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MECHANICAL PROPERTIES OF METAL HYDRIDES

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TECHNICAL REPORT

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ABSTRACT

The role of metal hydrides in the hydrogen embrittlement of a number of metal-hydrogen systems is reviewed. The stress induced formation and cleavage of hydrides at crack tips and the thermodynamics of the hydrides in the stress field of the crack tip are discussed.

The mechanical response of the hydrides to applied stresses is described. Many of the hydrides exhibit some plastic deformation under favorable stress systems with the amount of this deformation increasing as the temperature of testing is increased. Phonon properties and the elastic moduli of the hydrides are briefly discussed and it is suggested that these parameters do not provide a basis for understanding the mechanical properties of the hydrides. The deformation response of the hydrides is consistent with the expected behavior of dislocations in the ordered hydride structures.

INTRODUCTION

The mechanical properties of the metal hydrides have not been extensively investigated despite the fact that there exists compelling scientific and engineering reasons for doing so. The response of the hydrides to an external stress is of importance in the design of systems which use hydrides for storage of hydrogen (energy storage, compressors, etc.) as these often depend on the creation of fresh surfaces for the activation of the hydrides and have to contend with the dimunition of the hydride particle size as the hydrogen is cycled in and out of the system. The mechanical properties of the hydrides also play a central role in the hydrogen embrittlement of alloy systems based on the hydride forming metals such as the group Vb metals, Nb, V, Ta, alloys based on Ti, Mg, Zr as well as others which have more specialized applications. In all of these applications and alloy systems, the general expectation is that the hydride will be "brittle" and in general this expectation is in accord with casual observations. Very few hydrides have been carefully studied wih respect to their mechanical properties and in fewer still are the reasons for the mechanical response understood.

In the present paper we will review the available information on the mechanical properties of the metal hydrides and will discuss some of the reasons for their limited ductility.

ROLE OF HYDRIDES IN HYDROGEN EMBRITTLEMENT

The mechanism of hydrogen embrittlement in those systems which form stable hydrides and which are stressed under conditions of temperature and hydrogen fugacity such that hydrides can form is relatively well understood and has been recently reviewed (1,2). It appears that the behavior of these systems can be completely understood based on a mechanism in which stress

induced hydride forms at stress concentrations such as crack tips and the cracking of the hydrides is followed by repeated stress induced hydride formation and fracture (3). Embrittlement results from the formation of the second phase hydride which is assumed to be a "brittle" phase. As discussed by a number of investigators (4-7), this mechanism is applicable to temperature and hydrogen concentration regimes in which the hydride is thermodynamically stable as well as those in which hydrides are not observed in the absence of an external stress. In the latter case hydride formation can be stabilized by an external stress. Even in the regime where stable hydrides form, the precipitated hydrides merely act as crack nuclei; propagation of the "brittle" fracture requires the repeated stress induced formation and fracture of the hydrides at the crack tip. In the absence of such continued hydride formation, the precipitated hydrides act as any other brittle phase and while the ductility is reduced, the fracture between the hydrides is by a ductile rupture mechanism.

Hydride thermodynamics in an externally applied stress field has been extensively discussed in the literature (4-11). Non-uniform stresses, such as those which exist at the tips of cracks, can have significant effects on the distribution of hydrogen in solid solution as well as on the stability of hydrides, while uniform stresses have relatively small effects (11). At a stress concentration in a tensile stress field, the chemical potential of hydrogen in a uniform solid solution is decreased relative to the unstressed volume as a result of the positive volume of solution of the hydrogen. Since the volume change on forming the hydride from the solid solution is generally positive, the chemical potential of the hydride is also reduced relative to the solid solution with which it is in equilibrium at the crack tip. (In addition to the free energy changes associated with the volume change, terms

due to the shear stresses and shape changes are also significant.) As a result of these terms, the concentration of hydrogen in the bulk which results in precipitation of the hydride at the stress concentration is given by (11):

$$\ln \; C_H^S = \eta + \frac{\Delta H_{sol}}{RT} - (\frac{\sigma_S}{RT}) \{ V_\beta - (V_m + C_H^\beta V_H) \} / (C_H^\beta - C_H^S) \qquad (1)$$

where C_H^S and C_H^β are the solvus and hydride concentrations respectively, η is a constant, ΔH_{sol} is the enthalpy of the stress free solvus (which is generally < 0 in hydride forming systems), σ_S is the spherical component of the local stress field, V_β, V_m and V_H are the molal volumes of the hydride, the molal volume of the metal and the partial molar volume of hydrogen in the solid respectively, R is the gas constant and T is the temperature. In addition to these terms, the formation of the hydride is constrained by the solid solution matrix and the volume increase on forming the hydride is accommodated by elastic and plastic deformation of the matrix. The plastic accommodation free energy terms can be decreased by the applied stresses as these assist the formation of prismatic loops and other accommodation dislocation structures (5,7,10). An estimate of the magnitude of this free energy term, which results from the constraint of the hydride volume change, has been obtained for the Nb-H system by calculating the total elastic free energy necessary to insert a hydride into the equivalent "hole" in the solid solution under the assumption of a spherical hydride morphology (10). The value is

$$\Delta G_{\alpha - \beta}^{elastic} + \Delta G_{\alpha - \beta}^{plastic} = \frac{2\mu}{3\alpha} \frac{\Delta V_{\alpha - \beta}^2}{V_\alpha} \approx 3100 \; J/mole \qquad (2)$$

where $\alpha = 1 + 4\mu/B$, μ is the shear modulus, B is the bulk modulus, $\Delta V_{\alpha - \beta}$ is the molar volume change on forming the hydride, and V_α is the molar volume of

the solid solution. The lower bound of the bulk solid solution hydrogen concentration which can lead to hydride precipitation under stress at an elastic singularity may therefore be written:

$$\ln C_H^S = \eta + \frac{\Delta H_{sol}}{RT} - (\frac{\sigma_s}{RT})\{V_\beta - (V_m + C_H^\beta V_H)\}/(C_H^\beta - C_H^S)$$

$$- (\frac{2\mu}{3\alpha})(\frac{\Delta V_{\alpha-\beta}^2}{V_\alpha}) \quad .$$

(3)

In the case of platelike or lenticular hydrides, such as in the V-H system, the magnitude of the last term in eqn. 3 is reduced.

As a result of these considerations, the reported phase diagrams for the metal hydrogen systems should not be considered true equilibrium diagrams. The phases which form are constrained by the matrix in which the precipitation takes place and the true equilibrium phase boundaries, i.e. those which would pertain to the equilibrium between phases which are not constrained in their shape or volume changes, generally lie above those commonly observed. While the effects of uniformly aplied external stresses on the phase diagram are generally small, quite appreciable changes in the phase equilibria can be observed in regions of high non-uniform streses (5,11). These considerations apply to all systems but are particularly significant in the metal hydrogen systems as the free energies of the hydride phase transitions are quite small and stress related free energy terms resulting from the large volume and shape changes can be comparable. An example of these effects is shown in Fig. 1 which shows the precipitation of the hydride in the Nb-H system in front of a stressed notch. This precipitation was observed at temperatures above the normal solvus temperature at which general hydride precipitation occurred in the bulk of the specimen.

Using the above concepts, the equilibrium phase boundaries can be estimated and are shown in Fig. 2 for the Nb-H system. The area between the observed phase boundaries and those which are estimated as the equilibrium boundaries is the region in which stress induced hyride formation may occur at crack tips. In hydride forming systems hydrogen embrittlement has been observed (4-6,12) to result from stress induced hydride formation in region of the phase diagram which are nominally single phase solid solutions. An example of the drastic nature of this effect is shown in Fig. 3 which shows the fracture surfaces of two specimens having almost the same composition in the solid solution range of the Nb-H system. The lower composition specimen failed by ductile rupture with extremely high ductility while at a slightly higher composition, fracture was extremely brittle and resulted from the stress induced formation of the γ hydride (NbH_2) (6).

Hydrogen embrittlement by this stress induced hydride formation and fracture at the crack tips has been directly observed in the V-H (13,14), Nb-H (4-6), Ti-H (9-15), and Zr-H (16) systems.

MECHANICAL PROPERTIES OF HYDRIDES

While the folklore of hydrides suggests that they are brittle materials few studies of their mechanical properties have been carried out. In those cases which have been studied, there generally has been some evidence for plastic deformation in compression, particularly at temperatures above 300 K, but the response to a tensile stress has generally been a brittle fracture. Information is available only on a few binary hydrides.

Beevers and Barraclough (17,18) have been shown that the tetragonal ϵ phase of the Zr-H system in the composition range of $ZrH_{1.71}$ to $Zr_{1.92}$ fractured without significant plastic deformation at temperatures below 295 K

and exhibited increasing plasticity when tested in compression as the temperature was increased. While some slip deformation was noted at the lower H/Zr concentrations, the principal deformation process was the displacement of the twin related domain boundaries and the formation of new twin related domains. These results were confirmed by Ryzhov et al. (19,20) who also reported slip on the $\{111\}$ <110> system and increasing amounts of deformation up to temperatures as high as 1173 K. The creep deformation of $YH_{1.92}$ was also noted at elevated temperatures (21) and the activation enthalpy for creep was about equal to that of H diffusion in the yttrium hydride. In the case of the ε $ZrH_{1.85}$ phase the creep activation enthalpy was somewhat higher than for H diffusion.

Deformation of the titanium hydrides having a fluorite structure ($TiH_{1.53}$-$TiH_{1.80}$) and a tetragonal structure ($TiH_{1.91}$ and $TiH_{1.99}$) was shown to occur by slip on the $\{111\}$, $\{110\}$ and $\{100\}$ planes (22). These hydrides exhibit appreciable plasticity when tested in compression above 238 K; about 2% strain being attained prior to microcracking at the lower temperatures and about 15 to 30% being attained at 423 K. At all compositions, the yield and fracture stresses decreased as the hydrogen jump frequency increased indicating a correlation between the deformation characteristics and the hydrogen diffusivity.

Less extensive investigations have been made of the behavior of the Group Vb hydrides under stress. The β f.c. orthorhombic hydride of niobium was shown to exhibit some plastic deformation at about 300 K by the motion of the boundaries between the twin related domains which form as a result of the transformation from the solid solution (10). No evidence was obtained for slip in this hydride and the specimens fractured in a completely brittle manner when tested in tension at temperatures below 300 K (23). The cleavage

planes were the {100} and {110} (referred to the cubic axes). Electron channel pattern studies of the cleavage planes showed that the fracture was accompanied by very little local deformation. Studies of the plasticity which accompanies the formation of the β hydride in the α solid solution matrix indicated that very little dislocation generation occurs in the hydride either during precipitation or during dissolution (24) while the surrounding solid solution is heavily deformed. This again indicated the high resistance of the hydride to dislocation motion. The fracture surface energy was determined f′ the β NbH$_{0.82}$ to be 4.59 J/m^2 for {110} and to be 6.21 J/m^2 for {100} cleav؛ planes (23). These values are within a factor of two of the surface energy values for the b.c.c. metals (23) and confirm that significant plastic deformation did not take place during the fracture process.

The only other report of the behavior of the Group Vb hydrides is for the β V$_2$H which was reported to exhibit brittle fracture along the {110} domain boundary planes when tested in a multidomain specimen. A single domain crystal was reported to be "ductile" at room temperature when tested in compression (25).

DISCUSSION

Both theoretical and experimental studies have shown that the hydrides have significantly different band structures than the metals from which they are formed (26-18). In a number of hydrides this results in a change of the transport properties from metallic to ionic or semiconducting. In the few cases for which data are available the change does not appear to reflect itself in a significant difference in the atomic bonding as measured by "small strain" measurements such as phonon dispersion measurements (29,30) or elastic constant determinations (31). Increased phonon frequencies and atomic force

constants are observed for both the high concentration solid solutions and the hydrides which form from these solid solutions by hydrogen interstitial ordering (29,30). These effects are particularly significant in view of the large lattice expansion observed on adding H to the metal to form the alloys and which would be expected to lead to a decrease in the force constants. The hydrogen local mode energies increase on going from the solid solution α' to the β hydride phase in the Nb-H system suggesting an increase in the Nb-H potential as a result of the H ordering (32).

Measurements of the elastic moduli in the solid solution $\alpha-\alpha'$ phase up to H/Nb values of about 1 (33) do not suggest any decrease in the atomic bonding despite the large volume increase on alloying with H. This is shown in Fig. 4 which shows the variation of the bulk modulus with H/Nb. Similarly, changes in the elastic moduli have been determined during the α' to β phase transition for $NbH_{0.78}$ and these measurements show an increase in the moduli on H ordering (31) (Fig. 4).

The available data do not suggest any reason for the limited ductility of the hydride phases based on changes in the atomic bonding. Indeed the fracture energy measurements (23) suggest that the reasons for the limited ductility do not involve changes in the inherent bonding characteristics. Since the plastic response of the hydrides results from the motion of domain boundaries as well as from dislocation motion, at least two deformation mechanisms must be considered. It will be seen that both of these result in limited plastic strain as a result of the ordered arrangements of the hydrogen interstitials.

Hydrides which generally have symmetries lower than that of the solid solutions with which they are in equilibrium contain domains which correspond to the different possible ordering arrangements of the interstitial H

solutes. These domains are twin related (34,35) with the magnitude of the twinning shear depending on the appropriate lattice parameters. These twin boundaries have been observed to be mobile under stress and to result in a limited amount of deformation as a result of the twinning shear (10). In addition, new twin variants can form under the applied stress. Consideration of the twinning shears in hydrides suggests that the magnitude of the strain by this twinning mechanism is limited to a few percent.

Several of the hydrides appear to exhibit larger plasticity than could result from twinning and in these cases the deformation appears to result from thermally activated dislocation motion. The observed slip planes correspond to the close packed planes and the slip directions to the close packed directions in the metal sublattice. However, the extent of the dislocation motion is very limited at low temperatures and domain boundaries appear to inhibit the dislocation motion. It has been suggested (23) that this limited dislocation mobility results from the fact that the usual dislocation Burgers vectors, which correspond to the lowest energy dislocations in the metal sublattice and to the dislocation structures which were inherited from the parent solid solutions, are not lattice translation vectors for the hydride structures. For example, if the total symmmetry of the lattice is considered, half of the $a/2$ <110> Burgers vectors in the f.c. orthorhombic β NbH structure and in the monoclinic β V_2H structure do not correspond to lattice translation vectors. Therefore, dislocation motion on half of the low energy slip systems in these structures will lead to disordering of the H sublattice and hence to a high friction stress. For the β NbH structure, the magnitude of this friction stress has been estimated to be about 90 MPa or $\mu/400$ (23). Similar considerations apply to all of the other low symmetry hydride structures in which the slip systems are similarly limited by the H sublattice ordering.

Dislocation motion on those slip systems which disorder would be possible if the dislocations moved in pairs of $a/2$ <110> Burgers vectors separated by order faults. These do not seem to be observed (24,35).

The limitation on the number of active slip systems results in an inability to blunt sharp cracks and hence stress concentrations in the hydrides are less likely to be reduced by local plastic deformation. In tension crack propagation and brittle fracture will occur. In compression some dislocation motion on those slip systems which do not lead to hydrogen disordering will be observed prior to fracture. The amount of slip on those systems will however be severely limited since cross slip of the dislocations leads to disordering on the cross slip planes. Furthermore, the Burgers vectors which lead to conservative dislocation motion will vary between domains due to the different H sublattice ordering. Consequently, even those dislocations which are mobile in a particular domain will have their motion limited by the presence of the domain boundaries; as has apparently been observed in the case of the V_2H hydrides (25). In single domain crystals this limitation does not apply and more extensive dislocation motion on those systems which do not disorder should be observed.

Since dislocation motion in the hydrides is limited by disordering of the H sublattice, the friction stress will vary with the temperature as does the H diffusivity. This is in agreement with the observations made in TiH_2 (22).

In a qualitative sense, the above considerations based on the crystallography of the hydrides and the effects of dislocation motion on the hydrogen ordering can account for all the available observations. Further understanding will depend on a) direct observation of dislocation structures in the hydrides, b) measurements of the temperature dependence of the deformation, and c) studies of the variation of the dislocation mobility with

particular crystallographic directions in single domains. It does appear however that the limited ductility results from the dislocation structure rather than from some intrinsic property of the atomic bonding in the hydrides.

ACKNOWLEDGEMENTS

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Figure Captions

Fig. 1. Hydride precipitation along slip bands in front of a stressed notch. $NbH_{0.0094}$ specimen at 126 K. The tensile stress axis was horizontal. The region away from the notch contains homogeneously nucleated hydrides (Ref. 5).

Fig. 2. —————   Phase boundaries obtained for bulk alloys.

— · — T_e^0, $\alpha'-\gamma$ (NbH_2); phase boundary for the formation of unconstrained γ. (See Ref. 10.)

- - - - T_e^σ, $\alpha'-\gamma$ (NbH_2); phase boundary for the formation of unconstrained γ under a tensile spherical stress component of $\mu/30$. (See Ref. 10.)

- - - - T_e^σ, $\alpha-\beta$ (NbH); phase boundary for the formation of unconstrained β under a tensile spherical stress
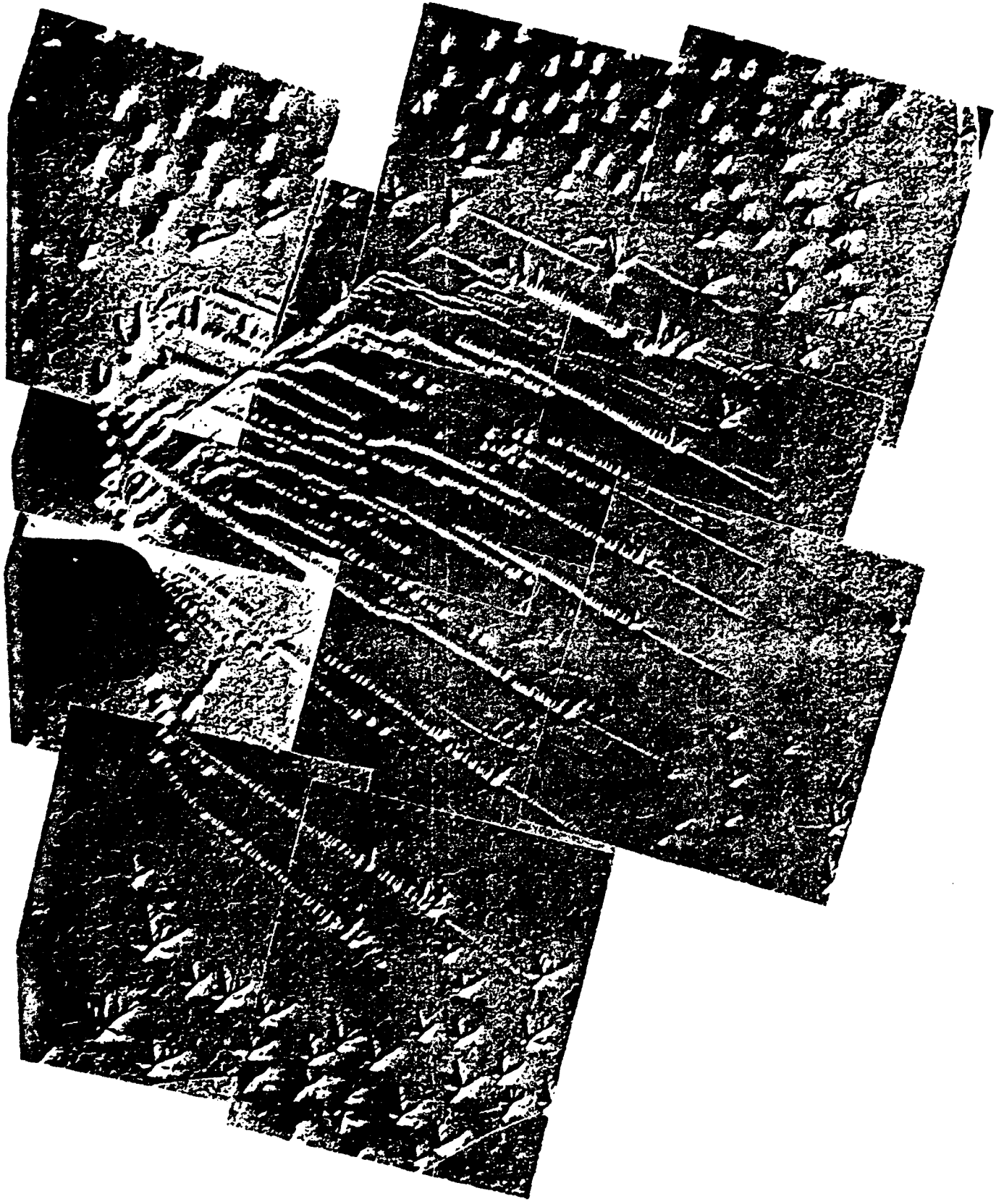
component of $\mu/30$. See Ref. 10.)

$- - - - -$ T_s^σ, observed solvus; experimentally observed boundary between ductile and brittle fracture behavior (Ref. 6). This corresponds to the phase boundary between the α' and the stress induced γ phase.
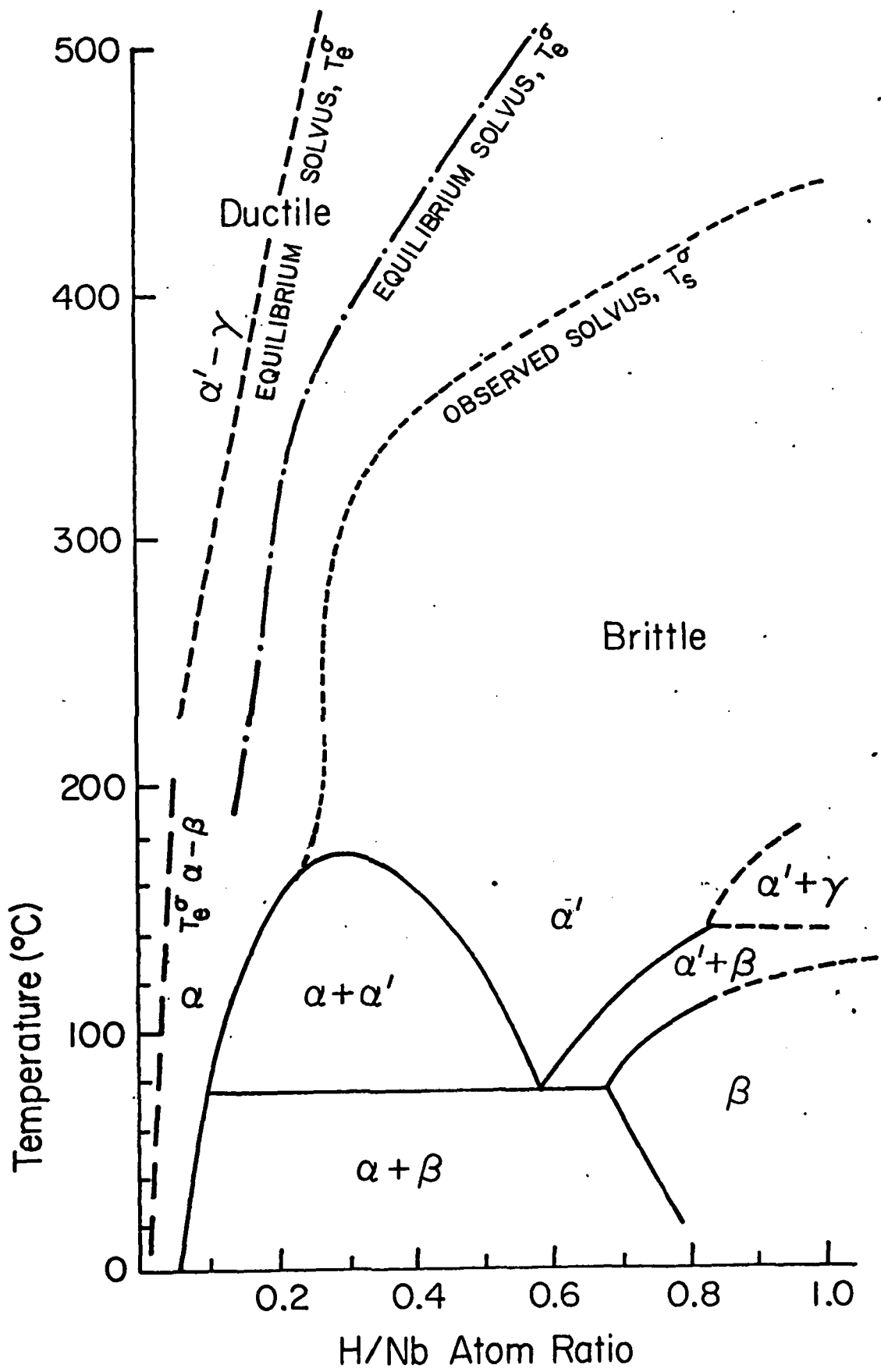
Fig. 3. Fracture surfaces of Nb-H alloys tested at 573 K.

A. $NbH_{0.26}$. Fracture was completely ductile. The fracture occurred along the line indicated by the arrows after severe necking. Strain to failure was 17%.

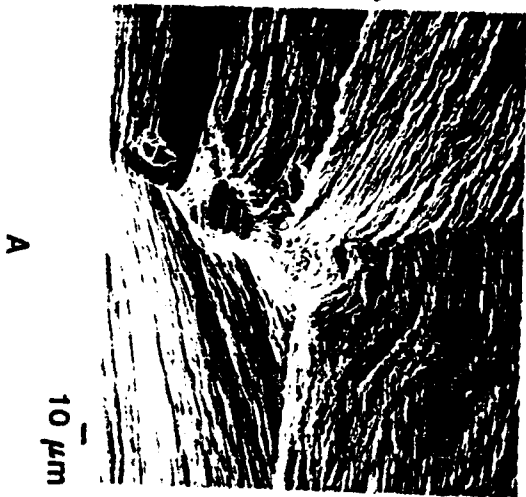B. $NbH_{0.30}$. Fracture was by cleavage after a strain of 18%.

Fig. 4. A. Variation of the bulk modulus with temperature near the α'-β phase transition.
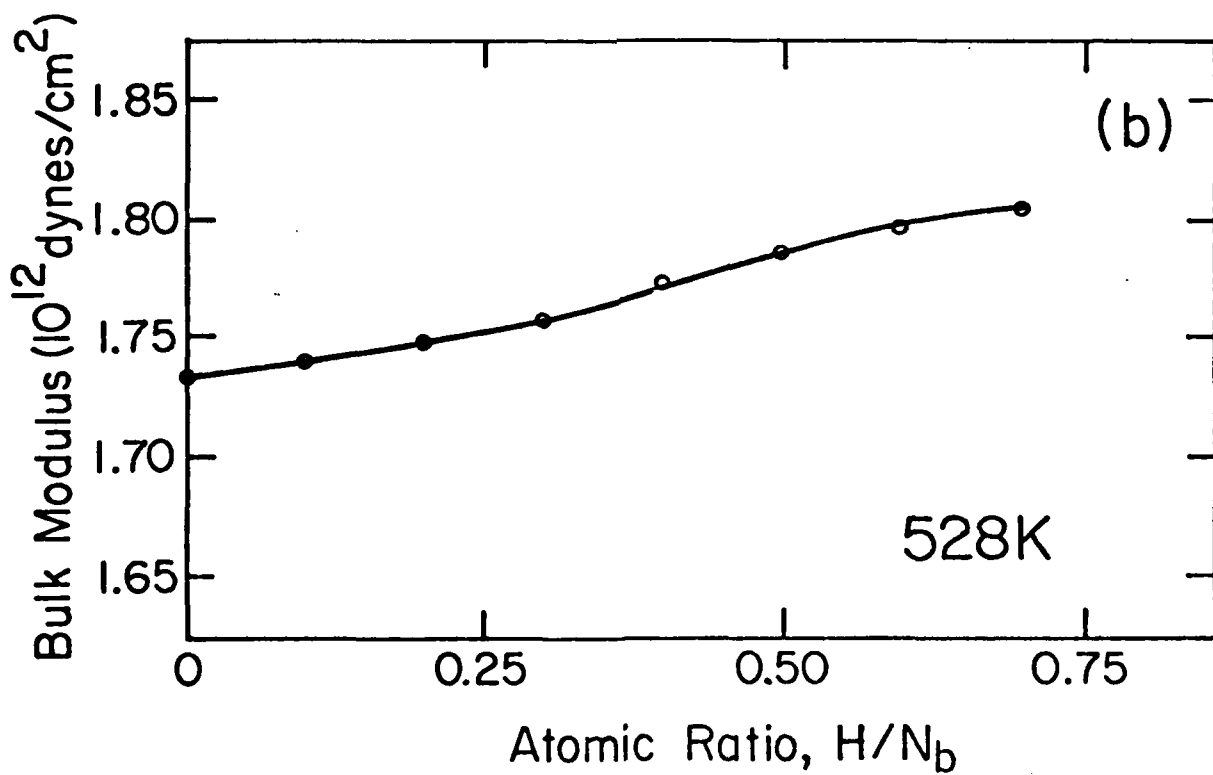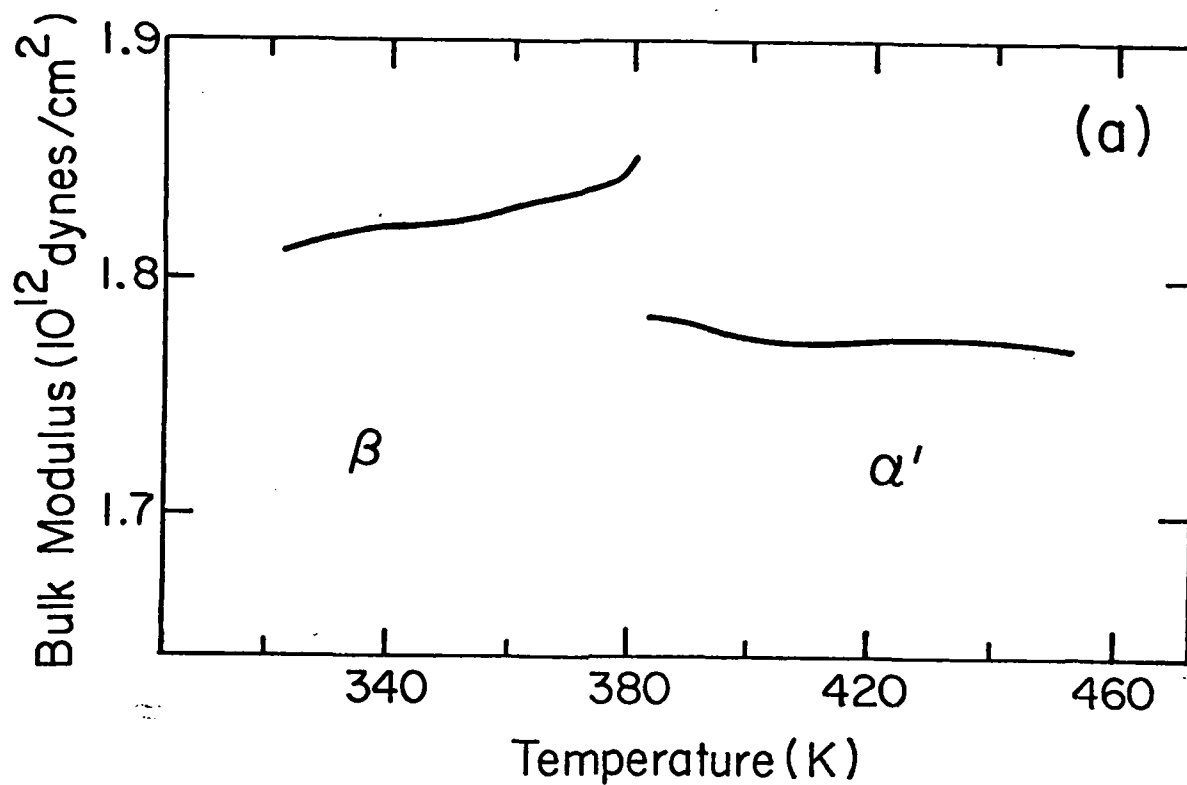
B. Variation of the bulk modulus with H/Nb ratio at 528 K.

Birnbaum Fig. 2

A

10 μm

B

100 μm

Birk... Fig 4

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