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**ENVIRONMENTAL REACTIONS AND THEIR EFFECTS ON
MECHANICAL BEHAVIOR OF METALLIC MATERIALS**

Technical Progress Report

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ABSTRACT

The results of research performed under ERDA Contract No. AT(11-1)-1676 are presented in two sections. The first section presents new results obtained in two current project areas: (1) surface oxide softening of the Group VB refractory metals; (2) mechanical behavior of Nb-H and Nb-D alloys. The results include: (a) experimental verification of a model of surface oxide softening of body-centered cubic metals; (b) determination of a stress-differential effect in surface oxide softening; and (c) characterization of hydrogen and deuterium strengthening in Nb and Nb-O alloys. The second section reviews the long-range research accomplishments of the program since its inception and evaluates the major contributions in several individual projects areas that have involved topics on: interstitials in metals, mechanical behavior of body-centered cubic metals, solute-defect interactions and internal friction mechanisms in solids.

I. CURRENT RESULTS

A. Surface Oxide Softening of the Group VB Refractory Metals (V.K. Sethi)

We have shown in previous progress reports that the low temperature mechanical properties of niobium and tantalum are drastically altered by the application of a thin surface oxide film. The effect is summarized in Figure 1, which shows a set of resolved shear-stress strain (τ - γ) curves for uncoated and coated niobium at 77°K. The τ - γ curve for uncoated niobium shows parabolic work hardening behavior and the critical resolved shear stress (CRSS) measured at 2% shear strain is 180 MN/m^2 . When a 65nm thick surface oxide film is applied to the niobium crystals the CRSS is reduced to 120 MN/m^2 and the τ - γ curve exhibits serrations. If the coated niobium is prestrained at 295°K prior to the 77°K deformation the flow behavior is further altered. The serrations disappear and the τ - γ curve exhibits a three stage work hardening behavior. In addition the CRSS is reduced to 60 MN/m^2 and the ductility is increased. Similar prestraining treatment does not affect the mechanical behavior of uncoated niobium to such a large extent.

We have proposed a model⁽¹⁾ that explains surface oxide softening in terms of the ability of the oxide-metal interface to act as an effective source of edge dislocations. Because of the high mobility of edge dislocations relative to screw dislocations, plastic deformation can be effected at greatly reduced applied stresses. In the past contract year, our main effort has been to obtain new results on oxide-coated niobium that would confirm the validity of the proposed mechanism. The experiments chosen were classical ones dealing with surface effects in crystal plasticity⁽²⁾, as described below.

Single crystal rods of niobium 260mm long were prepared from 6.3mm diameter polycrystalline rods by electron-beam zone melting in a vacuum of 10^{-3} Pa. The crystals, oriented with the tensile axis approximately parallel to [325], were ground flat by hand to 2.0mm x 2.0mm square cross-section with the face orientations such that primary (011)[$\bar{1}\bar{1}1$] edge dislocation lines were parallel to one pair of faces

(E faces) and primary screw dislocation lines were parallel to the other set of two faces (S faces). After final shaping, the specimens were chemically polished to remove all surface damage. Individual crystals were then purified by the solid state outgassing technique described elsewhere⁽¹⁾.

Oxide films 65nm were deposited anodically in a 10% phosphoric acid solution at constant voltage of 30V at 295°K. Some of the specimens were oxidized on all four surfaces while some specimens were selectively oxidized on either the E faces or the S faces by covering one pair of faces with lacquer during anodization.

Mechanical testing was done in tension at a base resolved shear strain rate of 5.5×10^{-4} /sec at 77°K. Some specimens were prestrained at 295°K to 0.10 shear strain prior to testing at 77°K to give maximum possible oxide softening. Shear stress-shear strain curves were computed by resolving slip on the primary (011)[$\bar{1}\bar{1}$ 1] system.

The results of these experiments are illustrated in Figure 2 in the form of τ - γ curves. The flow behavior of uncoated and coated (all four faces) niobium crystals of square cross section at 77°K is identical to that observed for crystals of circular cross section presented in our previous contract reports and publications. When only E faces are coated the τ - γ curve is serrated and the CRSS is comparable to that of niobium crystals coated on all four sides. On the other hand, when only S faces are coated the τ - γ curve is serrated but the CRSS is hardly affected by the presence of surface oxide film, and is as large as that of uncoated niobium. Figure 2 also illustrates that the prestrain at 295°K affects the mechanical behavior of the crystals that are preferentially coated on E faces as if they were coated on all four sides, while it affects the mechanical behavior of S coated crystals as if they were not coated at all.

These observations, coupled with the slip trace analyses and etch pit studies on specimens preferentially coated on E or S faces (which are currently being investigated and will be reported in later reports), strongly support the proposed model.

Specimens coated on the E faces preferentially generate dislocation loops with a large edge component which can move large distances across the active glide plane. Loops generated from coated S faces are limited from moving large distances by their large immobile screw components; hence these crystals do not exhibit any appreciable softening.

Another major result withing this part of the current program involves determination of the stress-differential effect in surface oxide softening, i.e. a difference in yield and flow stresses in tension versus compression for oxide-coated metals. In our model of surface oxide softening, it is argued that the growth stresses associated with the oxidation introduce large tensile stresses in the metal substrate immediately adjacent to the oxide. Under applied stress the growth stresses are large enough to allow heterogeneous nucleation of dislocations. The softening effect should be maximum when the applied stress and the internal stress in the substrate are of the same sign, i.e. tensile. On this basis the surface oxide softening of niobium was determined for both tensile and compression specimens at a common low temperature, 77°K.

The experimental results have been presented in detail in ERDA Report No. C00-1676-46 and in the publication based on that report⁽³⁾. Figure 3 gives a summary of those results. It is evident from the plots of the CRSS versus oxide thickness for all materials examined, including the unoxidized niobium, that the yield stress is larger in compression than in tension and that the stress-differential becomes larger with increasing oxide thickness. These are the results expected from our model.

Other results concerning surface oxide softening involving slip traces, dislocation etch pits, dislocation substructures by transmission electron microscopy and effects of interstitial and substitutional solutes are in progress and will be reported in detail in subsequent reports and papers.

B. Mechanical Behavior of Nb-H and Nb-D Alloys (R. Fournier)

Our program on the mechanical behavior of Nb-H alloys has been continued during the past year, with emphasis centering on the characterization and study of the low temperature strengthening mechanisms and the effects of a second interstitial solute, oxygen.

The results presented in this report include:

1. a study of the flow stress in Nb-H single crystals as a function of hydrogen concentration, temperature and prestrain at 295°K;
2. investigation of the isotope effect on the strength of Nb-H alloys at 77°K;
3. determination of the influence of oxygen on the mechanical behavior of Nb-H alloys.

Figure 4 shows the strengthening in Nb arising from different hydrogen concentrations. At temperatures above 77°K we find that for concentrations greater than 200 ppma, the strengthening is linear. Contrasting this behavior, Figure 4 shows that at 77°K the hydrogen strengthening exhibits a maximum as a function of hydrogen concentration. Also evident is the effect of prestrain on the CRSS: at 175°K and 131°K, it is found that deforming to 0.03 shear strain at 295°K reduces the flow stress, whereas the opposite is true at 77°K for concentrations less than 800 ppma H.

Interpretation of the curves in Figure 4 by any one hardening theory is difficult. The most probable origin of the maxima observed at 77°K involves transition of the hydride from coherent to semi-coherent to incoherent particles as the hydrogen concentration is increased. At low concentrations, where maximum strengthening is observed, hydrides are known to be coherent ⁽⁴⁾ and would strengthen the matrix appreciably by interaction of the coherency strains with dislocations. As the hydrogen content is increased, hydrides increase in size and become incoherent. The incoherent particle-matrix interface is capable of acting as an effective source of prismatic dislocation loops during deformation ⁽⁴⁾. This increase in dislocation density is thought to be responsible for the softening

observed at concentrations above 800 ppma H at 77°K⁽⁵⁾.

The higher CRSS of prestrained crystals at 77°K is probably a result of more extensive hydride nucleation at dislocations. Thus prestrained specimens, with a larger dislocation density, obtain a more finely dispersed and more coherent second phase that strengthens more than the coarser particles present in unprestrained material. By contrast, the major effect of prestrain at temperatures above 77°K appears to be that of increasing the mobile dislocation density of the crystal.

Figure 5 shows that there is no isotope effect on the strengthening of Nb-H single crystals at 77°K. This behavior is consistent with the similarities of hydrogen and deuterium in niobium with regard to the hydride structures⁽⁴⁾, the lattice distortions⁽⁷⁾ and the high diffusivities⁽⁸⁾.

Some of our results on the effect of oxygen on the CRSS of Nb-H alloys at 77°K are included in Figure 5. We find that depending on the relative amounts of hydrogen and oxygen present a hardening or softening exists in the ternary alloys. This is shown by inclusion of some of the numerical data given in Table I. The results are interpreted as follows: the softening observed at 200 ppma H is a direct result of oxygen-hydrogen cluster formation reducing the effective hydrogen concentration available for precipitation. Under these circumstances the extent of coherent precipitate hardening, which predominates in Nb+200 H alloys, is reduced by the formation of oxygen-hydrogen complexes. Increasing the oxygen concentration increases the softening effect (compare entries 1 and 2 in the Table). At a concentration of 800 ppma H ternary alloys produce synergistic hardening. See entries 3 and 4 in Table I. For these concentrations, we find that in Nb-H single crystals incoherent particle strengthening predominates. Thus the addition of oxygen which interacts with hydrogen allows smaller, less incoherent hydride precipitates to occur. As a result, there is less prismatic punching that produces the hydride softening observed at higher hydrogen concentrations. If we

also take into account solution hardening by oxygen and the possibility of hardening from oxygen-hydrogen clusters, then an increased strengthening with increased oxygen concentration is to be expected, as observed in Figure 5 and Table I.

A complete description of the mechanical behavior of Nb-H alloys will rely on correlation of mechanical properties with the wealth of information that is being accumulated on hydrogen diffusion, hydrogen-defect interactions and hydride phase structure, thermodynamics, transformations and kinetics⁽⁹⁾. We hope to supply some of this information in our future investigations by performing in situ tensile experiments on Nb-H alloys in the CWRU high voltage electron microscope. These experiments will allow direct observation of the hydride phase transformations during mechanical testing. Our current investigations on mechanical testing are presently being extended to temperatures below 77°K and we are determining the effect of oxygen on deuterium strengthening. Finally detailed analytical interpretations of the results presented in this report are being undertaken and should be complete by the end of the next contract period.

II. LONG-RANGE RESEARCH ACCOMPLISHMENTS

This research program was initiated in February, 1967 under the title "Dislocation-Solute Atom Interactions in Alloys" with two graduate students and a research associate. It has been a two or three person effort (in addition to the principal investigator) each year since that time. The people who have been involved in the program and the project areas they have worked in are given in the long lists of ERDA Technical Progress Reports (47), publications (53), technical presentations (64) and theses (10) enclosed as part of this current Technical Progress Report. From these lists it is certainly fair to say that quantitatively our program has been successful. An average yearly output of one thesis, five reports, three papers and six talks from a group has usually been a total of three research people speaks for itself. However, we are far more pleased with the qualitative

reputational impact the research has made. The Department of Metallurgy and Materials Science at Case Western Reserve University is often regarded as the place to come, visit, call or write concerning topics of interstitial solutes in metal, mechanical behavior of body-centered cubic metals, hydrogen in metals and internal friction of solids. To a large extent that reputation has evolved from the ERDA research of the principal investigator and related collaborative work he has done with several of his colleagues.

This section of the current Progress Report summarizes briefly the major accomplishments of the program over its duration. No attempt is made to rank order the worth or long-range significance of the many individual research projects. Instead, a set of six major findings that usually cut across more than one individual project are listed and described briefly. Consistent with our attempt not to rank the individual projects, the order in which the findings are listed is for the most part chronological. In what follows, we give the finding and the research persons who dealt with the problem and the brief description.

1. Carbon-vacancy interaction in austenite is large. (M.G. Ulitchny, J.A. Slane)

This project used internal friction techniques to investigate carbon-defect interactions in austenitic steels. It was found that the internal friction results and other data in the literature on strain aging of austenite and self-diffusion of iron in carbon-containing austenite could be interpreted consistently in terms of a large (~ 30 kJ/mol) carbon-vacancy binding energy.

2. Interstitial solutes effect solution hardening and solution softening in body-centered cubic metals at low temperatures, $T \leq 0.15T_m$. (K.V. Ravi, M.G. Ulitchny, A.K. Vasudevan, V.K. Sethi)

The origin of the large temperature-dependent strength of bcc metals at low temperatures represented a major controversial research area during the 1960's and early 1970's. Our research on high purity niobium and tantalum showed that (1) a large fraction of the strength comes from solid solution hardening by residual

interstitial solutes; (ii) however there exists also a large remnant intrinsic (Peierls) strength of the pure bcc metal; (iii) the phenomenon of solution softening of substitutional and interstitial alloys based on niobium and tantalum has its origin in the effect which solute (impurity) interactions have on the interstitial hardening component of the low temperature strength.

3. Dislocation-solute interaction energies and solute-solute interactions that characterize non-ideality of interstitial solid solutions of bcc metals have been determined. (G.J. Klems, A.A. Sagues, R.P. Krupitzer, F. Povolo)

These projects used several types of internal friction processes to analyze solute-defect interactions that related to other problems of interest to us. Snoek relaxation peaks, substitutional-interstitial "Snoek-like" interaction peaks, Köster or cold-work peaks, and amplitude-independent and amplitude-dependent dislocation background damping processes were examined. The hydrogen-dislocation interaction energy in alpha iron was confirmed to be ~ 25 kJ/mol. A carbon-dislocation energy of ~ 50 kJ/mol was found to be consistent with detailed analyses of cold-work peaks. Various substitutional-interstitial and interstitial-interstitial energies in the range 10-40 kJ/mol were determined and were found to be large enough to account for solution softening in bcc alloys (see finding no. 2) in terms of effects that solute interactions have on interstitial solution hardening.

4. A comprehensive model of internal friction of carbon in ferrous martensites has been developed. (G.J. Klems)

As part of the program to determine solute-defect interactions in iron-base alloys, the internal friction of carbon in a wide range of martensitic alloys was examined in the temperature range $25^{\circ}\text{C} \sim 500^{\circ}\text{C}$. All results for dislocated martensites could be successfully interpreted in terms of the existence of a relaxation peak caused by Schoeck-type carbon atmosphere dragging by unit dislocations at $\sim 250^{\circ}\text{C}$ at 1 Hz. All results for twinned martensites could be successfully

interpreted in terms of a similar type of relaxation peak caused by carbon-twin boundary interaction at $\sim 160^\circ\text{C}$ at 1 Hz.

5. Hydrogen strengthening and hydrogen embrittlement in bcc metals have been related to various hydrogen-defect interactions. (K.V. Ravi, M.G. Ulitchny, A.A. Sagues, R. Fournier)

In a series of individual projects on niobium and niobium-oxygen alloys, we have been able to show that (i) hydrogen strengthening in the metal or interstitial alloys at low temperatures is appreciable; (ii) hydrogen in solution and in particular small hydrogen-interstitial impurity clusters cause observable hardening and softening effects; and (iii) these hardening and softening effects are explainable in terms of how hydrogen-defect interactions affect hydride precipitation in the materials. This project is a continuing one for which a major effort will be made to use in-situ deformation of specimens in the high voltage electron microscope. This work should enable us to understand better the relationship between hydride precipitation and morphology and the observed hydrogen strengthening effects.

6. Solute gradients and surface films can cause large hardening or softening effects in bcc metals. (V.K. Sethi)

One of the most dramatic results we have obtained involves experiments which have shown that deposition of thin (50-100nm) oxide films on niobium or tantalum single crystals can virtually eliminate the large temperature dependence of the strength of these metals at low temperatures (see finding no. 2). We have developed a model which explains this effect in terms of how nucleation of edge dislocations at the film-substrate interface can permit circumvention of the normal Pellerls strength of pure bcc metals that is associated with screw dislocation mobility. Similar effects have been found when a steep solute or point defect gradient exists in a material near the surface. In addition to having potential commercial significance, this effect will permit many new types of fundamental research

experiments related to dislocation dynamics to be done.

More detailed descriptions of each of these findings are available in the published papers based on the ERDA program. The list of publications is given on pp. 21-23 of this report. The relevant papers for each finding are given in Table II.

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TABLE I

Added strengthening (the strength above that of unalloyed Nb [155 MN/m²]) from binary Nb-H and Nb-O alloys and from ternary Nb-H-O alloys. All materials are prestrained at 295°K, then quenched to and tested at 77°K.

1.	$\Delta\tau$ (Nb + 200H) + $\Delta\tau$ (Nb + 500 O) = 152 MN/m ²
	$\Delta\tau$ (Nb + 200H + 500 O) = 142 MN/m ²
2.	$\Delta\tau$ (Nb + 200H) + $\Delta\tau$ (Nb + 1000 O) = 160 MN/m ²
	$\Delta\tau$ (Nb + 200H + 1000 O) = 122 MN/m ²
3.	$\Delta\tau$ (Nb + 800H) + $\Delta\tau$ (Nb + 500 O) = 102 MN/m ²
	$\Delta\tau$ (Nb + 800H + 500 O) = 120 MN/m ²
4.	$\Delta\tau$ (Nb + 800H) + $\Delta\tau$ (Nb + 1000 O) = 110 MN/m ²
	$\Delta\tau$ (Nb + 800H + 1000 O) = 156 MN/m ²

TABLE II

Papers on which the long range research findings are based. See pp. 21-23 of this report.

<u>Finding No.</u>	<u>Paper Nos.</u>
1	14
2	3, 6, 7, 8, 10, 12, 13, 16, 17, 18, 22
3	1, 2, 5, 9, 15, 19, 20, 25
4	21, 25, 26
5	1, 4, 5, 8, 11, 23, 24, 32
6	22, 27, 28, 29, 30, 31, 33

Figure 1. Resolved shear stress-shear strain curves for niobium single crystals of circular cross section tested at 77°K in the conditions indicated.

Figure 2. Resolved shear stress-shear strain curves for niobium single crystals of square cross section tested at 77°K in the conditions indicated. E and S faces are defined in the text.

Figure 3. The critical resolved shear stress in tension and in compression and the stress-differential at 77°K and 295°K as a function of oxide thickness.

Figure 4. The critical resolved shear stress (CRSS) of niobium single crystals at various temperatures as a function of hydrogen concentration.

Figure 5. The critical resolved shear stress (CRSS) at 77°K of Nb-H, Nb-D and Nb-H-O alloys as a function of concentration.

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

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.
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6. K.V. Ravi and R. Gibala, "The Strength of Niobium-Oxygen Solid Solutions", Acta Met. 18, 623 (1970).
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9. A.A. Sagues and R. Gibala, "Substitutional-Interstitial Solute Interactions in Ta-Re-N Solid Solutions", Scripta Met. 5, 689 (1971).
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11. M.G. Ulitchny and R. Gibala, "Solute Interactions Involving Hydrogen and Their Influence on Mechanical Behavior of Niobium Single Crystals", Proceedings of the International Congress on Hydrogen in Metals, p. 128, Editions, Science et Industrie, Paris (1972).

12. K.V. Ravi and R. Gibala, "Dislocation Dynamics in Niobium-Oxygen Solid Solutions, Metall. Trans. 3, 3081 (1972).
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14. M.G. Ulitchny and R. Gibala, "Internal Friction and Strain Aging of Austenitic Alloys", Metall. Trans. 4, 497 (1973).
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19. A.A. Sagues and R. Gibala, "Substitutional-Interstitial Interactions in Ta-Re-N and Ta-Re-O Alloys", Acta Met. 22, 1423 (1974).
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23. R. Gibala, "Hydrogen-Defect Interactions in Iron Base Alloys", Proc. Int. Conf. on Stress Corrosion Cracking and Hydrogen-Embrittlement of Iron-Base Alloys, NACE, Houston (1975).
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31. V.K. Sethi and R. Gibala, "Surface Oxide Softening of Niobium Single Crystals", *Acta Met.* 25, 321 (1977).
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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, Los 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. C.J. Klems, R.E. Miner, F.A. Hultgren and R. Gibala, "Internal Friction in Twinned Fe-Ni-C Martensites", AIME Meeting, Detroit, October 1971.
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.
21. M.G. Ulitchny and R. Gibala, "Thermally Activated Deformation Behavior of Nb-O-N Single Crystals", AIME Meeting, Boston, May 1972.
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.
27. A.K. Vasudevan and R. Gibala, "Alloy Softening in Nb-Mo-O and Nb-Mo-N Solid Solutions", AIME Meeting, Philadelphia, May, 1973.
28. A.A. Sagues and R. Gibala, "Substitutional-Interstitial Solute Interactions in Ta-Re-N and Ta-Re-O Solid Solutions", AIME Meeting, Philadelphia, May, 1973.

29. R. Gibala, "Hydrogen-Defect Interactions in Iron-Base Alloys", International Conference on Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Base Alloys, Unieux-Firminy, France, June, 1973.
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35. R. Gibala, "Dislocation Relaxation in BCC Metals", Max-Planck Institut für Metallforschung, Stuttgart, Germany, April, 1974.
36. R. Gibala, "Internal Friction in Ferrous Alloys", Université de Paris, Colloque Internationale, Paris, France, April, 1974.
37. R. Gibala, "Relaxation de Dislocation dans Metaux Cubiques Centres", Laboratoire de Mécanique et Physique des Matériaux, Université de Poitiers, Poitiers, France, April, 1974.
38. R. Gibala, "Interactions Hydrogene-Defaut dans Metaux", Centre d'Etudes Nucléaires de Grenoble, Grenoble, France, April, 1974.
39. R. Gibala, "Relaxation de Dislocation dans Metaux Cubiques Centres", Ecole Polytechnique Fédérale de Lausanne, Lausanne, Switzerland, July, 1974.
40. R. Gibala, "Interstitials in BCC Metals", General Electric Company, Lamp Division, Cleveland, Ohio, October, 1974.
41. R. Gibala, "Effects of Interstitials on Mechanical Behavior of BCC Metals", Rice University, Houston, Texas, December, 1974.

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43. R. Gibala, "Internal Friction of Ferrous Martensites", Conference on Martensite Transformations, M.I.T., Cambridge, Mass., April, 1975.
44. R. Gibala, M. Wuttig, W. Kunz and P. Moser, "Anelastic and Magnetic Relaxation in Face-Centered Cubic Ni-Co-C Alloys", AIME National Spring Meeting, Toronto, Canada, May, 1975.
45. R.P. Krupitzer and R. Gibala, "Effects of Preferred Orientation on Snoek Phenomena in Commercial Steels", AIME National Spring Meeting, Toronto, Canada, May, 1975.
46. V.K. Sethi and R. Gibala, "Low Temperature Mechanical Behavior of Oxide-Coated Single Crystals of Niobium", AIME National Spring Meeting, Toronto, Canada, May, 1975.
47. A.A. Sagues, M.G. Ulitchny and R. Gibala, "Hydrogen Strengthening in Niobium and Niobium Base Alloys", International Conference on Effects of Hydrogen on Behavior of Materials, Jackson Hole, Wyoming, September, 1975.
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50. V.K. Sethi and R. Gibala, "The Effect of Anodic Oxide Coatings on the Mechanical Behavior of Niobium and Tantalum Single Crystals", International Conference on Metallurgical Coatings, San Francisco, California, April, 1976.
51. V.K. Sethi and R. Gibala, "The Effect of Oxygen Distribution on the Low Temperature Mechanical Behavior of Niobium Single Crystals", Second International Conference on Mechanical Behavior of Materials, Boston, Mass., August, 1976.
52. V.K. Sethi and R. Gibala, "Surface Oxide Softening in Single Crystals of Niobium and Tantalum", Fourth International Conference on Strength of Metals and Alloys, Nancy, France, August, 1976.
53. R. Gibala, "Effects of Interstitial Distributions on Mechanical Behavior of BCC Refractory Metals", AIME Fall Meeting, Niagara Falls, N.Y., September, 1976.
54. T.E. Mitchell, V.K. Sethi and R. Gibala, "Interstitial Solution Hardening in BCC Refractory Metals", AIME Fall Meeting, Niagara Falls, N.Y., September, 1976.

55. R. Gibala, "Effects of Interstitial Distributions on Mechanical Behavior of BCC Metals", General Motors Corporation, Warren, Michigan, January 1977.
56. V.K. Sethi and R. Gibala, "An Analysis of Surface Oxide Softening in Niobium Single Crystals", AIME Annual Meeting, Atlanta, Ga., March 1977.
57. R. Gibala, "Hydrogen-Defect Interactions in Metals", Polish Academy of Sciences, Commemorative Symposium on Contributions of Michal Smialowski, Hydrogen Embrittlement and Corrosion of Metals, Warsaw Poland, May 1977.
58. R. Gibala, "Surface Film Softening in BCC Metals", Max-Planck-Institut fur Eisenforschung, Dusseldorf, West Germany, May 1977.
59. R. Gibala "Effects of Interstitial Distributions on Mechanical Behavior of BCC Metals", Institut fur Festkorperforschung der Kernforschungsanlage, Julich, May 1977.
60. R. Gibala, "Effect of Interstitial Distributions on Mechanical Behavior of BCC Metals", Universitat Gottingen, Institut fur Physik, Gottingen, West Germany, May 1977.
61. R. Gibala, "Recent Experiments and Interpretations on Hydrogen-Defect Interactions in Metals", Max-Planck Institut fur Metallforschung, Stuttgart, West Germany, May 1977.
62. R. Gibala, "Effect of Purity, Prestrain and Cooling Rate on Hydrogen Strengthening in Niobium Single Crystals", Second International Congress on Hydrogen in Metals, Paris, France, June 1977.
63. R. Gibala, "Plasticite des Metaux Cubiques Centres a Basse Temperature" Centre d'Etudes Nuclearies de Grenoble, Grenoble, France, June 1977.
64. R. Gibala, "Effect of Interstitial Distributions on Mechanical Behavior of BCC Alloys", Michigan Technological University, Houghton, Michigan, August, 1977.

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THESES

1. M.G. Ulitchny (M.S. , 1969), "Internal Friction and Strain Aging of Austenite".
2. K.V. Ravi (Ph.D., 1969), "The Strength of Niobium-Oxygen Solid Solutions".
3. G.J. Klems (Ph.D., 1971), "Internal Friction of Ferrous Martensites."
4. M.G. Ulitchny (Ph.D., 1972), "Mechanical Behavior of Interstitial Solid Solutions of Niobium".
5. A.A. Sagues (Ph.D., 1972), "Substitutional-Interstitial Solute Atom Interactions in Ta-Re-N and Ta-Re-O Solid Solutions".
6. J.A. Slane (M.S., 1973), "Carbon-Vacancy Interactions in Austenites".
7. R.P. Krupitzer (M.S., 1973), "Effects of Preferred Orientation on Snoek Phenomena in Commercial Steels".
8. A.K. Vasudevan (Ph.D., 1974), "Solution Softening in Niobium Base Substitutional Alloy Single Crystals".
9. V.K. Sethi (Ph.D., 1976), "Effects of Solute Gradients and Oxide Films on the Mechanical Behavior of Niobium and Tantalum".
10. R. Fournier (M.S., 1977), "Mechanical Behavior of Niobium-Hydrogen Single Crystals".

EFFORT DISTRIBUTION OF THE
PRINCIPAL INVESTIGATOR

During the contract period February 1, 1977 to January 31, 1978, Professor Ronald Gibala, principal investigator for Contract No. AT(11-1)-1676, has allotted his time as follows:

1. February 1, 1977 - May 31, 1977.

30% of these four months was spent on Contract research. 30% of this time was charged to the Contract.

2. June 1, 1977 - August 31, 1977.

75% of this two month period during the summer session was spent on Contract research, and 75% of the time was charged to the contract.

3. September 1, 1977 - January 31, 1978.

25% of these five months of the academic year was spent on Contract research. The full 25% was financed by the Contract.