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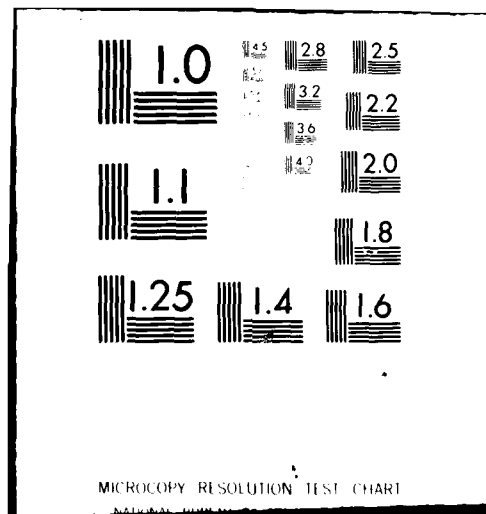
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Research Department
Grumman Aerospace Corporation
Bethpage, New York 11714

March 1981

Final Technical Report
1 January 1976 — 1 January 1981

Prepared for
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
Building 410
Bolling AFB, D. C. 20332

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STRESS-INDUCED HYDROGEN CONCENTRATION CHANGES IN TITANIUM

by

P. Adler

and

R. Schulte

Final Technical Report on Contract No. F44620-76-C-0071

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The changes in hydrogen concentration that accompany application of a stress-gradient were characterized in titanium alloys so that the influence of stress on hydrogen embrittlement can be understood. In situ measurement of hydrogen was made using the nuclear microprobe for hydrogen (NMH), a nondestructive depth-profiling technique that utilizes the $^7\text{Li}(p,n)^8\text{Be}$ resonant nuclear reaction. Stress gradients were applied by four-point bending. Single phase alloys of α -Ti and β -Ti, as well as different α/β morphologies of Ti-6Al-4V alloy, were examined. Hydrogen redistribution			

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was observed in all the β -phase alloys under an elastic stress gradient, but no further change accompanied plastic deformation. The extent of hydrogen change in high-purity alloys was approximately two times greater than in commercial alloys. Based on this difference and measurements of the partial molal volume of hydrogen in selected alloys, it was concluded that a portion of hydrogen in the commercial alloys was not available for redistribution and was, therefore, trapped. Factors such as hydrogen content, oxygen content, and cold working were not found to change the extent of hydrogen trapping. The practical significance of trapping lies in its possible use to reduce the deleterious influence of hydrogen. The diffusion coefficient in these alloys was also determined under both elastic and plastic loadings and was found to be relatively uniform, approximately $1 \times 10^{-6} \text{ cm}^2/\text{s}$, and consistent with those previously reported for β -Ti. *times 10 to the minus 6 cm. per s*

There was no evidence of hydrogen redistribution in the α -Ti and Ti-6Al-4V alloys that were examined. This was attributed to the low diffusion coefficient of hydrogen in α -Ti at room temperature and to trapping at the α/β interface in the Ti-6Al-4V morphology where an apparent continuous β -phase existed. To explain hydrogen redistribution for conditions of plane stress and plane strain, a two-dimensional model of the elastic stress distribution and plastic zone size in the vicinity of a crack front was developed. Concentration increases of the order of 380% in high-purity β -phase alloys under plane strain can be anticipated.

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ABSTRACT

The changes in hydrogen concentration that accompany application of a stress-gradient were characterized in titanium alloys so that the influence of stress on hydrogen embrittlement can be understood. In situ measurement of hydrogen was made using the nuclear microprobe for hydrogen (NMH), a nondestructive depth-profiling technique that utilizes the $^1\text{H}(^7\text{Li}, \gamma)^8\text{Be}$ resonant nuclear reaction. Stress gradients were applied by four-point bending. Single phase alloys of β -Ti and α -Ti, as well as different α/β morphologies of Ti-6Al-4V alloy, were examined. Hydrogen redistribution was observed in all the β -phase alloys under an elastic stress gradient, but no further change accompanied plastic deformation. The extent of hydrogen change in high-purity alloys was approximately two times greater than in commercial alloys. Based on this difference and measurements of the partial molal volume of hydrogen in selected alloys, it was concluded that a portion of hydrogen in the commercial alloys was not available for redistribution and was, therefore, trapped. Factors such as hydrogen content, oxygen content, and cold working were not found to change the extent of hydrogen trapping. The practical significance of trapping lies in its possible use to reduce the deleterious influence of hydrogen. The diffusion coefficient in these alloys was also determined under both elastic and plastic loadings and was found to be relatively uniform, approximately $1 \times 10^{-6} \text{cm}^2/\text{s}$, and consistent with those previously reported for β -Ti.

There was no evidence of hydrogen redistribution in the α -Ti and Ti-6Al-4V alloys that were examined. This was attributed to the low diffusion coefficient of hydrogen in α -Ti at room temperature and to trapping at the α/β interface in the Ti-6Al-4V morphology where an apparent continuous β -phase existed. To explain hydrogen redistribution for conditions of plane stress and plane strain, a two-dimensional model of the elastic stress distribution and plastic zone size in the vicinity of a crack front was developed. Concentration increases of the order of 380% in high-purity β -phase alloys under plane strain can be anticipated.

SUMMARY OF RESULTS

In situ nuclear techniques for hydrogen measurement have been developed and applied to studies of hydrogen concentration change in a stress gradient. This has led to a direct characterization of the behavior of hydrogen in β -Ti alloys, in α -Ti alloys, and in different morphologies of an $\alpha + \beta$ -Ti alloy. Analytic and experimental approaches were also developed to describe the hydrogen distribution in the vicinity of an advancing crack front. As a result of the information gained during this investigation, the influence of stress on hydrogen redistribution in high strength alloys can be described more quantitatively, thus leading to a greater understanding of the role of hydrogen in embrittlement.

General Approach

Initially, techniques were developed so that the extent and kinetics of hydrogen concentration change in an applied stress gradient could be measured. For this purpose, the nuclear microprobe for hydrogen (NMH), a Grumman-patented technique for in situ hydrogen profiling, was utilized in combination with a four-point bending device. Under four-point bending, a linear elastic stress gradient is introduced across the thickness of a sample. This gradient gives rise to a chemical potential driving force that induces the hydrogen redistribution. The behavior of hydrogen was characterized by measuring the change in hydrogen concentration in different titanium alloys as a function of the applied stress gradient and the time following a reversal of the stress gradient. These results were compared with the relationship for concentration change proposed by Li, Oriani, and Darken, i.e.,

$$\ln \left(\frac{C_S}{C_0} \right) = \frac{\bar{V}_H \sigma_h}{RT}$$

where C_S = hydrogen concentration in stressed region

C_0 = hydrogen concentration in unstressed region

$\bar{V}_H$ = partial molal volume of hydrogen in specific lattice

σ_h = hydrostatic tensile stress

R = gas constant

T = absolute temperature

and with solutions of the diffusion equation.

In addition, the hydrogen distribution in the vicinity of a crack front was considered both analytically and experimentally. Direct measurements of the hydrogen concentration distribution in compact tensile specimens under load were made to compare calculated distributions for plane stress to those actually observed.

β -Phase Alloys

The β -phase of titanium was selected for initial study because of its high solubility for hydrogen and the high mobility of hydrogen in this phase. Three commercial-purity and two high-purity β -phase alloys were examined. They were appropriately heat treated to produce a single phase material. The response of hydrogen to elastic stress gradients and plastic deformation was measured and found to be consistent with the stress dependent form of the Li, Oriani, and Darken relationship, with no further change accompanying plastic deformation. This is illustrated in Fig. 1 for a high-purity alloy. However, the extent of hydrogen concentration change for a specific stress gradient differed between alloys. This is reflected by the parameter $\bar{V}_H^S$, obtained from evaluation of the slope of the exponential function expressing concentration change. (This quantity has been designated as $\bar{V}_H^S$ rather than $\bar{V}_H$ for reasons that will be apparent shortly.) For high-purity alloys, $\bar{V}_H^S$ was approximately equivalent to that reported for the partial molal volume of hydrogen in the β -phase at high temperature; for the commercial alloys, $\bar{V}_H^S$ was approximately half that of the high-purity material. It was concluded that $\bar{V}_H^S$ was indicative of the actual response of hydrogen in a stress gradient for a particular material and was not necessarily the partial molal volume of hydrogen, $\bar{V}_H$, in that material. Trapping of hydrogen or some other factor appears to affect the availability of hydrogen for redistribution. This results in a value of $\bar{V}_H^S$ that is lower than $\bar{V}_H$.

To determine $\bar{V}_H$ for the specific alloys under investigation, hydrogen charging and x-ray diffraction techniques for precision lattice parameter measurement were developed. The molar volume was measured for a series of hydrogen charged samples and the value of $\bar{V}_H$ was then determined for each alloy by extrapolating the molar volume data to the 100% hydrogen composition intercept. $\bar{V}_H^S$ and $\bar{V}_H$ values are compared in Table 1. For the commercial

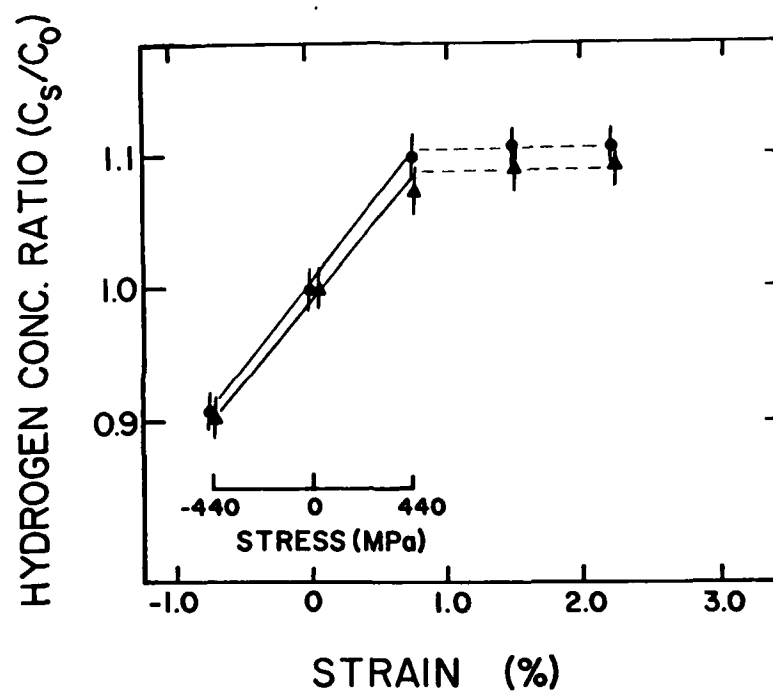


Fig. 1 Variation of Hydrogen Concentration with Applied Strain (2 samples) for High-Purity β -Phase Ti-31V Alloy. Least squares exponential fit is shown within elastic limits of alloy.

alloys, $\bar{V}_H^S < \bar{V}_H$ whereas $\bar{V}_H^S \approx \bar{V}_H$ for the high-purity alloys. These results are consistent with the idea that a portion of the existing hydrogen can be trapped and is not available for redistribution when stress is applied.

TABLE 1 COMPARISON OF PARTIAL MOLAL VOLUMES FOR β -TITANIUM ALLOYS

Alloy	Type	Lot	Partial Molal Volume (cm ³ /mol)	
			$\bar{V}_H^S$	$\bar{V}_H$
Ti-8Mo-8V-2Fe-3Al	Commercial Purity	1	0.8 $\pm$ 0.2	1.5 $\pm$ 0.2
		2	1.2 $\pm$ 0.2	1.6 $\pm$ 0.2
Ti-3Al-8V-6Cr-4Mo-4Zr (β -c)	Commercial Purity	-	0.6 $\pm$ 0.2	--
Ti-30Mo	Commercial Purity	1	0.8 $\pm$ 0.2	--
		2	1.1 $\pm$ 0.2	--
Ti-31V	High Purity	1	1.8 $\pm$ 0.2	--
		2	--	1.6 $\pm$ 0.2
		3	1.6 $\pm$ 0.2	--
Ti-18Mo	High Purity	1	1.4 $\pm$ 0.2	--

In order to identify the cause of hydrogen trapping in titanium alloys, specimens were prepared in which the amount of hydrogen content, cold working, and oxygen content were systematically varied. Samples of commercial Ti-8Mo-8V-2Fe-3Al alloy were hydrogen charged over a range from 175 to 3200 WPPM (0.9 to 16 at. % hydrogen) to determine whether saturation of trapping sites by hydrogen would occur. No significant change in $\bar{V}_H^S$ was indicated over this entire composition range, as is shown in Fig. 2. Hence, the proportion of hydrogen that had undergone redistribution remained essentially unchanged. The influence of dislocations in this alloy was also investigated. Samples were given a 10% reduction in thickness by cold rolling to introduce a significant density of dislocations. No significant reduction in $\bar{V}_H$ was indicated, suggesting that dislocations do not act as traps in this material. To evaluate the effect of oxygen content, samples of high-purity Ti-31V containing 700, 1500, 2000, and 7000 WPPM (0.2 - 22 at. % oxygen) were examined. The values of $\bar{V}_H^S$ determined for these samples were relatively the same, 1.6 $\pm$ 0.2 cm³/mol, indicating that oxygen content is not an

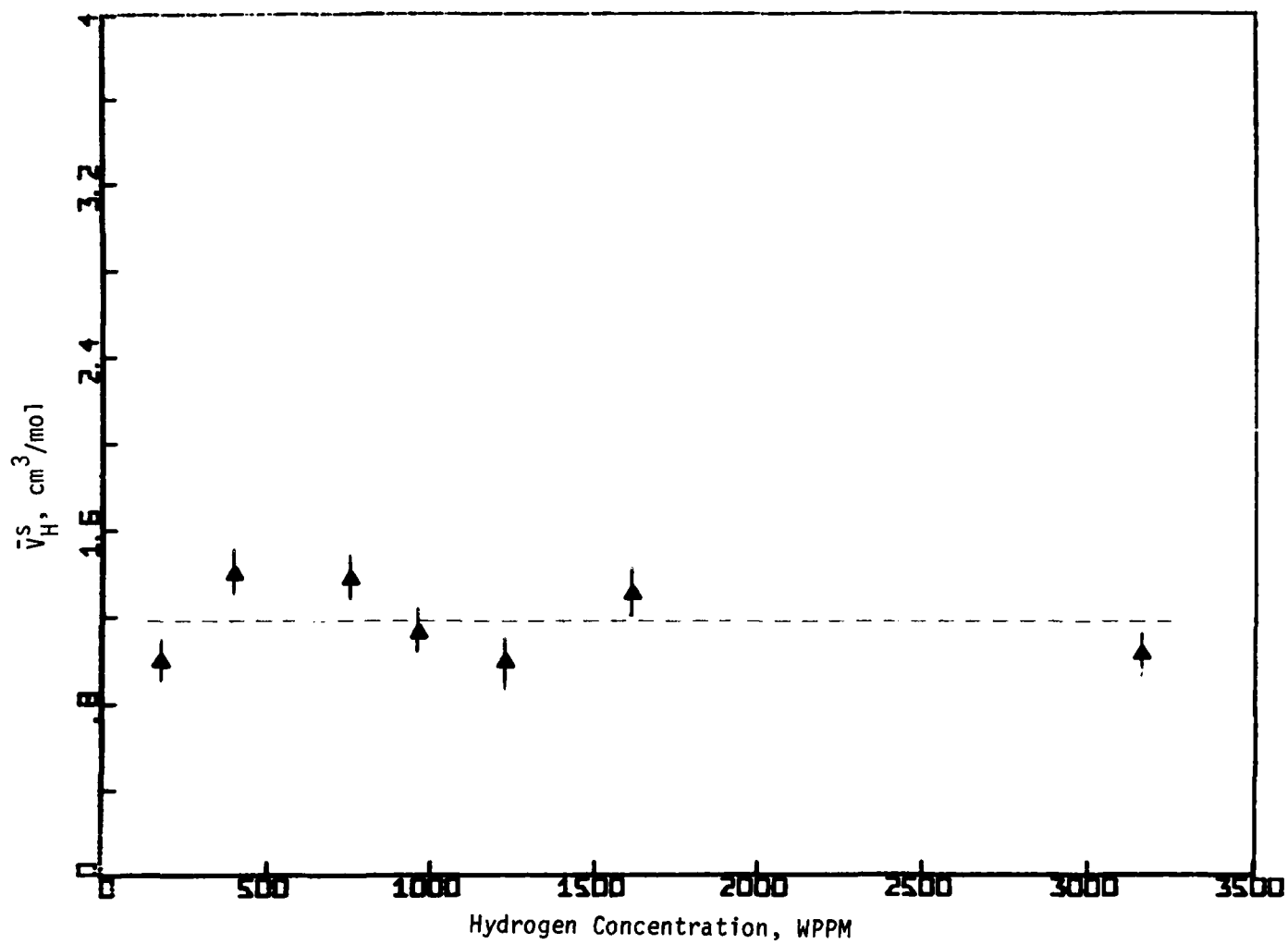


Fig. 2 Effect of Hydrogen Concentration on $\bar{V}_H^S$ of Ti-8Mo-8V-2Fe-3Al Alloy.

influential factor. Therefore, it appears that neither hydrogen content, dislocation density, nor oxygen content are of primary importance to the behavior of hydrogen in a stress gradient. If the cause of hydrogen trapping could be identified, it could possibly be used to reduce the deleterious influence of hydrogen.

Throughout the course of this investigation, the kinetics associated with hydrogen transport in a stress gradient were evaluated. Measurements were made of the time-dependent concentration change following a reversal of the stress gradient. Typical data are shown in Fig. 3 for a high-purity alloy. The diffusion coefficient was determined using a constant D solution of the diffusion equation. The value of D was found to be approximately $1 \times 10^{-6} \text{ cm}^2/\text{s}$ for all the β -alloys examined. This value is consistent with values of D in the β -phase of titanium obtained by others and indicates that stress-induced migration occurs by normal kinetics involving thermal activation.

α -Ti and $\alpha+\beta$ -Ti Alloys

Similar investigations of hydrogen concentration change in a stress gradient were conducted on two commercial α -phase alloys, C.P. Ti and Ti-5Al-2.5 Sn, and three morphologies of an $\alpha + \beta$ alloy, Ti-6Al-4V. In no case, however, was hydrogen redistribution observed. For the α -phase alloys, this was attributed to the low diffusivity in α -Ti at room temperature, i.e., $D = 10^{-11} \text{ cm}^2/\text{s}$, and the relatively long diffusion path, 0.3 - 1 mm, in the samples studied. For the $\alpha+\beta$ alloy, one of the microstructures examined, the β -annealed, contained elongated α -grains in what appeared to be a continuous β -matrix. Trapping of hydrogen at the $\alpha + \beta$ interface region (or at regions of high stress near this interface) was proposed to explain the lack of transport in this morphology.

Because of the unsuccessful efforts to measure hydrogen concentration change in a stress gradient in these systems, other approaches were attempted. In one, use of a thin film of α -Ti on a β -alloy substrate was explored. Diffusion in thin α -Ti films was not expected to limit hydrogen redistribution. Films of pure α -Ti of approximately $1 \mu\text{m}$ thickness were sputtered onto β -Ti substrates that had been sputter-etched. Samples were examined under four-point bending, with the β -substrate serving as the source or sink

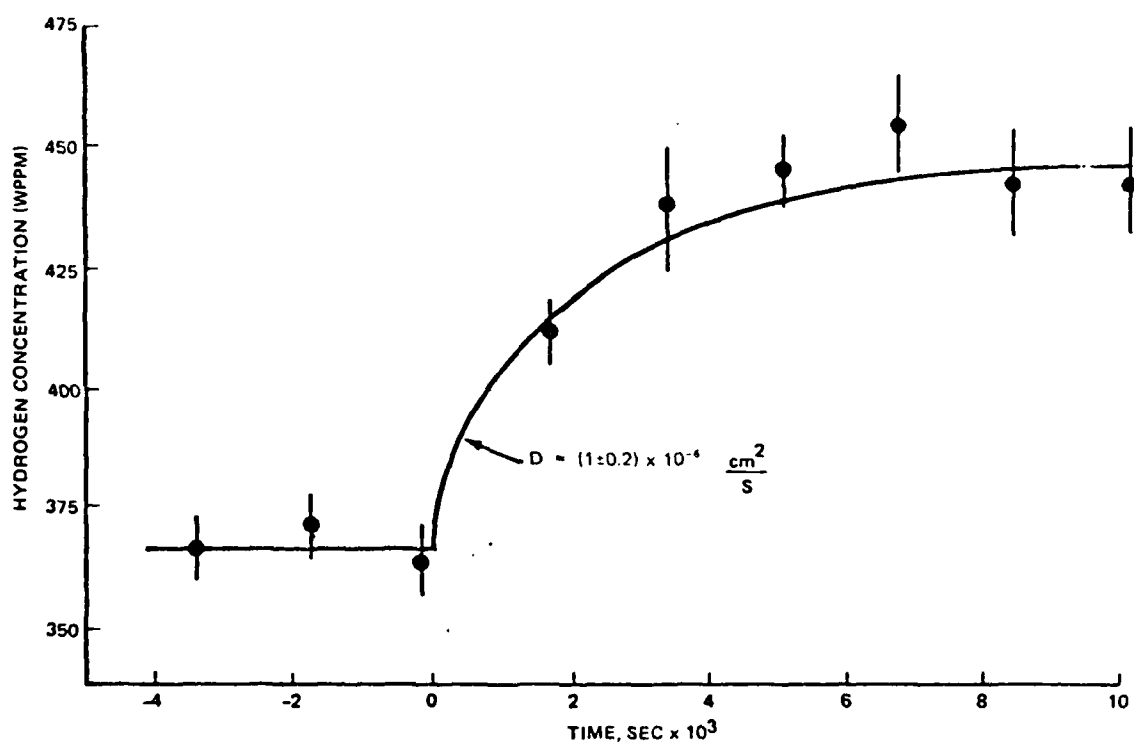


Fig. 3 Time-Dependent Hydrogen Concentration Change in High-Purity β -Phase Ti-31V Alloy Accompanying Reversal of Stress Gradient. Diffusion coefficient indicated based on least squares fit of data.

for hydrogen. No hydrogen concentration change in the film was observed, however, when either a positive or negative stress gradient was imposed on the sample. Therefore, the simulation did not appear to be valid and the approach was abandoned.

Long term holding of relatively thin samples was another method that was used to evaluate hydrogen redistribution in these alloys. For $D = 10^{-10}$ to 10^{-11} cm^2/s , the diffusion time through a 0.5 mm thickness sample would be approximately 70 to 700 days. Samples of C.P. Ti and β -annealed Ti-6Al-4V were held under four-point bending, in a stress gradient of approximately two times their yield strength, for a period of 245 days. The hydrogen concentration at the front (tension) and back (compression) surfaces was then measured and compared to unloaded control samples. Unfortunately, the range of experimental error in these measurements exceeded the hydrogen concentration change that was projected to occur and, therefore, no conclusions could be drawn.

Despite the many attempts to evaluate hydrogen concentration change in a stress gradient, no evidence for such a change was indicated in any of the α -Ti and Ti-6Al-4V samples studied. While stress-induced hydrogen redistribution may be important in these systems, the techniques used in this work were incapable of characterizing this behavior because of the low hydrogen concentration and the low diffusivity of hydrogen in these alloys.

Crack Front Considerations

Analytic and experimental approaches were developed to describe the hydrogen distribution in the vicinity of a crack front. The analytic approach involved two-dimensional consideration of the elastic stress distribution and plastic zone size for conditions of plane stress and plane strain. A typical representation of the stress distribution for a compact tensile specimen under plane stress is shown in Fig. 4. For material with a yield strength of 779 MPa, the maximum anticipated increase in hydrogen concentration under plane stress would be approximately 20% for $\bar{V}_H^S = 0.8 \text{ cm}^3/\text{mol}$ and 40% for $\bar{V}_H^S = 1.6 \text{ cm}^3/\text{mol}$. For plane strain, however, the expected increase would be 100% for $\bar{V}_H^S$ and $0.8 \text{ cm}^3/\text{mol}$ and 380% for $\bar{V}_H^S = 1.6 \text{ cm}^3/\text{mol}$. These calculations indicate that large increases in localized hydrogen concentration can occur under applied load where conditions of plastic constraint exist.

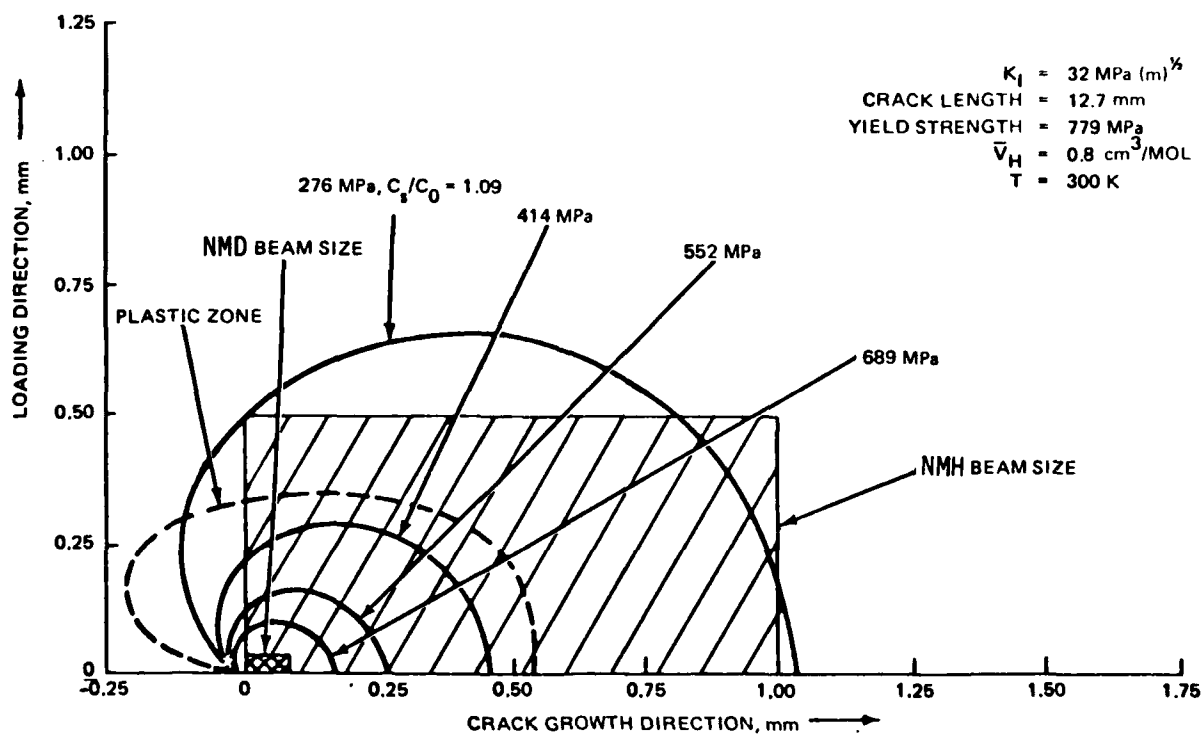


Fig. 4 Comparison of Analytic Description of Stress and Hydrogen Distribution in Vicinity of Crack Front under Plane Stress with Nuclear Microprobe for Hydrogen (NMH) and Nuclear Microprobe for Deuterium (NMD) Spatial Resolution Capability.

Two-dimensional modeling of the stress distribution was necessary for comparison to hydrogen measurements in the crack front vicinity. Using nuclear microprobe techniques, the hydrogen concentrations can only be determined in the near-surface region. Unfortunately, the expected increase in the hydrogen concentration in the crack front region is at a minimum for the condition of the plane stress at the surface. Hydrogen concentration measurements were made in Ti-30Mo compact tensile specimens in the vicinity of the crack front. This material had a $\bar{V}_H^S$ value of $1.1 \pm 0.2 \text{ cm}^3/\text{mole}$. The smallest beam size that could be achieved with reliability, using the NMH, was large in relation to the region of hydrogen concentration increase, as is illustrated in Fig. 4. No significant increase in hydrogen content in the crack front region was found to a stress intensity of $34 \text{ MPa } \sqrt{\text{m}}$. Subsequently, specimens were side-grooved to promote plane strain near the surface, but, again no increase was observed. It was concluded that the spatial resolution of the NMH was not sufficient to measure the localized concentration change under plane stress.

Preliminary work was done using another nuclear microprobe technique, the nuclear microprobe for deuterium (NMD). For the NMD, a beam size of approximately $0.08 \times 0.08 \text{ mm}$ can be used compared to $1 \times 1 \text{ mm}$ for the NMH. The spatial resolution that can be achieved for each of these approaches is illustrated in Fig. 4. The NMD measures the hydrogen isotope (deuterium) and requires charging samples with deuterium. The deuterium concentration is determined using the $^2\text{H}(^3\text{He}, \text{p})^4\text{He}$ reaction. A finely-collimated beam of $1.2 \text{ MeV } ^3\text{He}$ is used, and the integrated yield of high energy protons that are emitted is proportional to the deuterium concentration. Compact tensile specimens of Ti-30Mo were deuterium charged to a uniform concentration through their cross-section. The device for loading the compact-tensile specimens, however, limits the sensitivity of the NMD measurements and extensive modification of this apparatus is required to make the appropriate measurements. As a result, these measurements must be made in future work.

PUBLICATIONS

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P.N. Adler, E.A. Kamykowski, F.J. Kuehne, E.J. Schneid, and R.L. Schulte, "Stress Induced Hydrogen Concentration Changes in β -Phase Titanium Alloys," presented at Fall AIME Meeting, Oct 17, 1978, St. Louis, MO.

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R.L. Schulte, P.N. Adler, and J. Montgomery, "Hydrogen Trapping in Commercial Titanium Alloys," to be published.

P.N. Adler, R.L. Schulte, and E. Ting, "Hydrogen Concentrations in the Vicinity of a Crack Front," to be published.

PERSONNEL INVOLVED

Dr. Philip N. Adler - Principal Investigator

Dr. Robert L. Schulte - Co-Investigator

Dr. John M. Papazian - Materials

Edmund Ting - Materials

James Kennedy - Materials

Jonathan Montgomery - Materials

Fred J. Kuehne - Nuclear

Dr. Edward A. Kamykowski - Nuclear

Dr. Edward J. Schneid - Nuclear

COUPLING ACTIVITIES

- o IRAD Program - "Fracture in Titanium"
- o Grumman Aircraft Programs - Diagnostics for influence of hydrogen in service failures
- o Professor Don Koss, Michigan Technological University - Discussions relating to high-purity β -phase alloys; supplier of high-purity Ti-31V alloy
- o Dr. Neil Paton, Rockwell International - Cooperation in hydrogen charging samples; collaboration in work on hydrogen availability for stress-induced migration; supplier of high-purity Ti-18Mo and samples of Ti-31V containing oxygen
- o Professor Harold Margolin, Polytechnic Institute of New York - Discussions and experiments on hydrogen segregation at α/β titanium interfaces; Co-principal Investigator on USN/ONR Program "Hydrogen Segregation at Titanium Alloy α/β Interface," (N00014-80-C-0742)
- o David W. Taylor Naval Ship R&D Center - Hydrogen charging and In-situ Analysis of Titanium Alloys (N61533-81-M-0014 and N61533-80-M-3751)
- o Naval Sea Systems Command "Hydrogen Migration in Dissimilar Metal Titanium Weldments," (N00024-81-C-5309)

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