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A PROGRESS REPORT FOR RESEARCH ON
DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS
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conducted at ¹ ₁₀₀?

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A PROGRESS REPORT FOR RESEARCH ON
DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

Three projects suggested in the original proposal to the AEC were undertaken in part or in total during the past year. The progress on these projects is reported herein.

I. Dislocation-Interstitial Solute Interaction in Close-Packed Metals.
(Investigators - M.G. Ulitchny, R. Gibala)

A program for investigation of dislocation-interstitial solute interactions in close-packed metals by utilizing the anelastic behavior of the interstitial solid solutions was undertaken at the beginning of the present contract period. Face-centered cubic iron-nickel alloys doped with carbon were chosen for initial studies because of the known existence of substitutional - interstitial (s-i) interaction relaxation peaks in these materials⁽¹⁾ and the availability, in part, of mechanical properties studies associated with the effects of interstitials.^(2,3) Alloys of high nickel contents (≥ 35 at.pct.) were chosen because of their low M_s and M_d temperatures.⁽⁴⁾ The alloys have been obtained in rod form of two purity levels: commercial Invar, which is nominally 36 at.pct.Ni, and high purity Fe-36%Ni castings (99.9+ Fe and Ni), and have been swaged and drawn to 0.025 in. to 0.040 in. wires.

The main effort in the first eight months of work on this project has been in designing, building and perfecting a high temperature (room temperature to 1200°K) vacuum torsion pendulum required for the anelasticity experiments. The pendulum, shown in Figure 1, is a modified version of a design developed by Peters.⁽⁵⁾ Also built during the present contract period was a gas train and furnace system for controlled addition of carbon to the Fe-Ni alloys. This work has now been completed,

and preliminary data are being obtained. At present, we have only confirmed the existence of the s-i interaction peak observed by Wu and Wang,⁽¹⁾ which we will use as a measure of the amount of interstitials in solution, See Figure 2. Strain-aging and precipitation experiments are in progress, and definitive results are expected before the end of the present contract period. We anticipate being able to establish the existence of a carbon-dislocation interaction (i.e. cold-work) peak, similar perhaps to ones observed for hydrogen in austenitic steels.⁽⁵⁾

An important aspect of this problem will involve comparison of the results in fcc metals with their counterparts in the more well-established studies of bcc metals.⁽⁷⁾ Because the mechanism of the cold-work peak (CWP) is still not well established in bcc metals, we have examined critically the investigations on bcc metals as part of the past year's activities on this project. Our present feelings are essentially unchanged from that indicated in a recent publication,⁽⁸⁾ that a mechanism involving vibrating dislocations which drag their attendant interstitial atmospheres with them best describes the physical situation. Of considerable importance to future work on the CWP, we feel, will be experiments which relate the dislocation distributions determinable by transmission electron microscopy to the relaxation parameters of the CWP.^(9,10) Our views are expressed in a paper submitted to and accepted by Scripta Metallurgica, as part of this project. Preprints listed as Report No. C00-1676-2 are included with this progress report.

II. Solute Partitioning and Its Effects on Strengthening in BCC Refractory Metals (Investigators - K.V. Ravi, R. Gibala)

An experimental program to study interstitial hardening and the effect of interstitial partitioning (in solution, precipitated, defect-associated, etc.)

on the hardening has been initiated in September, 1967 of the present contract period. The niobium-oxygen and niobium-nitrogen systems have been chosen for investigation because of their terminal solid solubility behaviors are reasonably well understood. We have decided that the base material for the study will be zone-refined and vacuum outgassed (10^{-8} to 10^{-9} mm Hg) niobium single crystals. Preliminary studies (see Section III) have indicated that these materials have very low residual interstitial levels. Addition of interstitials in controlled amounts will be accomplished either by a volumetric technique ⁽¹¹⁾ or by diffusion annealing of deposited anodic films. ⁽¹²⁾ These techniques are being perfected at present. We anticipate initial results on hardening behavior by the end of the present contract period.

A necessary corollary to this study is the investigation of the nature of interstitial solute partitioning processes in bcc metals, including solubility, solute-solute interactions, solute-dislocation interactions, etc. To this end, a large part of Professor Gibala's activity associated with this project has been in the assembly and writing of a review paper entitled tentatively "Interstitials in the BCC Metals". The paper is being co-authored with Professor C.A. Wert of the University of Illinois. Preprints of the paper are included with the progress report as Report No. C00-1676-3. Selected topics from the paper will be presented as an invited talk at the Annual Meeting of the Metallurgical Society of AIME in February, 1968. A slight shortened and revised version will appear in the many-volumed series Energetics of Metallurgical Phenomena.

III. Dislocation-Substitutional and -Interstitial Interaction in BCC Metals (Investigators - F. Povolo, R. Gibala).

Dislocation - solute atom interactions in the bcc refractory metals

are being investigated by study of the temperature and concentration dependence of amplitude dependent (hysteretic) internal friction due to dislocations.⁽¹³⁾ Present experiments are being conducted on niobium and dilute niobium-base alloys (molybdenum, vanadium, and zirconium as solutes).

Internal friction in these experiments is measured by the Marx three-component piezoelectric technique,⁽¹⁴⁾ in a method similar to that employed by Fiore and Bauer.⁽¹⁵⁾ The equipment is shown in Figure 3. The main feature of this set-up is its valve-less construction, which permits measurements in a vacuum of better than 10^{-6} torr at the furthest point from the pumping system. Modifications being planned will lower the ultimate vacuum to $\sim 10^{-8}$ mm Hg. during measurements. The same system with a modified head (Figure 4) is used to vacuum outgas previously zone-melted specimens near the melting point in vacuums of 10^{-8} to 10^{-9} torr. The need for the high vacuum will be evident from the discussion below.

The results obtained thus far are best given summarily in chronological order. Earliest studies were made on single crystals of Nb and Nb-V, -Mo, and -Zr binary alloys (≤ 1 at.pct. solute) which were zone-melted by usual techniques (vacuum of $\sim 10^{-5}$ torr, zone speeds of approximately 20 in. per minute). Figure 5 shows typical internal friction results on these materials. In both the pure metal and alloys there is only a slight amplitude dependence of the dislocation damping at all temperatures and then only at the highest attainable amplitudes. There are also no clear differences in the damping behavior of either the metal or the alloys as far as magnitude, "breakaway" amplitude, slope of the amplitude dependent damping, etc.

It became clear from such results that residual interstitial levels were dominating the observations. Thus long time, high temperature solid state vacuum outgassing of the zone-melted specimens was undertaken

in an attempt to further purify the base materials. The effect of the outgassing treatments—a substantial decrease in breakaway amplitude and an increase in the slope of the amplitude dependent damping—is shown in Figure 6 for a Nb - 1% Zr alloy and indicates the success of the treatments. Similar results were obtained for unalloyed Nb and Nb-V alloys so that the results can not be attributed to zone refining of the substitutional solute; nevertheless, this is an important experimental problem.

The effect of temperature, which should redistribute the solute atoms to and from dislocation sites, was observed to be complex. Over the temperature range 23°C to 800°C, increasing the temperature, which should rarefy the solute atmospheres at dislocations, generally had the expected effect of decreasing the breakaway amplitude and increasing the slope of the amplitude dependent damping. Figure 7 shows such results for a nominal Nb- 1% V alloy over a limited temperature range. Similar results are reflected in the data given in Figure 8 for pure Nb. Here the amplitude dependent damping is plotted to according to the form suggested by the Granato-Lucke theory.⁽¹³⁾ According to the theory, the plots of $\ln (Q_H^{-1} \cdot \epsilon_0^{\frac{1}{2}})$ vs $1/\epsilon_0$ (where Q_H^{-1} = amplitude dependent damping and ϵ_0 = strain) should be straight lines. The slopes should increase as the solute atmospheres at dislocations rarefy with increasing temperature. That the plots are not straight line in almost all cases makes detailed analysis of the results in terms of the dislocation - solute interaction processes operating difficult at present. From the complicated effects of temperature over the entire range investigated, it appears that more than one pinning process (with different temperature dependences) are operating and that the damping must be described as a sum of Granato-Lucke processes, as a sum of modified Granato-Lucke processes,⁽¹⁶⁾ or perhaps by a different model altogether.

At this preliminary stage in the investigation, we can not say for sure how the data should best be analyzed. However, it is clear that a model perhaps different in detail from the unmodified Granato-Lucke theory might be necessary. One very striking observation has evolved from the observations thus far. This is that while the amplitude dependent damping is not adequately described by the Granato-Lucke approach, it is accurately described by a simple linear law, i.e. $\bar{\phi}_H^{-1}$ is proportional to ϵ_0 . In cases where the damping is not strictly linear, it is then closely given by a sum of two straight lines. Typical data for unalloyed Nb are shown in Figure 9. Data obtained on the dilute alloys are similar.

The unusual changes in the slopes of the damping curves with temperature should be noted. While we are unsure of the interpretation of these effects at present, we point out that (1) they indicate substantial reshuffling of pinning points is occurring with increasing temperature, and (2) the effects are apparently related to the positions of the oxygen and nitrogen Snoek peaks in niobium.⁽¹⁷⁾

In summary, research on this project has thus far established the following:

1. Substitutional solutes have only minor effects on the dislocation damping in bcc refractory metals, even after extended purification treatments, compared to residual interstitials.
2. The classical Granato-Lucke theory does not adequately describe the strain amplitude dependence of the dislocation damping.
3. A linear dependence of damping on strain amplitude best characterizes the results on Nb and some of its dilute substitutional alloys.
4. The temperature dependence of the amplitude dependent damping shows the effect of at least two stages of pinning over the range 23°C to 800°C.

The aim of this project in the coming year is three-fold:

- (1) to extend the measurement described above to temperatures below room temperature,
- (2) to establish as a baseline the dislocation damping associated with residual interstitials in the unalloyed niobium, and then
- (3) to follow through with measurements on the dilute alloys to establish better the role of the substitutional solutes in the pinning processes.

We expect results on the unalloyed niobium to be in a form ready for publication by the end of the coming year.

In summary for the entire research program during the present contract period, we can say the following:

1. All of the originally proposed projects have been initiated, however not all of them from the inception of the contract on February 1, 1967. The reason was simply that not enough acceptable prospective graduate assistants were available from the beginning of contract period.
2. In some instances, the scope of the individual projects was expanded or complemented, mainly through written papers based on critical examination of the technical literature.
3. With the exception of the problem of the initial unavailability of a graduate assistant for work on one of the three experimental projects (No. 11 above), all of the proposed projects have proceeded closely to the anticipated time schedule given in the original proposal.

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Technical Reports, Articles Prepared
for Publication, etc. (See appended material.)

PREPRINTS

1. "Comments on 'The Cold-Work Peak in Alpha Iron'", by R. Gibala, Accepted for publication by Scripta Metallurgica.

2. "Interstitials in the BCC Refractory Metals," by R. Gibala and C.A. Wert, to appear in Energetics of Metallurgical Phenomena. (University of Denver Summer Lecture Series, 1966.)

Figure 1. Photograph of the high temperature vacuum torsion pendulum used in experimental studies of cold-work peaks.

Figure 2. Internal friction of an Fe-36%Ni-.04%C steel, showing a Ni-C pair reorientation peak.

Figure 3. Photograph of the equipment for measuring internal friction in the khz range in vacuums of 10^{-6} torr and lower.

Figure 4. Adaption of the equipment shown in Figure 3 for high temperature vacuum outgassing of bcc refractory metals.

- A = SPECIMEN ON CERAMIC HOLDER
- B = HIGH FREQUENCY INDUCTION COIL
- C = WAX JOINTS
- D = IONIZATION GAUGE
- E = LIQUID N₂ TRAP
- F = BAFFLE
- G = TO LIQUID N₂
- H = DIFFUSION PUMP
- I = TO MECHANICAL PUMP

ALL GLASS CONSTRUCTION BEYOND BAFFLE

Figure 5. Dislocation damping of zone-melted niobium and niobium-base alloys. Damping values are uncorrected for transducer mass.

Figure 6. Dislocation damping in (A) zone-melted and (B) zone-melted and vacuum outgassed Nb-1% Zr alloys (nominal).

Figure 7. Temperature dependence of the dislocation damping of a Nb-1% V alloy (nominal).

Figure 8. Temperature dependence of the dislocation damping of unalloyed Nb. The data are plotted according to the Granato-Lücke theory. (13) Temperatures are in °C.

Figure 9. Demonstration of the linear dependence of amplitude-dependent dislocation damping on strain amplitude in unalloyed niobium.