OBSTACLE-INDUCED HARDENING AND SOFTENING IN METALLIC MATERIALS

Technical Progress Report

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ABSTRACT

During the past contract year, new results have been obtained in two on-going project areas: (1) surface oxide softening and solute gradients in niobium and tantalum, and (2) mechanical properties of Nb-H, Nb-D, and Nb-O-H alloys. The important results include: (a) characterization of the large softening and ductilizing effect of niobium and tantalum caused by thin (50-100 nm) surface oxide films; (b) determination of the role of interstitial impurities in the strengthening and ductility changes caused by hydrogen and deuterium in niobium; and (c) observation of hydride precipitate softening in niobium.

A. Surface Oxide Softening in Niobium and Tantalum (V.K. Sethi)

In previous progress reports we reported that thin surface oxide films reduce the low temperature flow stress of Nb and Ta. For example at 77°K Nb with a 65 nm oxide film has a yield strength of 120 MN/m² compared to 180 MN/m² for pure Nb⁽¹⁾. We also reported that when the oxide-coated metals are prestrained at "athermal" temperatures (295°K for Nb and 450°K for Ta) into stage I of work hardening, the amount of "surface oxide softening" is enhanced. In the past year we have determined the optimum experimental conditions (in terms of oxide thickness and extent of prestrain) to achieve maximum softening.

All experimental techniques for preparing ultra-high purity (interstitial concentration < 10 ppma) single crystals of Nb and Ta oriented for easy glide have been described in previous progress reports, publications, and theses⁽²⁾. Various combinations of oxide thickness ($0 < \delta < 154$ nm) and high temperature prestrain ($0 < \gamma_{295} < 0.20$) were used to find the optimum conditions for maximum surface oxide softening in niobium. The results of our experiments on Nb crystals are summarized in Fig. 1, which illustrates the critical resolved shear stress (τ) at 77°K of oxide-coated Nb as a function of oxide thickness (δ) and prestrain (γ_{295}). Some of the important features are as follows:

(a) τ for oxide-coated Nb for all oxide thicknesses is lower than that of uncoated Nb.

(b) With increasing oxide thickness τ has a minimum at $\delta = 70$ nm.

(c) τ of all oxide-coated Nb specimens is further decreased by prestrain. The reduction is a function of the amount of high temperature prestrain and is maximum for $\gamma_{295} = 0.1$.

(d) Maximum softening (lowest τ) is exhibited by Nb that is coated to $\delta = 70$ nm and is prestrained to $\gamma_{295} = 0.1$. The value of $\tau_{\min}$ so obtained is 60 MN/m²

as compared to 120 MN/m^2 for coated but unprestrained Nb and 180 MN/m^2 for uncoated and unprestrained Nb at 77°K .

Various combinations of oxide thickness δ and high temperature prestrain γ_{450} were used to determine the maximum surface oxide softening in Ta. The results are shown in Fig. 2 as a three dimensional plot of τ at 77°K as a function of δ and γ_{450} . The smallest value of τ at 77°K (80 MN/m^2) is obtained for $\delta = 90 \text{ nm}$ and $\gamma_{450} = 0.075$.

We have proposed a mechanism for surface oxide softening in which the oxide film, which is known to be highly stressed, acts as an efficient source of mobile non-screw dislocations which can move out from the surface over large distances on active glide planes^(1,3). The dependence of τ on δ can be described in terms of the growth stresses which are a function of the film thickness⁽⁴⁾. Growth stresses increase rapidly with thickness for the first 10-50 nm and are relatively independent of thickness for subsequent growth. Hence according to the proposed mechanism, the magnitude of the softening increases with increasing film thickness because the increasing growth stresses facilitate the operation of the surface sources at lower stresses. However the softening competes with the film hardening interactions of the kind observed in fcc metals⁽⁵⁾, which operate in bcc metals as well. The net results of the two effects is a minimum in τ as a function of δ . High temperature prestrain enhances the observed softening by supplying a high initial density of mobile dislocations across the entire glide plane and by creating very fine, wavy, and well-distributed slip steps on the surface of the specimen. The fine slip lines under the influence of growth stresses and applied stress act as sources of mobile dislocations.

The effect of additional variables on surface oxide softening have also been examined: substrate purity; deformation mode (tension, compression, bending); the order in which prestrain and oxide coating are performed⁽²⁾. In addition, microstructural characterization experiments of surface oxide softened Nb have

been performed as follows: slip trace analysis; etch pitting of emergent edge and screw dislocations; transmission electron microscopy of dislocation substructures⁽³⁾. These experiments will be described in detail in subsequent reports and papers.

Mechanical Properties of Nb-H and Nb-O-H Alloys (R. Fournier)

In the last progress report we presented a study of the isotope effect on the mechanical properties of Nb-H alloys at low temperatures $T \leq 295^\circ\text{K}$ as part of a larger program on effects of interstitials on properties of bcc refractory metals. In the past year we have extended this research by examining the effects of three materials parameters:

- i) prestrain at 295°K , which increases the mobile dislocation density and alters the nature of hydride precipitation⁽⁶⁾;
- ii) cooling rate which also influences the precipitate ion behavior⁽⁶⁾;
- iii) addition of a second solute, oxygen, which is known to interact with hydrogen⁽⁷⁾ and to strengthen the matrix^(1,2).

The effects of these parameters are complicated and interrelated. For example, prestraining alters the precipitate size, distribution, and morphology in addition to its effect on mobile dislocation density. The addition of oxygen by its interaction with hydrogen modifies the partitioning of hydrogen among hydrides and clusters⁽⁷⁾ and by its matrix strengthening permits additional stress-induced hydride precipitation at temperatures above the "stress-free" solvus⁽⁸⁾.

In spite of these complications, a number of features can be noted from our results on hydrogen strengthening in Nb-H and Nb-O-H alloys in Figs. 3 and 4, respectively. In these figures, we compare strengthening of Nb and Nb-500 ppma O by 800 ppma H that are (i) either unprestrained or prestrained to $\gamma_{295} = 0.03$ prior to cooling to the test temperature, and (ii) either quenched to the test

temperature by rapid immersion in the cryogenic liquid or slow-cooled ($3^{\circ}\text{K}/\text{min}$) to the test temperature. The findings include:

1. The presence of interstitial impurities in small concentrations enhances hydrogen strengthening in Nb. The various degrees of strengthening achieved by 800 ppma H relative to "pure" Nb given by the four curves labeled $\diamond, \nabla, \circ, \Delta$ in Fig. 3 are small compared to that observed by previous investigators⁽⁹⁻¹¹⁾ who used less pure base metals relative to residual interstitials. This is illustrated in part by inclusion of some results of Chen and Arsenault⁽¹¹⁾ in Figs. 3 and 4 and by the fact that we begin to observe their approximate levels of hydrogen strengthening when oxygen is intentionally added to our purer base metal (Fig. 4).

2. Thermo-mechanical processing (i.e. initial dislocation substructure and cooling rate) have appreciable effects on hydrogen strengthening. Comparison of the curves labeled $\diamond, \nabla, \circ, \Delta$ in Fig. 3 or Fig. 4 show large temperature-dependent differences in critical resolved shear stress (CRSS) depending upon whether the materials are prestrained or not and quenched or slow cooled. The differences are magnified by the presence of interstitial oxygen as evidenced by comparison of Figs. 3 and 4. Detailed interpretation of these effects are being made in terms the manner in which the thermo-mechanical treatments alter hydride particle size, distribution and morphology⁽¹²⁾.

3. Hydrides can cause "precipitate softening" in Nb-H alloys. The strengthening by hydrogen in unprestrained and slow-cooled Nb-H and Nb-O-H alloys ($\diamond$ in Figs. 3 and 4) begins to level off at temperatures below 120°K . It is well-known that this treatment (i.e. slow cooling without the high density of nucleation sites provided by dislocations) results in large incoherent hydrides compared to fine dispersions that quenching and prestrain introduce^(6,13). Such large hydride particles generate substantial numbers of glissile edge-type dislocations loops when they precipitate because of the large misfit ($\sim 12\%$)

with the matrix. In principle, these dislocations can permit deformation at a lower flow stress if the initial density of generated dislocations is sufficient and the density can be increased during deformation by multiplication mechanisms. This process is much like the surface oxide softening discussed in section A, except now the mobile dislocations come from dispersed precipitates.

Such precipitate softening appears to occur also in Nb-H alloys given other treatments. In Fig. 5, we show the behavior of high-purity Nb-H alloys of different hydrogen contents prestrained to $\gamma_{295} = 0.03$ and quenched to 77°K, the test temperature. The CRSS first increases substantially with the first few hundred ppma of hydrogen. The CRSS then decreases with additional hydrogen while the ductility increases and the stress-strain curves are serrated. This behavior is quite similar to that observed for surface oxide softening given earlier reports and papers⁽¹⁻³⁾.

4. "Alloy softening" occurs in Nb-O-H alloys. This observation has been made previously by other investigators^(10,11,14). It is a statement that the component of the CRSS of a Nb-O-H alloy due to interstitials O and H is less than that expected from any additivity law⁽¹⁵⁾ involving the CRSS's of Nb-H and Nb-O alloys. Only cursory data are presented in Figs. 3 and 4 to show alloy softening. At 77°K there is one datum point for Nb-O alloys which is used in Table I to illustrate the occurrence of alloy softening. The important feature that appears from these results is that alloy softening in Nb alloys containing hydrogen is very much dependent upon the thermo-mechanical treatment the material has undergone prior to testing. For the particular data given in Table I, the slow-cooled Nb-O-H alloys exhibit alloy softening, whereas the quenched alloy actually exhibit a synergistic strengthening effect.

All of the features discussed in subsections 1-4 are being pursued in more complete detail. The program will include microstructural characterization of the materials in the proposed contract year.

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TABLE I
Critical Resolved Shear Stresses of Selected
Nb-O, Nb-H, and Nb-O-H Alloys

<u>Material</u>	<u>Prestrain at 295°K</u>	<u>CRSS(MN/m²)</u>	<u>Δ(CRSS) *</u>
1. Pure Nb	0.03	155	0
2. Nb-500ppma O	0.03	190	35
3a. Nb-800ppma H	0.03(Q) **	215	60
3b. Nb-800ppma H	0.03(SC) **	230	75
4a. Nb-500ppma O-800ppma H	0.03(Q)	270	$\begin{matrix} 115 \\ \text{AS}^{\#} \left(\begin{matrix} 80 \\ 95 \\ 110 \end{matrix} \right) \text{AH}^{\#} \end{matrix}$
4b. Nb-500ppma O-800ppma H	0.03(SC)	235	
5a. Δ (CRSS) ₂ + Δ (CRSS) _{3a}	0.03(Q)	---	
5b. Δ (CRSS) ₂ + Δ (CRSS) _{3b}	0.03(SC)	---	

* Δ (CRSS) $\equiv$ CRSS(alloy) - CRSS(pure Nb)

** Q = quenched alloy; SC = slow-cooled alloy

AS = alloy softening; AH = synergistic alloy hardening

Fig. 1 Three dimensional view of the effect of prestrain and oxide film thickness on the critical resolved shear stress of niobium at 77°K.

Fig. 2 Three dimensional view of the effect of prestrain and oxide film thickness on the critical resolved shear stress of tantalum at 77°K.

Fig. 3. Temperature dependences of the critical resolved shear stress of Nb and Nb-H alloy single crystals.

Fig. 4. Temperature dependences of the critical resolved shear stress of Nb and Nb-H-O alloy single crystals.

Fig. 5. Resolved shear stress-shear strain curves for high purity Nb-H alloys quenched to the test temperature.

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A SUMMARY OF REPORTS SUBMITTED TO
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Report No.

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C00-1676-2	R. Gibala, Comments on the Cold-Work Peak in Alpha Iron, October 31, 1967.
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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".

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

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.
30. M. G. Ulitchny, A.K. Vasudevan and R. Gibala, "Solution Hardening and Softening in Nb-Base Solid Solutions", Third International Conference on the Strength of Metals and Alloys, Cambridge, England, August, 1973.
31. A.A. Sagues and R. Gibala, "Crystallographic Orientation Dependence of Interstitial Anelasticity in Ta-Re-N and Ta-Re-O Alloys", Fifth International Conference on Internal Friction and Ultrasonic Attenuation in Crystalline Solids, Aachen, Germany, August, 1973.
32. G.J. Klems, R.E. Miner, F.A. Hultgren and R. Gibala, "Internal Friction in Ferrous Martensites", Fifth International Conference on Internal Friction and Ultrasonic Attenuation in Crystalline Solids, Aachen, Germany, August, 1973.
33. M.G. Ulitchny and R. Gibala, "Mechanical Properties of Nb Single Crystals Containing H and H+O", International Conference on "Hydrogen in Metals: Effects on Properties, Selection and Design", Seven Springs, Champion, Pa., September, 1973.
34. A.K. Vasudevan and R. Gibala, "Adoucissement par Alliage du Niobium par Addition de Molybden et Oxygene", Colloque Plasticité, Toulouse, France, March, 1974.
35. R. Gibala, "Dislocation Relaxation in BCC Metals", Max-Planck Institut fur 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 Mechanique et Physique des Materiaux, 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 Federale 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.

42. R. Gibala, "Anelastic and Magnetic Relaxation in Ni-Co Alloys", Case Western Reserve University, February, 1975.
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. Krupitzor 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.
48. V.K. Sethi and R. Gibala, "Effect of Oxide Coatings on the Mechanical Properties of Niobium Single Crystals Deformed at Low Temperatures", NATO Advanced Study Institute on Surface Effects in Crystal Plasticity, Hohegeiss, W. Germany, September, 1975.
49. V.K. Sethi and R. Gibala, "Low Temperature Mechanical Behavior of Oxide-Coated Single Crystals of Niobium and Tantalum", ASM/AIME Materials Science Symposium, Cincinnati, Ohio, November, 1975.
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.

EFFORT DISTRIBUTION OF THE
PRINCIPAL INVESTIGATOR

During the contract period February 1, 1976 to January 31, 1977,
Professor Ronald Gibala, principal investigator for Contract No.
AT(11-1)-1676, has allotted his time as follows:

1. February 1, 1976 - May 31, 1976.

40% of these four months was spent on Contract research. 35% of this time
was charged to the Contract and 5% was supported by CWRU.

2. June 1, 1976 - August 31, 1976.

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

3. September 1, 1976 - January 31, 1977.

30% of these five months of the academic year was spent on Contract research.
The full 30% was financed by the Contract.