MASTER

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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

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DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

I. Interaction of Interstitial Solutes with Defects in Austenitic Alloys.

One aspect of the research program has been the investigation of internal friction behavior of face-centered cubic materials which contain interstitial solutes for the existence of a cold-work peak (hereafter abbreviated cwp) of the type generally observed in bcc materials.⁽¹⁾ These peaks are associated with a dislocation-interstitial interaction process, and a majority of investigators have concluded that the theory of Schoeck⁽²⁾ or modifications of it best describes the properties of the cwp. The purpose of this part of the program was to gain insight into the understanding of dislocation-interstitial interaction and the strain-aging behavior of fcc interstitial solid solutions. Also, the program was to allow investigation of these phenomena in fcc and bcc materials from a viewpoint different from that of only examining mechanical behavior.⁽³⁾

The experimental details of this work and the results and conclusions have been presented in previous reports^(4,5) and are being prepared for publication. Briefly, we have investigated the anelastic behavior of Fe-Ni-C and Fe-Cr-Ni-C austenites in the temperature range 25°C to ~400°C by torsional pendulum techniques. We have not been able to discern a cwp peak in these strain-aged austenites; at the same time the "bound-pair" carbon peak (bpp) observed in these alloys⁽⁶⁾ is enhanced by increased quenching temperature, quenching rate, deformation, and electron irradiation (Fig. 1).

These results led us to interpret the bpp in terms of a stress induced carbon-vacancy reorientation process, as did Wu and Wang⁽⁶⁾ in their earlier investigation. It was also possible to correlate closely the existence of the bpp in the deformed Fe-Ni-C alloys with the occurrence of strain aging.

While these results are internally self-consistent and correlate well with mechanical behavior studies, there are noteworthy alternative interpretations. The most important of these is the contention that the peak mechanism involves stress-induced reorientation of carbon in the substitutional alloy rather than a carbon-vacancy complex.⁽⁸⁾ Several factors dictate consideration of this mechanism:

1. The effect of quenching temperature on the bpp (Fig 1A) has not been observed by some investigators⁽⁸⁾.
2. The effect of quenching rate (Fig. 1B) could be a result of carbide precipitation. (We have noted precipitation in furnace-cooled specimens like that in Fig. 1B)
3. The enhancement of the bpp by deformation (Fig. 1C) is more difficult to explain, but could be an effect of the deformation texturing.⁽⁹⁾ Some investigators have suggested that the enhancement is caused by a closely overlapping cwp.
4. The effect of irradiation (neutrons, however) also has not been observed by other investigators.⁽⁹⁾
5. In some austenites (e.g. nickel-rich Fe-Ni-C alloys⁽⁸⁾), the peak heights correlate closely with both substitutional and interstitial content.

6. The carbon-vacancy model necessarily infers a high dipolar strain and binding energy of the defect causing the anelasticity. Neither is substantiated by other types of experiments, except perhaps for mechanical behavior studies. (7)

We are examining these problems in the write-up of our research and are attempting to clarify the situation in the follow-up experiments submitted in the renewal proposal.

II. Internal Friction of Martensite.

In studies that are complementary to those described above, we have investigated the internal friction of Fe-Ni-C martensites in alloys with approximately 14-30 at. pct. Ni and 0-4.5 at. pct. C in the temperature range 20-500°C. The research has mainly focused on the mechanism of an internal friction peak observed in a variety of ferrous martensites in the temperature range 150°C - 250°C at frequencies of 1 hz. Experimental procedures and preliminary results were presented previously. (5) These studies have included systematic investigations of effects of carbon and nickel contents, tempering, and cold work, as well as a comprehensive review of the literature on the structure and internal friction of ferrous martensites.

This work is presently being written up by G. J. Klems as part of his thesis research. Some of the conclusions reached are the following:

1. The "martensite peak" observed at $\sim 160^\circ\text{C}$ in Fe-Ni-C martensites (Fig. 2) is not a Snoek-like peak in the body-centered-tetragonal structure as suggested by Kê and Ma. (10) The peak is too broad and is characterized by a frequency factor ($\sim 10^{12-1}$ sec) which

is approximately two orders of magnitude too small for anelasticity caused by simple atom rearrangements.

2. The peak is not a Schoeck-type ⁽²⁾ cold-work peak.

Tempering would decrease the dislocation loop length, hence decrease the relaxation time τ_r and peak temperature T_p . The opposite effects on τ_r and T_p are observed experimentally (Fig. 3). In addition, cold work is observed to decrease the peak height, whereas again the opposite effect occurs for the cwp. Finally, the activation energy (~ 20 - 21 kcal/mole) is notably lower than expected for a cwp.

3. The martensite peak at $\sim 160^\circ\text{C}$ in Fe-Ni-C alloys is a different peak than the "220°C peak" observed in Fe-C martensitic alloys. ⁽¹¹⁾ The two peaks exhibit widely different responses to tempering. For example, the Fe-Ni-C peak disappears rapidly with tempering at temperatures just above the peak ($\sim 300^\circ\text{C}$), whereas the Fe-C peak is stable up to temperatures at which complete recrystallization occurs ($\sim 600^\circ\text{C}$). The Fe-Ni-C peak heights are typically a factor of two or more higher than Fe-C peak heights, and at constant frequency the temperature ranges in which they occur are distinctly different (150 - 170°C vs 200 - 250°C). The responses to cold work also appear to be different.

4. The mechanism of the Fe-Ni-C peak appears to be one associated with atomic and/or dislocation motion near ϵ -carbide precipitates. Transmission electron microscopy has demonstrated that ϵ -carbide forms rapidly during tempering of Fe-Ni-C martensites at temperatures just above ambient and begins to dissolve slowly in the temperature range near the peak temperature. ⁽¹²⁾ Thus there is a good correlation between the appearance and disappearance of

the peak and the precipitation and dissolution of ϵ . However we have not yet been able to distinguish whether dislocations are associated with the process (as in the model of Mura et al.⁽¹³⁾) or simply atom motion at the matrix- ϵ interface is involved (similar to the precipitation models of Nowick⁽¹⁴⁾ and Schoeck⁽¹⁵⁾).

III. Dislocation-Solute Atom Interaction in BCC Metals.

Work has been continuing in an attempt to understand more clearly dislocation-solute atom interactions and the mobility of dislocations in bcc materials from measurements of dislocation damping, primarily the amplitude-dependent (hysteretic) damping, δ_H . We have concluded the following from work done in previous years⁽⁵⁾.

1. Use of δ_H to determine dislocation-solute interaction energies E_B is done most successfully from analysis of thermal unpinning studies at low temperatures, where the solute atoms are immobile. At high temperatures, the manifold problems of atmosphere equilibration, solute diffusivity, dislocation pipe diffusion and contamination make the experiments and their interpretations difficult.

2. Unless measurements are made near 0°K, only theories of δ_H which incorporate thermal unpinning can be used effectively to determine E_B . Thus, for example, the classical Granato-Lücke theory⁽¹⁶⁾ for δ_H which considers only mechanical unpinning is invalid.

3. To compare theory and experiment, it is imperative either to correct experimental data for the fact that they are always obtained on specimens subjected to non-homogeneous stresses⁽¹⁷⁾ or to incorporate such corrections into theoretical expressions for δ_H .⁽¹⁶⁾ We have programmed our data on computer to make the former correction.

4. In niobium single crystals prepared by high vacuum outgassing (room temperature/ $\sim 9^\circ\text{K}$ resistivity ratios of ~ 1000), the activation energy Q for thermal unpinning* and the average value of E_B for residual solutes which cause the low temperature pinning have values in the range ~ 0.2 - 0.3 ev. These values are approximately that for the activation energy for the α dislocation relaxation peak, i.e. 0.25 ev. In addition, it appears even from casual examination of experimental data (e.g. Fig. 4) that the α peak (at $\sim 240^\circ\text{C}$ in Fig. 4) represents the major precursor event to large scale amplitude-dependent damping in niobium. Thus we believe that the α peak also has its origin in thermally activated depinning and that the only difference between α peak damping and δ_H is in the roles played by internal and external stresses in effecting the depinning.⁽⁵⁾ These results are being prepared for publication.

Recently, we have begun to examine other internal friction methods for determining binding energies. Figs. 5a and 5b illustrate a type of analysis that makes use of Snoek peaks, amplitude-independent damping δ_I , resistivity ρ , and transmission electron microscopy to determine binding energies for various interstitials to dislocations. E_B is determined from knowledge of (1) how the interstitials (oxygen

*Strictly, Q is stress-dependent. We refer here to the limiting value of Q as $\delta_H \rightarrow \delta_I$, where δ_I is the amplitude-independent damping.

and nitrogen in the tantalum shown in Fig. 5) separately and collectively affect δ_I and ρ , (2) the magnitudes of the Snoek peaks, and (3) the dislocation density and type of substructure. The procedure is outlined in Fig. 5b. Applying this approach to the results of Fig. 5a (but with only order of magnitude knowledge of the dislocation density), we obtain an averaged E_B for oxygen and nitrogen of ~ 0.7 ev. We hope to be able to determine separate binding energies for oxygen and nitrogen and possibly to incorporate use of the cold work peaks as another tool to determine the concentration of interstitials at dislocations⁽¹⁸⁾ and the number of dislocation sites available to interstitial occupancy.⁽¹¹⁾

IV. Alloy Hardening and Softening in BCC Refractory Metal Alloys.

This part of the research program has dealt directly with the strengthening of bcc metals by alloying, both with interstitial and substitutional solutes. The initial goal of this project was to investigate the role of interstitial partitioning in the strengthening of bcc alloys. We had hoped, on the basis of many kinds of physical evidence⁽²⁰⁾ that intentional interstitial partitioning between solid solution, clusters, pre-precipitates, and equilibrium precipitates might effect various types of dispersed barrier hardening processes in alloys that are (or would be) useful technologically, particularly at elevated temperatures. Thus, work focused on the refractory metals and mainly on niobium.

In dealing with this problem, it was important to know and understand the baseline hardening of the bcc metals by interstitial and substitutional solutes. However, examination of the literature showed this field to be hopelessly confused. For example, a great

controversy centered around whether the high strength of bcc metals at low temperatures was caused by an intrinsic lattice resistance (21,22) or by residual interstitial solutes. In addition, both substitutional and interstitial solutes had been observed to cause alloy softening, rather than hardening, with initial additions up to a few at. pct. (23) Other features such as multiplicity of slip planes, deviations from Schmid's law, complex (screw) dislocation core geometries, and others further complicated this important field. (24) Thus, we undertook a project to examine many of these problems.

In the past three years, our research has helped clarify many of these problem areas. Much of the initial work has been presented in previous reports, (5) technical talks, and first publications (see enclosed listings). Briefly, we have shown that (25,26)

1. interstitial solute hardening is thermally activated in bcc metals;
2. interstitial hardening represents a major part of the high strength of bcc metals at low temperatures; and
3. in virtually all cases, alloy softening in bcc metals results from solute-association processes.

These conclusions were based on (1) initial studies of the effects of purification on the mechanical behavior of niobium, (2) baseline results on the strength of niobium-oxygen solid solutions (which exhibited alloy softening), and (3) careful examination of the extensive amount of work done by other investigators in this field.

(5,25)
 With the above facts (and others) behind us, work has progressed in three directions in the past year.

(1) Some of the work done in previous years has been analyzed in greater detail. For example, we have determined from strain rate sensitivity measurements the dislocation velocity-stress exponent m^* for Nb-O alloys which exhibit alloy softening. m^* was found to depend strongly on interstitial concentration and temperature in the same manner that the flow stress and activation parameters are ⁽²⁶⁾. This is demonstrated in part by Fig. 6, where the parameter $(m^*T)_0$ (i.e. m^*T as $T \rightarrow 0$) is plotted as a function of oxygen concentration. The initial decrease and subsequent increase of $(m^*T)_0$ as a function of interstitial concentration parallels the concentration dependence of the flow stress. This is expected if m^* (and the thermally activated plastic flow in general) is impurity controlled; it is not expected if an intrinsic lattice strength is rate controlling.

(2) We are attempting to discern the specific origin of alloy softening in niobium-oxygen alloys. Previous work ⁽²⁶⁾ has shown that alloy softening in the Nb-O system is probably of ⁽²⁰⁾ extrinsic origin, viz. a result of solute association processes. However, we could not establish the particular mechanism(s) involved. This project involves the determination of the strength of Nb-O alloys either (a) prepared in various different ways, or (b) with various other interstitial solutes (C, N, H) added or removed. It is hoped that such investigations will reveal the origin of alloy softening as well as demonstrate how alloy hardening is manifested in controlled ternary alloys.

Only preliminary results have been obtained in this project, but these are very revealing. Fig. 7 demonstrates that the strength of Nb-O alloys may depend critically on the details of specimen preparation. In Fig. 7, the three alloys for which the stress-strain curves are shown were prepared identically except for the details listed in the figure caption. Specimens A and C are different in the extent to which they were purified prior to the addition of oxygen and in the manner in which they were diffusion annealed. Specimen B is different from Specimen A only in that they were diffusion annealed in a static and dynamic vacuum, respectively. We are following up these and other experiments presently.

(3) As a more direct part of the longer range goal of the program, the mechanical properties of alloys of controlled substitutional and interstitial content are being examined. As a prototype system, we are initially testing Nb-Mo alloys to which various interstitial solutes are added. We expect to determine the extent to which the various interstitials effect alloy softening. At present, the baseline data for "pure" Nb-Mo alloys are being generated to compare selectively with other investigators. (27,28)

Fig. 8 shows some initial results. As expected, alloy softening does not occur if the alloys are relatively pure with respect to residual interstitial solutes. Interestingly, there appears to be an appreciable orientation dependence of the flow stress in these alloys. Part of this may arise because of the differences in residual interstitial contents in the alloys of Peters and Henrickson (27) and Statham. (28), (29)

V. Anelasticity of BCC Refractory Metal Alloys.

In previous years, our use of Snoek-type anelasticity of bcc materials in the program was limited to determination of interstitial concentrations and detection of precipitation in Nb-O alloys. (25)
 This was true even though there existed considerable experience in (20,30,31) this area within the group. However, the results of several experiments have demonstrated the importance of this type of work in providing complementary information to studies of the type described in Sections III and IV. This was demonstrated in part in Section III where Snoek anelasticity was used with dislocation damping to determine dislocation-solute atom binding energies (Fig. 5). In this section, we will demonstrate anelasticity experiments which give important information concerning the nature of the alloy softening discussed in Section IV.

1. Anelasticity of Interstitial Alloys. We believe we have seen internal friction effects which if identified will disclose the origin of the alloy softening observed in Nb-O alloys. While using internal friction to routinely determine interstitial contents, we noted the oxygen Snoek peak (OSP) occurred several degrees above its expected peak temperature T_p for the frequency (~ 0.7 Hz) used. This was true even for relatively low concentration alloys (e.g. < 0.1 at. pct. oxygen) for which solute clustering should not be important in (30) affecting the peak temperature. Moreover, the peak showed a reproducible hysteresis in T_p on heating and cooling. Fig. 9 shows a typical result. The OSP occurs at $\sim 165^\circ\text{C}$ on heating and $\sim 148^\circ\text{C}$ on cooling. Upon heating the 165°C position is reproduced. The

(20)

vertical arrow indicates the expected position of the peak. The nitrogen Snoek peak (NSP) may be similarly affected, although the slight shift indicated in Fig. 9 is within experimental accuracy. (Strain aging and stress relief were occurring on heating.)

Our initial response to these results was one of disbelief - that they were probably an experimental artifact. However, we have noted similar types of results have appeared (unpublished) in other laboratories. ^(32,33,34) We are investigating the origin of this phenomenon, which surely involves clustering, ordering, or precipitation of interstitials, as part of our work on alloy softening in Nb-O alloys.

2. Substitutional - Interstitial Interaction in BCC Alloys.

We have previously argued that substitutional-interstitial interaction was responsible for the alloy softening observed in bcc alloys. ⁽²⁶⁾ In at least two alloy systems, Nb-Mo ⁽²⁸⁾ and Ta-Re, ⁽³⁵⁾ this has been demonstrated conclusively. We are examining these and possibly other alloy systems to see to what extent the anelastic behavior discloses substitutional-interstitial association as the particular type of interaction responsible for the alloy softening. In this respect, it is important to remember that basically the anelasticity can distinguish only three types of interstitial behavior:

- (a) interstitials in ordinary sites in solid solution, contributing to the Snoek peak;
- (b) interstitials in "extraordinary" sites (e.g. associated with other defects) in which they can contribute to anelasticity, but with a relaxation time different from that for the Snoek peak;

- (c) interstitials in sites for which they cannot contribute to anelasticity, e.g. in precipitates, large clusters, etc.

Careful experiments analyzed in detail are required in order to determine conclusively that interstitials do or don't interact more than just randomly with substitutional solutes or other defects.

Initial representative results in this area are given in Fig. 10 for Ta and Ta-Re alloys. Here we show that the addition of ~2 at. % Re to Ta which contains small amounts of oxygen and nitrogen in solution broadens both Snoek peaks. Thus solute interaction of the type (b) above can account for some of the alloy softening observed in this system. Smialek et al. (35) have shown that of the various interstitial solutes nitrogen most affects the low temperature strength of Ta and Ta alloys. Hence we expect that further experiments will disclose that the nitrogen Snoek peak behavior will correlate closest with the occurrence of the alloy softening.

The above experiments will be extended to Nb-base alloys soon. In addition we have prepared equipment for doing single crystal anisotropy studies (31) to aid in the determination of the specific solute clusters which cause these anelastic effects.

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NOTE: In the past contract year, the principal investigator has spent approximately 40% of his full time (defined as eleven months) with the research described in this total report. This time commitment breaks down to approximately 25% during the nine month academic year and 75% of a two month summer period. This will be true through to the end of the present contract period.

Fig. 1 Effects of (a) quench temperature, (b) quench rate, (c) deformation, and (d) electron irradiation on the carbon bound pair peak in Fe-Ni-C alloys. Quench temperature is 1100 $^{\circ}\text{C}$ except where otherwise designated. Frequency ~ 1 Hz.

Fig. 2 The 160°C martensite peak in an Fe-23.7 at. pct. Ni-3.5 at.pct. C alloy. The two curves represent successive heating runs on the same specimen. Frequency ~ 1 hz.

Fig. 3 The effect of tempering on the 160°C peak in a Fe-16.5 at. pct. Ni-3.1 at. pct. C martensitic alloy. Curve 1 is the purely austenitic alloy. Curve 2 is the as-quenched martensite. Curves 3-5 represent the peak after tempering at 200°C for one hour, at 200°C for 8 hrs, and at 250°C for 2 hrs, respectively.

Fig. 4 Dislocation damping in high-purity niobium ($\rho \approx 1000$). The designation I refers to amplitude-independent damping for both the undeformed and the deformed (1% in compression) material. Frequency ≈ 50 kHz.

Fig. 5a Dislocation damping and Snoek anelasticity in high purity tantalum. Frequency $\sim 0.5 \text{ Hz}$.

FIG. 5b. ANALYSIS OF DISLOCATION DAMPING AND SNOEK ANELASTICITY
OF HIGH PURITY TANTALUM

1. From the Granato-Lucke theory ⁽¹⁶⁾

$$\Delta \delta_I \propto \Delta L^4 \propto \Delta \left(\frac{1}{C_T^{(1)}} \right)^4$$

where

$$C_T^{(1)} = C_O^{(1)} + C_N^{(1)} \propto n_T^{(1)}$$

2. From the Snoek peaks

$$C_O^{(1)} = k_1 Q_{\max}^{-1}(O)$$

$$C_N^{(1)} = k_1 Q_{\max}^{-1}(N)$$

3. From the resistivity ratio Γ

$$\Gamma^{-1} = k_3 C_T^{(1)} = k_3 (C_O^{(1)} + C_N^{(1)}) \propto n_T^{(1)}$$

4. Thus

$$C_T^{(1)} = C_T^{(1)} - C_T^{(2)} \propto n_T^{(1)}$$

5. The average binding energy E_B for interstitials to dislocations is obtained from

$$\frac{n_T^{(1)}}{n_L - n_T^{(1)}} = \frac{n_T^{(2)}}{n_L - n_T^{(2)}} \exp \left(\frac{E_B}{kT} \right)$$

Where n and n_L are determined as in ref. 18. Individual binding

energies for oxygen and nitrogen can be obtained from additional selective determinations of $\Delta\delta_I$ and Γ after aging at different temperatures in the range between the oxygen Snoek peak and temperatures above the nitrogen Snoek peak.

LEGEND FOR FIGURE 5 (b)

$\Delta\delta_I$ = change in the amplitude - independent damping after cycling between 25°C and 450°C.

L = "loop length" or distance between interstitial pinning points along a dislocation.

$C_T^{(L)}, C_O^{(L)}, C_N^{(L)}$ = concentration of total (T), oxygen (O), and nitrogen (N) interstitials at dislocations (L). Total (T) is assumed to include only oxygen plus nitrogen.

$C_T^{(i)}, C_O^{(i)}, C_N^{(i)}$ = concentrations as above initially in solution.

$C_T^{(A)}$ = Total concentration in solution after atmosphere equilibration $(= C_O^{(A)} + C_N^{(A)})$.

$n_T^{(L)}, n_T^{(i)}, n_T^{(A)}$ = total number per unit volume of interstitials at dislocations (L), initially in solution (i), and in solution after atmosphere equilibration (A).

n_L = total number per unit volume of sites at dislocations available for occupancy by interstitials.

n_l = total number per unit volume of lattice sites available for occupancy by interstitials.

$Q_{\max}^{-1}(O), Q_{\max}^{-1}(N)$ = Snoek peak heights for oxygen (O) and nitrogen (N).

k_1, k_2, k_3 = experimentally determined proportionality constants.

Fig. 6 The limiting value of m^*T as $T \rightarrow 0$ for niobium-oxygen solid solutions.

Fig. 7 Resolved shear stress-resolved shear strain curves for a Nb-0.043 at. pct. O alloy A. diffusion annealed 1-1/2 hrs in an open vacuum of $\sim 8 \times 10^{-8}$ torr; B. diffusion annealed 1-1/2 hrs in a closed system at $\sim 8 \times 10^{-8}$ torr; C. diffusion annealed 2 hrs in a closed system at $\sim 2 \times 10^{-8}$ torr.

Fig. 8 Temperature and concentration dependence at 77° K of the resolved shear stress of niobium-molybdenum alloys. τ = resolved shear stress; τ^* = thermal component of the shear stress.

Fig. 9 Hysteresis in the position of the oxygen Snoek peak in niobium during heating and cooling.

Fig. 10 Anelasticity of Ta-O, N and Ta-2.1 at. pct. Re-O, N alloys. Frequencies as given in the figure.

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

Contract No. AT(11-1)-1676

Report No.

COO-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.
COO-1676-2	R. Gibala, Comments on The Cold-Work Peak in Alpha Iron, October 31, 1967.
COO-1676-3	R. Gibala and C. A. Wert, Interstitial Alloys of BCC Metals, October 31, 1967.
COO-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.
COO-1676-5	F. Povolo, Comment on The Influence of Hydrogen Content on the Relaxation Spectrum of α Titanium, November 7, 1968.
COO-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.
COO-1676-7	K. V. Ravi and R. Gibala, The Strength and Alloy Softening of BCC Metals, May 30, 1969.
COO-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.
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