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TECHNICAL PROGRESS REPORT
DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

CONTRACT NO. AT(11-1)-1676
BETWEEN CASE WESTERN RESERVE UNIVERSITY
AND THE U.S. ATOMIC ENERGY COMMISSION

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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

I. Solution Hardening and Softening in Niobium and Tantalum Base Interstitial Alloys

In the past year we have completed a program designed to clarify the role of interstitial-interstitial interactions on the mechanical behavior of Group Va refractory metals at low temperatures $T \lesssim 0.15 T_m$, where T_m is the melting temperature. The total program has included determination and analysis of the yield and flow stress τ of single crystals of Nb-O, Nb-N, Nb-H, Nb-O-N, Nb-O-H, Ta-O, Ta-N, and Ta-O-N solid solutions. Major experimental variables have been interstitial content C_i (~ 0 -2000 at. ppm), test temperature T (77°K-500°K), and strain rate $\dot{\gamma}$ (6×10^{-5} to $6 \times 10^{-3} \text{ sec}^{-1}$). The inclusion of the last two variables has afforded information for determining thermally activated deformation parameters, the activation enthalpy ΔH^* and activation volume v^* .⁽¹⁾

The experimental results of this study are contained in toto in the thesis of Dr. M.G. Ulitchny.⁽²⁾ Many of these results were presented previously in preliminary form in AEC Report No. C00-1676-18. Several of the final results and their interpretation have appeared in other submitted reports and subsequent publications.⁽³⁾ Complete publication of remaining results and analyses are expected within the next year.⁽⁴⁾

The general results and conclusions of this study may be summarized as the following:

- (1) Interstitial solutes (O,N,C,H) added to high purity Nb

and Ta cause temperature and strain-rate dependent alloy hardening at low temperatures. See for example Figure 1 which gives the dependence of the yield stress of Nb on oxygen concentration.

(2) These interstitial solutes added to low purity Nb and Ta or to Nb and Ta alloyed with a second interstitial solute cause temperature and strain-rate dependent alloy softening, i.e. a reduction in the yield stress with respect to additions of the "primary" interstitial solute. Compare for example Figure 2, wherein the addition of oxygen to a Nb-50 at. ppm N alloy gives alloy softening, with Figure 1.

(3) The results of Figures 1 and 2 are also reflected in systematic concentration dependences of thermally activated deformation parameters, the activation enthalpy ΔH^* and the activation volume V^* . Figures 3-7 demonstrate these for various Nb-N and Nb-N-O alloys.

(4) There is a fairly definite and large "intrinsic" ("zero"-interstitial) low temperature strength of bcc metals, as can be seen from Figure 1. It seems necessary to account for the low temperature strength of bcc materials in terms of "composite" dislocation models, i.e. ones which recognize the interaction or addition of both impurity and Peierls-type contributions. (5-7)

(5) All present theories of low temperature strengthening of bcc materials, whether based on strictly impurity models, strictly intrinsic models, or composite models, give at best only qualitative agreement with experimental results.

(6) While it is clear that alloy softening in Nb and Ta

alloys originates from effects of solute interactions on (screw) dislocation mobility, its particular origin is not yet discernable. Most of our results suggest that, while alloy softening might occur from solute interactions which reduce either the number of thermally activated obstacles to dislocation motion and their effectiveness (their tetragonality), the origin of alloy softening more probably occurs from effects which these interactions have on the distribution of the obstacles. Analyses of the type made by Forman and Makin⁽⁸⁾ on dislocation passage through random arrays of obstacles which take into account thermal activation and solute interactions seem necessary to properly analyze alloy softening.

II. Substitutional Solute Hardening and Softening in Niobium-Base Alloys

In the past two years, we have conducted experiments on the mechanical properties of high purity (with respect to the residual interstitials) Nb-Mo and Nb-Mo-O alloys.⁽⁹⁾ It was found that the binary alloys showed alloy hardening in the temperature range 77°K-300°K, whereas the ternary alloys exhibited alloy softening at the lower temperatures in this range and at low oxygen concentrations. The results showed that the softening is caused by solute interactions in the same manner as described in section I.

In order to verify the generality of these results on the alloy softening phenomenon to include other Nb-base substitutional systems, we have recently conducted experiments on Nb-W, Nb-W-O and Nb-Re-Mo alloys. Figure 8 shows compositional dependence of

yield stress for Nb-W crystals tested at 77°K-300°K. Alloy softening is not exhibited at both test temperatures. For comparison the results of Koss⁽¹⁰⁾ are plotted which show alloy softening at 77°K. The difference in the curves lies in the interstitial impurity level and orientation dependence of the flow stress in these alloys.

Controlled additions of oxygen were made to the high purity Nb-W alloys, as described in Figure 9. Alloy softening is observed at all tungsten compositions at low temperatures. At high oxygen concentrations the hardening in all Nb-W-O alloys tends to parallel to that of Nb-O alloys (Nb-O data taken from Ulitchny⁽²⁾). At 300°K, alloy hardening was observed in all Nb-W-O alloys. The results of Nb-W-O alloys are very similar to those of Nb-Mo-O alloys. There is little doubt that the softening in both systems is due to the same mechanism and that they arise from substitutional-interstitial type of interactions.

Further experiments to demonstrate alloy softening in ternary substitutional solid solutions were conducted using Nb-Re-Mo alloys tested at 77°K. They show results similar to others from our studies. In Figure 10, one may note that the softening was observed in the as-grown, the outgassed, and the slow-cooled conditions in ternary alloys, but never in the binary alloys. The results are consistent with all the previous results on Nb-base substitutional, interstitial, substitutional-interstitial, and interstitial-interstitial alloys.

We summarize the present work as follows: in order to observe

alloy softening it is necessary that two obstacles be in solution (be they substitutional + substitutional, substitutional + interstitial, or interstitial + interstitial) and have widely different rates of hardening. Moreover, the 'background solute' must be present in numbers great enough that its hardening is appreciable.

III. Substitutional-Interstitial Solute Atom Interactions in Tantalum-Base Alloys

We have completed a study on the use of Snoek-type anelasticity to characterize bcc solid solutions which exhibit the phenomenon of alloy softening.⁽¹¹⁾ A particular alloy system for which a large amount of data has been obtained is Ta-Re to which either O or N have been added in controlled amounts. Preliminary results have been presented previously^(9,12) and were shown to correlate well with existing data on mechanical behavior of these alloys at low temperatures.^(13,14) In particular, interstitially impure Ta-Re alloys or high purity alloys to which interstitial nitrogen is intentionally added exhibit alloy softening; correspondingly the addition of Re to Ta-N alloys broadens the Snoek relaxation and reduces the magnitude of the maximum damping, as often observed in alloy systems in which interstitial solutes undergo association with substitutional solutes. We were also able to show that the relaxation spectra could be decomposed into three individual relaxation peaks: (a) the N Snoek peak, (b) a ReN pair peak, and (c) possibly a Re₂N "cluster" peak.

In the last year, we were able to determine the detailed in-

fluence of substitutional concentration (e.g. Figure 11), of interstitial concentration (e.g. Figure 12), of temperature (e.g. Figure 13), and of crystallographic orientation (e.g. Figure 14 and 15) on the relaxation strength and rate parameters of the Snoek-type spectra in both Ta-Re-N and Ta-Re-O alloys. From these results, we have been able to clearly identify ReN and Re₂N relaxation processes in addition to the Snoek peak and to determine (a) the binding energies and structure of the ReN pairs and Re₂N clusters and (b) the damping capacity of N atoms in these configurations. Results on Ta-Re-O alloys are still being analyzed into component relaxation peaks but promise to give the same type of detailed information. Early analyses suggest existence of Re_xO complexes in the relaxation spectrum.

The binding energies for the ReN pairs was found to be ~ 3000 cal/mole and for the Re₂N clusters ~ 3600 cal/mole. Such results were obtained from the temperature dependences of the relaxation strengths. These defects are definitely present in more than random numbers.

The orientation dependence studies, like those given in Figures 14 and 15, imply defects with tetragonal <100> or orthorhombic <100> symmetries. For example in Figure 16a Da and DC-type pairs are the most likely ReN defect. Defects DE and DB can be ruled out in principle, although in practice very precise experimental results may be needed to distinguish their occurrence from DA and DC defects. In Figure 16b, we show the defects ABC and ABD which have symmetries consistent with the experimental

results for the Re_2N relaxation process. These defects can maintain their identity during relaxation by rotation of the N atom about the substitutional solute pair AB. The defect AHB is a possible relaxator if it can dissociate and transform to ABC under stress. It appears that defect ABC is the most likely defect to be responsible for the Re_2N process. Its occurrence is consistent with the strong likelihood that DA and DC are responsible for the Re_2N process.

From the concentration dependences of the relaxation processes, we were able to determine that the damping capacities per N atom contributing to the N Snoek peak, the ReN peak and the Re_2N peak were approximately the same. Evidently, changes in "effectiveness"* of N as an obstacle to dislocation motion when it is associated with Re atoms is not an important factor in causing alloy softening.

IV. Other Programs

Several other programs have made significant advances in the past year, but either are not complete enough to report on in any detail or have been reported on earlier.

A. Carbon-Vacancy Interactions in Austenitic Steels

This program represents a follow-up investigation of previous work⁽¹⁵⁾ on internal friction, strain aging and self-diffusion in austenites that suggested strong carbon-vacancy binding in these materials. Two related projects have been completed but are not yet prepared in final written form: (1) the

* Effectiveness should be considered synonymous with tetragonality.

effect of electron irradiation on Snoek-type carbon relaxation peaks ("bound-pair" peaks, bpp) in austenites, and (2) the influence of interstitial solute-vacancy binding on self-diffusion in fcc materials.

The first project is an experimental one and has shown that in Fe-Ni-C austenites 2 mev electron irradiation produces vacancies which enhance the bpp caused by the presence of carbon. This represents good evidence of carbon-vacancy binding, as discussed previously. (15)

The second project is a theoretical one and has shown that enhanced self diffusivity observed by increasing carbon content in austenites (16) is consistent with a carbon-vacancy binding energy of ~ 9000 cal/mole. This result could explain the large sensitivity of austenitic materials to void formation and swelling by irradiation.

B. Effect of Texture on Snoek Anelasticity

An attempt has been made to determine and account for the extent of texture effects on Snoek anelasticity in bcc materials. The program has consisted of detailed x-ray texture analysis of low carbon steels on which internal friction measurements have been made as a function of angle to the deformation (e.g. rolling) direction. Expected averaged internal friction has been computed from theoretical analyses (17) and compared with experimental results. We find that for many common steel products texture appears not to be an important detriment to use of internal friction in determining carbon concentrations in solution.

C. Effects of Inhomogeneous Stresses on Dislocation Damping

This theoretical work has been completed and has been summarized in AEC Report No. C00-1676-21. It is now being applied to a large amount of experimental data we have on niobium and niobium alloys.

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Figure 1. Two percent resolved shear stress as a function of oxygen content at all test temperatures.

Figure 2. Two percent resolved shear stress as a function of oxygen content for (Nb+50 N)-O alloys at all test temperatures.

Figure 3. Activation enthalpy as a function of effective stress for purified niobium and (Nb)-N alloys.

Figure 4.. Activation volume normalized by the Burgers' vector cubed as a function of effective stress for purified niobium and (Nb)-N alloys.

Figure 5. Activation enthalpy as a function of effective stress for (Nb+170.0)-N alloys.

Figure 6.. Activation volume normalized by the Burgers' vector cubed as a function of effective stress for (Nb+170 O)-N alloys.

Figure 7. Activation volume normalized by the Burgers' vector cubed as a function of effective stress for (Nb+50 N)-O alloys.

Figure 8. Effect of W concentration on the yield stress of high purity (present work) and low purity (Koss) niobium.

Figure 9. Effect of O concentration on the yield stress of several Nb-W alloys.

Figure 10. Effect of solute concentration on the yield stress of niobium-base binary and ternary alloys containing Re and/or Mo.

Ta-Re-O

Figure 11. Normalized height spectra for Ta-Re-O alloys with increasing Re contents.

Figure 12. Internal friction for Ta-3.8% Re-N alloys containing different amounts of nitrogen. The middle curve corresponds to 600ppm N

Figure 13. Frequency of oscillation versus inverse peak temperature for various Ta-Re-N alloys containing 600 at. ppm N

Figure 14. Orientation dependence for Ta-3.8 at % Re-600 at ppm N alloys ($f \sim 2$ Hz).

Figure 15. Orientation dependence in Ta-Re-O system using longitudinal mode oscillations.

Figure 16a. ReN cluster models

Figure 16b. Re_2N cluster models.

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

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.
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- C00-1676-13 R. Gibala, M.G. Ulitchny, G.J. Klems, A.K. Vasudevan, and A.A. Sagues, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1970.
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- C00-1676-16 A.A. Sagues and R. Gibala, Substitutional-Interstitial Solute Interactions in Ta-Re-N Solid Solutions, July 14, 1971.
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- C00-1676-20 M.G. Ulitchny and R. Gibala, Internal Friction and Strain Aging of Austenite, April 10, 1972.
- C00-1676-21 F. Povolo and R. Gibala, An Analysis of Amplitude Dependent Internal Friction and Modulus Defect of Materials Subjected to Inhomogeneous Stresses, June 19, 1972.
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- C00-1676-23 R. Gibala, A.K. Vasudevan, J.A. Slane, R.P. Krupitzer, Technical Progress Report, Dislocation-Solute Atom Interactions in Alloys, October 31, 1972.

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13. K.V. Ravi and R. Gibala, "Dislocation Dynamics in Niobium-Oxygen Solid Solutions, Metall. Trans., in press.
14. M.G. Ulitchny and R. Gibala, "Internal Friction and Strain Aging of Austenitic Alloys", Metall. Trans., in press.
15. J.R. Cost, R. Gibala, and C.A. Wert, "Interstitials Alloys of BCC Metals", Energetics of Metallurgical Phenomena, Gordon and Breach, in press.
16. Thirteen other papers in preparation or submitted.

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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.
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17. M.G. Ulitchny and R. Gibala, "Mechanical Behavior of Niobium-base Interstitial Solid Solutions", AIME Meeting, Detroit, October 1971.
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19. R. Gibala, "Point Defects in Refractory Metals", Case Western Reserve University, October 1971.
20. M.G. Ulitchny and R. Gibala, "Internal Friction and Strain Aging Behavior of Ferrous Austenites", AIME Meeting, Boston, May 1972.
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22. R.E. Miner, F. Hultgren and R. Gibala, "An Analysis of the Cold-Work Internal Friction Peak in Iron", AIME Meeting, Boston, May 1972.
23. M.G. Ulitchny and R. Gibala, "Solute Interactions Involving Hydrogen and Their Influence on the Mechanical Behavior of Niobium Single Crystals", International Congress on Hydrogen in Metals, Paris, France, May 1972.
24. R. Gibala, "The Strength Differential Effect in Niobium-Oxygen Solid Solutions", ONR Workshop on the Strength Differential Effect in Metals and Polymers, Boston, June 1972.
25. R. Gibala, "The Strength and Structure of Solid Solutions", General Electric Research and Development Laboratory, Schenectady, N.Y., November 1972.
26. R. Gibala, "The Strength and Structure of Solid Solutions", University of Illinois, Urbana, November 1972.

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".

M.G. Ulitchny (Ph.D., 1972), "Mechanical Behavior of Interstitial Solid Solutions of Niobium".

A.A. Sagues (Ph.D., 1972), "Substitutional-Interstitial Solute Atom Interactions in Ta-Re-N and Ta-Re-O Solid Solutions".

By the end of the present contract period, the following additional theses will be completed:

A.K. Vasudevan (Ph.D.)
J.A. Slane (M.S.)
R.P. Krupitzer (M.S.)