$ peaks in martensite.

Figure 2. Effect of oxygen content on the strength of niobium single crystals from three studies⁽⁷⁻¹¹⁾.

Figure 3. Effect of nitrogen content on the strength of niobium single crystals from two studies(11).

Figure 4. Effect of the presence of nitrogen as an intentional contaminant on the strength of niobium-oxygen single crystals.

Figure 5. Effect of the presence of oxygen as an intentional contaminant on the strength of niobium-nitrogen single crystals.

Figure 6. Effect of oxygen and nitrogen on the strength of high purity tantalum single crystals (filled symbols) (14) and of lower purity tantalum single crystals (open symbols).

Figure 7. Summary of results on the influence of remnant interstitials on the interstitial hardening of niobium single crystals.

Figure 8. Effect of the presence of oxygen as an intentional contaminant on the strength of niobium-hydrogen single crystals.

Figure 9. Effect of rhenium content on the nitrogen Snoek peak in tantalum.

Figure 10. Computer analysis of the internal friction of a Ta-3.8%Re - 0.06%N alloy into three component relaxation peaks.

Figure 11. Effect of cooling rate on nitrogen Snoek anelasticity in Ta-Re-N alloys.

Figure 9.

Figure 10.

Figure 1.

FIG. 12. Concentration dependence of the resolved shear stress of Nb-Mo alloys at 77°K.
A. Outgassed at about 2600°K. B. Outgassed at about 2000°K.

FIG. 13. Concentration dependence of resolved shear stress of Nb-Mo alloys.

FIG. 14. Concentration dependence of resolved shear stress of Nb-2.7% Mo-O alloys. For comparison, Nb-O and Nb-Mo curves are shown at 113°K and 300°K.

FIG. 15. Oxygen concentration dependence of resolved shear stress for three Nb-Mo alloys. For comparison Nb-O curve, (dotted line) is shown.

A SUMMARY OF REPORTS SUBMITTED TO
THE UNITED STATES ATOMIC ENERGY COMMISSION

Contract No. AT(11-1)-1676

Report no.

C00-1676-1	R. Gibala, F. Povolo, M.G. Ulitchny, and K.V. Ravi, Technical Progress Report, Dislocation - Solute Atom Interactions in Alloys, October 31, 1967.
C00-1676-2	R. Gibala, Comments on the Cold-Work Peak in Alpha Iron, October 31, 1967.
C00-1676-3	R. Gibala and C.A. Wert, Interstitial Alloys of BCC Metals, October 31, 1967.
C00-1676-4	R. Gibala, F. Povolo, M.G. Ulitchny, and K.V. Ravi Technical Progress Report, Dislocation - Solute Atom Interactions in Alloys, October 31, 1968.
C00-1676-5	F. Povolo, Comment on The Influence of Hydrogen Content on the Relaxation Spectrum of α Titanium, November 7, 1968.
C00-1676-6	F. Povolo and R. Gibala, A Marx Three Component Oscillator for Internal Friction Measurements at Low and High Temperatures in High Vacuum, January 8, 1969.
C00-1676-7	K.V. Ravi and R. Gibala, The Strength and Alloy Softening of BCC Metals, May 30, 1969.
C00-1676-8	R. Gibala, F. Povolo, M.G. Ulitchny, K.V. Ravi, and G.J. Klems, Technical Progress Report, Dislocation - Solute Atom Interactions In Alloys, October 31, 1969.
C00-1676-9	R. Gibala, Hydrogen-Dislocation Interaction in Iron, December 24, 1969.
C00-1676-10	K.V. Ravi and R. Gibala, The Strength of Niobium-Oxygen Solid Solutions, December 20, 1969.
C00-1676-11	K.V. Ravi and R. Gibala, Low Temperature Strengthening in Niobium-Hydrogen Single Crystals, May 27, 1970.
C00-1676-12	K.V. Ravi and R. Gibala, The Strength Differential Effect in Niobium-Oxygen Solid Solutions, September 9, 1970.
C00-1676-13	R. Gibala, M.G. Ulitchny, G.J. Klems, A.K. Vasude-

van, and A.A. Sagues, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1970.

- C00-1676-14 K.V. Ravi and R. Gibala, Dislocation Dynamics in Niobium-Oxygen Alloys, May 18, 1971.
- C00-1676-15 M.G. Ulitchny, A.A. Sagues and R. Gibala, Alloy Softening in Niobium- and Tantalum-base Solid Solutions, June 10, 1971.
- C00-1676-16 A.A. Sagues and R. Gibala, Substitutional-Interstitial Solute Interactions in Ta-Re-N Solid Solutions, July 14, 1971.
- C00-1676-17 R. Gibala, A Report on the Discussion Meeting on Defects in Refractory Metals, at Mol, Belgium on September 20-22, 1971, October 31, 1971.
- C00-1676-18 R. Gibala, M.G. Ulitchny, G.J. Klems, A. A. Sagues and A.K. Vasudevan, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1971.

AEC Contract No. AT(11-1)-1676

RECENT PUBLICATIONS

1. R. Gibala, "Comments on 'The Cold-Work Peak in Alpha Iron'", Scripta Met. 2, 13 (1968).
2. F. Povolo and R. Gibala, "A Marx Three Component Oscillator for Internal Friction Measurements at Low and High Temperatures in High Vacuum", Rev. Sci. Instr. 40, 817 (1969).
3. K.V. Ravi and R. Gibala, "The Strength and Solution Softening of BCC Metals," Scripta Met. 3, 547 (1969).
4. F. Povolo, "Comment on The Influence of Hydrogen Content on the Relaxation Spectrum of α Titanium", Scripta Met. 3, (1969).
5. R. Gibala, "Hydrogen-Dislocation Interaction In Iron", Scripta Met. 4, 77 (1970).
6. K.V. Ravi and R. Gibala, "The Strength of Niobium-Oxygen Solid Solutions", Acta Met. 18, 623 (1970).
7. K.V. Ravi and R. Gibala, "The Strength Differential Effect in Niobium-Oxygen Solid Solutions", Proceedings, Second International Conference on the Strength of Metals and Alloys, American Society for Metals, Metals Park, Ohio, 1970.
8. K.V. Ravi and R. Gibala, "Low Temperature Strengthening of Niobium-Hydrogen Single Crystals", Met. Trans. 2, 1219 (1971).
9. A.A. Sagues and R. Gibala, "Substitutional-Interstitial Solute Interactions in Ta-Re-N Solid Solutions", Scripta Met. 5, 689 (1971).
10. K.V. Ravi and R. Gibala, "Dislocation Dynamics in Niobium-Oxygen Solid Solutions", Met. Trans., in press.
11. J.R. Cost, R. Gibala, and C.A. Wert, "Interstitials Alloys of BCC Metals," Energetics of Metallurgical Phenomena, Gordon and Breach, in press.
12. M.G. Ulitchny, A.A. Sagues and R. Gibala, "Alloy Softening in Niobium- and Tantalum-base Solid Solutions," Proceedings of the Discussion Meeting on Defects in Refractory Metals, in press.
13. Eight papers in preparation or submitted.

RECENT ORAL PRESENTATIONS

1. R. Gibala, "Internal Friction of BCC Metals", Purdue University, April 1968.
2. K.V. Ravi and R. Gibala, "The Strength of Niobium-Oxygen Solid Solutions", AIME Meeting, Pittsburgh, May 1969.
3. M.G. Ulitchny and R. Gibala, "Internal Friction and Strain Aging of Austenite", AIME Meeting, Philadelphia, October 1969.
4. K.V. Ravi and R. Gibala, "Thermally Activated Deformation of Niobium and Niobium Base Interstitial Solid Solutions", AIME Meeting, Philadelphia, October 1969.
5. R. Gibala, "Strength and Structure of Solid Solutions", Cornell University, February, 1970.
6. K.V. Ravi and R. Gibala, "The Effects of Aging on the Mechanical Behavior of Niobium-Oxygen Solid Solutions", AIME Meeting, Las Vegas, May 1970.
7. R. Gibala, "Strength and Structure of Solid Solutions", Argonne National Laboratory, August 1970.
8. K.V. Ravi and R. Gibala, "The Strength Differential Effect in Niobium-Oxygen Solid Solutions," Second International Conference on the Strength of Metals and Alloys, Pacific Grove, California, August 1970.
9. F. Povolo and R. Gibala, "Low Temperature Dislocation Damping in BCC Metals," AIME Meeting, Cleveland, Ohio, October 1970.
10. R. Gibala, "Interstitial Solid Solutions", Case Western Reserve University, February, 1971.
11. R. Gibala, "The Effect of Solute and Point Defect Interactions on Mechanical Properties," AIME Meeting, Atlanta, May 1971.
12. K.V. Ravi and R. Gibala, "The Influence of Interstitials on the Dislocation Velocity Exponent in Niobium-Oxygen Solid Solutions", AIME Meeting, Atlanta, May 1971.
13. M.G. Ulitchny, A.A. Sagues and R. Gibala, "Alloy Softening in Niobium- and Tantalum-base Solid Solutions", Discussion Meeting on Defects in Refractory Metals, Mol, Belgium, September, 1971.
14. R. Gibala, "Alloy Softening in Body-Centered Cubic Materials", Cleveland Section, AIME Meeting, October, 1971.
15. T.E. Mitchell and R. Gibala, "Solution Softening in BCC Metals: The Importance of Solute Interactions", AIME Meeting, Detroit, October, 1971.
16. A.K. Vasudevan and R. Gibala, "Alloy Softening in Nb-Mo-O Single Crystals", AIME Meeting, Detroit, October, 1971.

17. M.G. Ulitchny and R. Gibala, "Mechanical Behavior of Niobium-base Interstitial Solid Solutions", AIME Meeting, Detroit, October, 1971.
18. G.J. Klems, R.E. Miner, F.A. Hultgren and R. Gibala, " Internal Friction in Twinned Fe-Ni-C Martensites", AIME Meeting, Detroit, October, 1971.

THESES

M.G. Ulitchny (M.S., 1969), "Internal Friction and Strain Aging of Austenite."

K.V. Ravi (Ph.D., 1969), "The Strength of Niobium-Oxygen Solid Solutions."

G.J. Klems (Ph.D., 1971), "Internal Friction of Ferrous Martensites".