DISLOCATION-SOLUTE ATOM INTERACTIONS IN ALLOYS

Technical Progress Report

R. Gibala, V.K. Sethi and R. Fournier

Case Western Reserve University
Department of Metallurgy and Materials Science
10900 Euclid Avenue
Cleveland, Ohio 44106

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ABSTRACT

During the past contract year, new results have been obtained in two major project areas: (1) surface oxide softening and solute gradients in niobium and tantalum, and (2) mechanical properties of Nb-H and Nb-D alloys. The important results include: (a) observation and characterization of an extremely large softening and ductilizing effect of body-centered cubic metals (niobium and tantalum) by application of thin (500-1000^oA) oxide films coupled with injection of a high density of mobile dislocations; (b) determination of the relative strengthening and ductility changes caused by hydrogen and deuterium in niobium.

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1. Surface Oxide Softening and Solute Gradients in Niobium and Tantalum
(V.K. Sethi)

In previous progress reports, we have shown that the macroscopic flow stress of an alloy of a high purity bcc metal, for a fixed concentration of alloying element, is a strong function of the manner in which the solute is distributed. For example, by altering the shape of the concentration profile of oxygen in a niobium-oxygen alloy, the flow stress can be varied in the range $\sim 16 \text{ kg/mm}^2$ to $\sim 30 \text{ kg/mm}^2$ at $77^\circ\text{K}^{(1)}$. During the course of these investigations, it was discovered that the extreme gradient i.e., when all the oxygen is present on the surface in the form of an oxide layer, has a very large effect on the low temperature mechanical behavior of niobium. Specifically, very thin oxide coatings significantly reduce the yield and flow stress at low temperatures, $T < 300^\circ\text{K}$. We have termed this effect "Surface Oxide Softening" (SOS)⁽²⁾ and it refers to the fact that a surface oxide film which is only a few hundred angstroms thick can reduce the flow stress of niobium by as much as 70% at low temperatures. In this section, we describe the progress of a research program initiated during the past year to study and characterize surface oxide softening and to determine the mechanisms involved.

Experimental techniques for preparing ultra-high purity (interstitial concentration $< 10 \text{ ppm}$) single crystals of niobium and tantalum oriented for easy glide have been described in previous progress reports and publications. Oxide films are deposited by anodic oxidation as described previously^(3,4). The oxide thickness is a linear function of anodizing voltage and one volt corresponds to an added oxide thickness of $\sim 22 \text{ \AA}$ for niobium and $\sim 15 \text{ \AA}$ for tantalum⁽⁵⁾. Oxide coated samples are tested at dif-

ferent temperatures in tension on a floor model Instron machine at a base shear strain rate of $\sim 6 \times 10^{-4}$ /sec.

Fig. 1(a) shows the resolved shear stress-shear strain curves for various samples of niobium with different oxide thickness tested at 77°K. Oxide films affect the mechanical behavior in two ways: (a) the flow level is considerably reduced, e.g., the 2% resolved shear stress ($\tau_{2\%}$) of a sample with $\sim 650\text{\AA}$ thick oxide film is $\sim 12\text{kg/mm}^2$ as compared to $\sim 18\text{kg/mm}^2$ for pure niobium; (b) the flow is serrated over a considerable range of strains. The inset in fig. 1(a) shows the effect of oxide thickness on $\tau_{2\%}$, viz. a pronounced minimum is observed for an oxide thickness of $\sim 650\text{\AA}$. Similar results are obtained for tantalum and are shown in fig. 1(b).

When oxide coated samples are prestrained at an elevated temperature (300°K for Nb and 450°K for Ta) into stage I of work hardening, the serrations disappear, the flow stresses are further reduced and the ductility, measured as the extent of uniform elongation, is increased. Fig. 2 shows the resolved shear stress-shear strain curves for niobium samples with $\sim 650\text{\AA}$ thick oxide films (corresponding to the minimum observed in Fig. 1(a)), and prestrained $\sim 10\%$ at 300°K and then tested at various temperatures below 300°K. The flow stress is remarkably low, e.g. $\sim 6\text{ kg/mm}^2$ at 77°K. These results are very dramatic, especially in the light of the fact that more than 99.95% of the specimen volume is pure metal, and pure niobium is generally regarded to have a Peierls-Nabarro lattice friction stress of the order $16\text{--}20\text{ kg/mm}^2$ at 77°K. Also, a three stage work hardening behavior, usually not observed at temperatures much below 300°K, is observed at temperatures as low as 77°K. Similar behavior is observed for oxide coated ($\sim 900\text{\AA}$) and prestrained ($\sim 8\%$ at 450°K) tantalum samples at the same low temperatures. The τ - γ curves are shown in fig. 3.

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There is a great deal of evidence that plastic flow of high purity bcc metals at low temperatures is best explained by a concept of two different types of dislocations governing flow in the micro-strain and macro-strain regions⁽⁶⁾. Edge and other non-screw dislocations are highly mobile and are exhausted at small strains; screw dislocations are characterized by a large Peierls-Nabarro stress due to their non-coplanar dissociated core structures⁽⁷⁾, and control the macro-flow behavior. Immobility of screw dislocations in the bcc lattice at low temperatures leads to the strong temperature dependence of the flow stress that is normally observed. Fig. 4 shows the temperature dependence of the flow stress of niobium, oxide-coated and prestrained niobium, tantalum, and oxide-coated and prestrained tantalum. The temperature dependence of the flow stress of oxide-coated and prestrained niobium and tantalum is considerably smaller than that of pure metals, indicating that rate controlling mechanism has been completely altered.

Given the picture for flow of the bcc metals involving immobile screw dislocations, we have postulated⁽²⁾ that the oxide film, which is known to be highly stressed⁽⁸⁾, or the oxide-metal interface acts as an efficient source of large numbers of mobile non-screw dislocations which can move out from the surface over reasonably large distances on active glide planes. With a large number non-screw dislocations already available to carry deformation as a result of prestrain and their numbers continuously generated at the oxide-metal interface, the large macro-flow stress dictated by the Peierls stress of screw dislocations can be postponed until the depletion of sources. The serrated flow observed in the unprestrained material suggests that the proposed dislocation sources are stress activated inhomogeneously.

2. Mechanical Properties of Nb-H and Nb-D Alloys

(R. Fournier)

The investigation of the mechanical properties of Nb-H and Nb-O-H alloys has been undertaken in the past by our group ⁽⁹⁻¹¹⁾. The basis for this research has been the natural importance of hydrogen in refractory metals which are intended for reaction applications, e.g. Nb-Zr alloys first-wall materials in fusion reactors. Because hydrogen, hydrogen isotopes, and helium can be produced in concentrations amounting to hundreds of atomic ppm per year in fusion reactors, it seemed a natural and desirable first extension to examine the mechanical behavior of the Nb-D system. This is particularly true when one notes that there are important isotope effects in niobium relating to diffusion and interaction with oxygen ⁽¹²⁾, but not with solubility ⁽¹³⁾.

In the past year, we have re-examined our own work on hydrogen strengthening in niobium by employing a higher purity base material and have added equivalent results on deuterium in the same material. Some of these results are given in figs. 5 and 6 in the form of stress-strain curves at different test temperatures for one particular concentration of hydrogen or deuterium, 400 ppma. Each test was preceded by 3% prestrain at 300°K to aid in characterizing the material purity and to eliminate changes in mobile dislocation density from specimen to specimen as a variable. Figs. 5 and 6 show that the deformation behavior of the hydrogen and deuterium alloys is quite similar at all temperatures, including the occurrence of serrations at 77°K. These results are expected if we note the lattice parameter, the resistivity increment and solubility of hydrogen and deuterium in niobium are about the same ⁽¹³⁾.

The mechanism of strengthening of niobium by hydrogen and deuterium depends critically upon the temperature of deformation ^(9,14). A plot of the flow stress as a function of temperature for previous results ^(9,14) and the present studies is shown in fig. 7. Observable differences in the magnitude of and the

trend in the flow stress exist from investigation to investigation. Determinations of the nature and extent of these differences are being examined as part of this work. To be noted in fig. 7 are:

(i) The presently obtained flow stresses are smaller than those obtained by Ravi and Gibala⁽⁹⁾, Ulitchny and Gibala⁽¹⁰⁾ and Chen and Arsenault⁽¹⁴⁾.

(ii) Our results do not exhibit a maximum in the temperature dependence of the flow stress in either the hydrogen or the deuterium alloys that other investigators observe.

A difference in the purity of the base material may account for both results (i) and (ii); specific reasoning would primarily involve the interaction of hydrogen and deuterium with other interstitial impurities. The stress-induced reorientation of hydrogen in hydrogen-impurity clusters is in principle capable of producing a strengthening increment which may exhibit a maximum in the temperature dependence, since a mobile defect is involved^(9,11).

Hydrogen or deuterium embrittlement is evident in the temperature dependence of the ductility for both alloys, fig. 8. The data presented in fig. 8 is consistent with the fact that hydrides and deuterides have the same structure and lattice parameter⁽¹³⁾, but that a definite diffusional isotope effect exists⁽¹⁵⁾.

This study is approaching completion and includes in its remaining work:

(i) obtaining additional values of flow stress of Nb-H and Nb-D alloys at temperatures between 4.2°K and 300°K, mainly in the range 4.2°K to 77°K;

(ii) determining the hardening of niobium for different concentrations of hydrogen and deuterium;

(iii) investigating the effect of third components (e.g. oxygen) on the mechanical behavior of Nb-H and Nb-D alloys.

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Fig. 1 Effect of anodic oxide coatings on the mechanical behavior of niobium and tantalum single crystals at 77°K

Fig. 2 Resolved shear stress-shear strain curves for niobium single crystals oxide coated ($650\text{\AA}$) and prestrained 10% at 300°K

Fig. 3 Resolved shear stress-shear strain curves for tantalum single crystals oxide coated (900Å) and prestrained 8% at 450°K

Fig. 4 Effect of oxide coating and prestrain on the temperature dependence of the yield stress of niobium and tantalum single crystals

Fig. 5 Shear stress-strain curves for niobium-hydrogen single crystals

Fig. 6 Shear stress-strain curves for niobium-deuterium single crystals

Fig. 7 Temperature dependence of the resolved shear stress of niobium, niobium-hydrogen and niobium-deuterium single crystals

Fig. 8 Temperature dependence of the ductility of Nb-H and Nb-D alloys

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A SUMMARY OF REPORTS SUBMITTED TO
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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.
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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.
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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.
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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 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.
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40. R. Gibala, "Interstitials in BCC Metals", General Electric Company, Lamp Division, Cleveland, Ohio, October, 1974.
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During the contract period February 1, 1975 to January 31, 1976, Professor Ronald Gibala, principal investigator for Contract No. AT(11-1)-1676, has allotted his time as follows:

1. February 1, 1975 - May 31, 1975.

Approximately 40% of these four months was spent on Contract research. 20% of this time was charged to the Contract and 20% was supported by CWRU.

2. June 1, 1975 - August 31, 1974.

100% of this two month period (officially) during the summer session was spent on Contract research, and 100% of the time was charged to the contract.

3. September 1, 1975 - January 31, 1976.

40% of these five months of the academic year was spent on Contract research. Of this amount, 20% has been financed by the Contract and 20% was supported by CWRU.