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REACTION OF HYDROGEN WITH NIOBIUM AND TANTALUM IN THE ATR GAS LOOP

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By

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INTRODUCTION

This report reviews recent literature concerned with the hydrogen-niobium (columbium) and the hydrogen-tantalum systems. Hydrogen embrittlement of refractory metal components is a potential problem in the operation of the ATR gas loop. The extent of hydrogen pick-up and the relative rates of sorption-desorption must be considered for the ATR loop operation.

SUMMARY AND CONCLUSIONS

For similar conditions, the solubility of hydrogen in tantalum will generally be less than in niobium. Interstitial contamination (oxygen, nitrogen and carbon are the most common) of the pure metals will increase their resistance to hydrogen sorption, although these contaminants are also detrimental to the ductile properties of the metals. Both hydrogen-metal systems are considered to be a continuous solid solution at temperatures above 300 C. Above 900 C, both niobium and tantalum show comparatively little affinity for hydrogen. However, from 500 or 600 C to room temperature, the absorptive capacity of hydrogen in these metals becomes important and will generally increase as the temperature decreases.

Hydrogen levels of 500 ppm seriously embrittle niobium at room temperature. Such a concentration level of hydrogen will probably not be attained in the ATR loop for normal operating conditions above 200 C. Below 200 C, the possibility exists for hydriding to a brittle state. However, considering that the mass of contaminant to mass of refractory metal is prohibitive to high hydriding levels and that the kinetics of sorption are much slower, the probability of embrittlement is small. Mass considerations exclude the deliberate addition of hydrogen to the helium coolant.

The kinetics for sorption of hydrogen by either tantalum or niobium follow no consistant rate expression over a temperature range of 250 to 600 C. However, both show half order pressure dependence which is attributed to the role of atomic hydrogen in the sorption process. Oxygen acts to inhibit hydrogen sorption. Under ATR conditions, it should be expected that surface conditions will be important. A light oxide tarnish on the refractory metal should be an effective barrier to hydrogen, i. e., slow diffusion through a thin film. Oxide films on niobium dissolve into the metal at temperatures of 850 C in high vacuum.

The data reviewed indicates that, due to the low contaminant concentrations expected in the helium gas stream, hydrogen should not be significant in influencing the mechanical properties of niobium or tantalum in the ATR loop. Hydrogen concentrations can be predicted on the basis of hydrogen partial pressures, temperature, and total pressure. The effects of specific hydrogen levels on mechanical properties and subsequent behavior are not so easily predicted. It can be said qualitatively that hydrogen levels of 500 ppm will embrittle niobium. Hydrogen levels of 200 ppm will raise the proportional stress limit of niobium. Final analysis of hydrogen levels detrimental to the refractory metals must be made from observation in the ATR gas loop itself.

DISCUSSION

Hydrogen-Niobium System

Solubility (Occlusion Capacity). Niobium absorbs hydrogen readily at elevated temperatures forming a brittle, dark gray solid hydride. The limiting composition of the niobium hydride is considered to be NbH.(1) The niobium-hydrogen equilibrium has been studied in the temperature range of 100 to 900 C and 0. 1 to 1000 Torr hydrogen pressure,(2) and also in the temperature range of 300 to 1500 C and 0 to 760 Torr hydrogen pressure.(3) Figure 1 shows the equilibrium sorption isotherms plotted as external hydrogen pressure versus hydrogen/niobium atomic ratio. The data presented was taken from isopleths of both Albrecht(2) and Komjathy(3) over a temperature range critical to ATR gas loop operation.

Because the niobium-hydrogen composition varies with the external hydrogen pressure over the temperature range (Figure 1), the system can

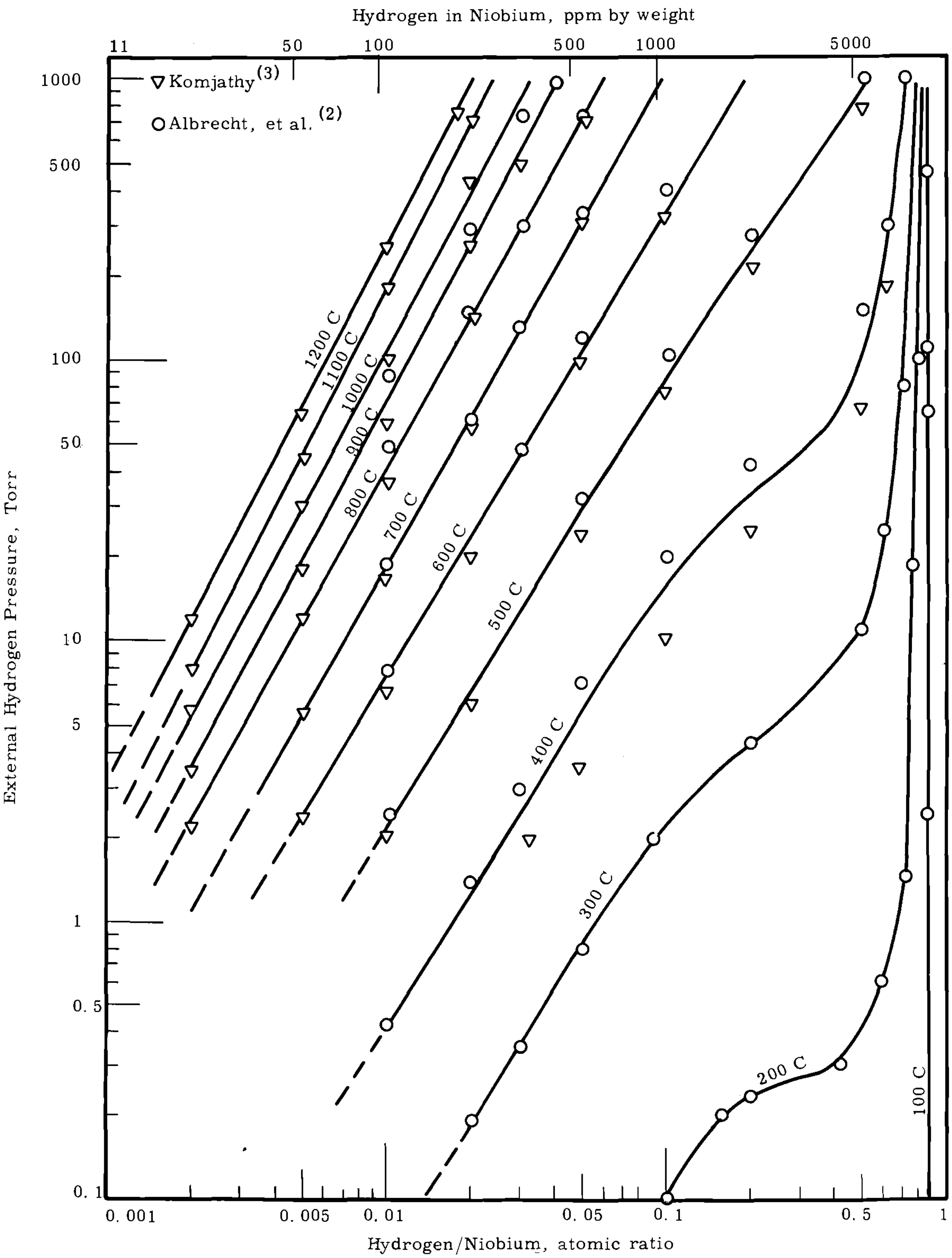

FIGURE 1

Logarithmic Plot of Isotherms
in the Hydrogen-Niobium System

be considered a continuous solid solution.(2, 3) However, X-ray studies indicate a two phase region at relatively low temperatures and pressures.(4) The critical point for the two phase region has been placed, from equilibrium and X-ray data, at about 140 C, 0.01 Torr hydrogen pressure and a 0.3 hydrogen/niobium ratio.(2, 4)

Equilibrium solubility comparisons (Table I) from three different studies show a variance in the amount of hydrogen absorbed by niobium at one atmosphere hydrogen pressure. Deviations have been attributed to the differences in the purity of the metal. The effect of contamination will be discussed later.

TABLE I

COMPARISON OF THE EQUILIBRIUM DATA AT 1 atm FOR THE NIOBIUM-HYDROGEN SYSTEM

Temperature, C	cc Hydrogen/gm Niobium		
	Paxton and Sheehan(5)	Sieverts and Moritz(6)	Albrecht et al.(2)
900	- -	4.0	- -
800	5.7	6.2	6.2
600	19.4	18.2	19.4
500	26.6	47.4	50.9
400	66.5	76.8	82.3
300	79.9	88.0	93.1

Sorption Kinetics. Albrecht, et al.(2) determined the rates of sorption to produce hydrogen to niobium ratios of 0.05, 0.10, 0.50, and 0.70 in the temperature range of 300 to 700 C. For temperatures up to 550 C, the initial rate of sorption was linear, decreasing after a time and depending on the conditions as the equilibrium value was approached. At 600 and 700 C, they found the rate to be diffusion controlled following a parabolic curve fit until 40% of saturation was obtained. At 700 C, a cylindrical specimen 0.6 cm in diam attained equilibrium solubility (0.05 H/Nb atomic ratio) in a 330 Torr hydrogen atmosphere in about 5 min.

Gulbransen and Andrew(7) found no consistent rate law governing the absorption of hydrogen from temperatures of 250 to 900 C and pressures up to 50 Torr. However, at 300 C they did note that the initial rate of sorption was a function of the square root of pressure over a pressure range of 1.8 Torr to 57 Torr hydrogen. At higher temperatures, Gulbransen and Andrew found lower reaction rates than did Albrecht. From continuous weight gain measurements on samples approximately 3.3 mils thick (0.008 cm) and 15 cm^2 surface area, they followed the increase in hydrogen absorption at 700 and 900 C over a 2 hr period. At the end of this time, approximately 6.5 $\mu g/cm^2$ weight gain was obtained at 900 C. The system was then exposed to high vacuum, 10^{-6} Torr, for 30 min with no decrease or increase in the sample weight. At 700 C, a weight gain of approximately 2.5 $\mu g/cm^2$ was observed over the 2 hr period. High vacuum conditions were imposed and a weight loss to approximately 0.5 $\mu g/cm^2$ was noted. Thus, Gulbransen concluded that there is a low temperature hydrogen reaction product stable _in vacuo_ to temperatures of 250 C and a high temperature product stable at 900 C to a vacuum of the order of 10^{-6} Torr. Apparently between 250 and 900 C, desorption proceeds very rapidly under condition of 10^{-6} Torr vacuum.

The formation of a stable hydride product at 900 C seems at variance with the equilibrium data for the niobium-hydrogen system. Unfortunately, Albrecht, et al., only carried their sorption-desorption tests to 700 C. They found that diffusion coefficients calculated from absorption data were greater than those calculated from desorption data. A surface reaction must influence the desorption of hydrogen noted above. Surface contamination might also be an explanation for the desorption process as well as for the apparent 900 C stable hydride proposed by Gulbransen.

To illustrate the effect of pressure, temperature and the atomic hydrogen to niobium ratio on the sorption kinetics, Table II* lists representative rate constants calculated by Albrecht, et al. A rate constant calculated from Gulbransen's data is also included for comparative purposes.

Hydrogen-Tantalum System

Solubility (Occlusion Capacity). Tantalum is a slightly less effective getter for hydrogen than niobium, but still dissolves a considerable amount of hydrogen at relatively low temperatures. An originally shiny, ductile

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* Table II appears on p. 12.

piece of tantalum when heated in hydrogen is converted to a hard, brittle, dark gray solid, tantalum hydride ($TaH_{0.76}$).(1) The absorption is accompanied by an expansion in the crystal structure of the metal. Desorption of the hydrogen can easily be accomplished at the higher temperatures; however, the metal will not regain its original ductility, electrical resistance or recover its original crystal lattice spacing.(1, 8)

Except at low temperatures, tantalum does not form a hydride.(9, 10) The tantalum-hydrogen system, like the niobium-hydrogen system, has two regions of solid solubility separated by a miscibility gap.(11) X-ray studies have shown the existence of tantalum or an α-phase and an intermediate hydride at room temperature.(11)

Figure 2 shows the solubility of hydrogen at 1 atm in tantalum for various temperatures. For comparative purposes, a portion of the niobium-hydrogen solubility curve is also shown. The much lower equilibrium solubility of hydrogen in tantalum than in niobium at equivalent pressure is obvious.

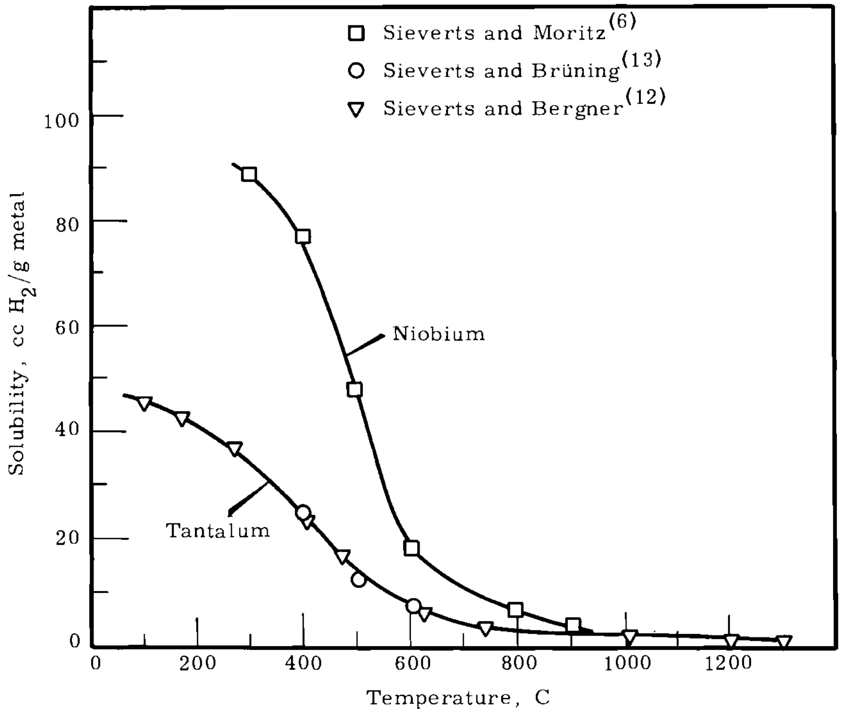

FIGURE 2

Solubility of Hydrogen in Tantalum and Niobium at 1 atm Pressure

Mary Andrews(9) studied the absorption of hydrogen by tantalum at low pressures (less than 60 μ) and found that hydrogen was not taken up from hydrogen gas at temperatures of 700 to 800 C. But, hydrogen was absorbed by tantalum at these temperatures from mixtures of diatomic and monatomic hydrogen gas formed by the dissociation reaction of hydrogen on a hot tungsten surface near the tantalum sample. The hydrogen absorbed from the monatomic-diatomic hydrogen mixture caused an increase in the electrical resistance of tantalum; however, there was no evidence of a compound being formed. Zubler(10) studied the absorption of hydrogen by a tantalum ring at pressures less than 200 μ and arrived at conclusions similar to those of Andrews. The role of atomic hydrogen and the half-order pressure dependence at temperatures of 300 to 500 C was, as in the case of niobium, demonstrated during initial sorption.(9, 10, 7)

Sorption Kinetics. The reaction of tantalum with hydrogen is similar to the niobium-hydrogen reaction. Tantalum does not react rapidly with hydrogen below 350 C;(12) but, as the temperature increases, the rate of reaction increases. Zubler(10) observed a parabolic rate of absorption over a temperature range of 200 to 500 C (pressure range of 0 to 200 μ hydrogen), using pressure changes to follow the reaction. Above 500 C, the rate was too fast to measure by his technique. Isothermal weight gain curves presented by Gulbransen and Andrew(7) show that, as in the case of niobium, no consistent rate law can be expressed for the sorption process over a temperature range of 350 to 500 C (pressures of 1.8 to 57 Torr hydrogen). The apparent discrepancy between Gulbransen's and Zubler's work may be differences in pressure ranges studied and experimental methods.

From Gulbransen's data, a linear rate law is observed at 400 C. The rate constant is calculated as 0.15 $\mu g/cm^{+2}$ min to 60 min. At 450 C the rate reaches a maximum of 14 $\mu g/cm^2$ in 35 min and levels off. At 500 C the rate maximized at 6 $\mu g/cm^2$ in 10 min before leveling off.

There is evidence, as for niobium, of a second stable hydride above 600 C. A low temperature form exists below 350 C in high vacuo (10^{-6} Torr), and a high temperature form stabilizes at temperatures above 700 C in high vacuo. The stability of a high temperature hydride is doubted in some quarters.(14) Since it is apparent at atmospheric pressure that a tantalum hydride is not stable above 600 C (Figure 2), the observation that a hydride is stable at 700 C must be partially due to contamination of the metal.(14)

Effect of Contamination on Hydrogen Sorption. Contamination of niobium and tantalum by oxygen or nitrogen is believed to influence both the stability and degree of the hydrogen solubility in these metals.(15) Rees(16) observed a steadily diminishing hydrogen solubility in zirconium with increasing amounts of oxygen, and attributed the effect to the blocking of sites by oxygen. Kofstad(17) indicated that if oxygen acts similarly in tantalum and niobium, the introduction of each oxygen atom would destroy the solvent power of an array of 11 or 12 metal atoms. Oxygen atoms, believed to reside in interstitial octahedral positions, would restrict the sorption of hydrogen in the interstitial tetrahedral sites of a body centered cubic lattice.(17) Each oxygen atom would then tend to block out 60 or 70 interstitial positions or the equivalent sites at a distance of "less than two atomic diameters from the impurity atom".(17)

It would seem that the addition of interstitial elements to niobium or tantalum would decrease the amount of hydrogen absorbed. However, this may not always be so. Experiments have shown that tantalum becomes passive to hydrogen when oxygen is completely removed from the metal by flashing at 2500 C in high vacuum.(15) The role of interstitial contaminants, such as oxygen, in the hydrogen sorption process is not clearly defined.

Effect of Hydrogen on Mechanical Properties. Interstitial additions to both niobium and tantalum have an important role in determining the mechanical properties of these metals. Hydrogen in tantalum appears to increase the hardness and reduce ductility and tensile strength.(18) Unfortunately, allowable limits for hydrogen in tantalum have not been established.

Figure 3 relates the hydrogen content in niobium to room temperature tensile properties while Figure 4 shows the effect of hydrogen on room

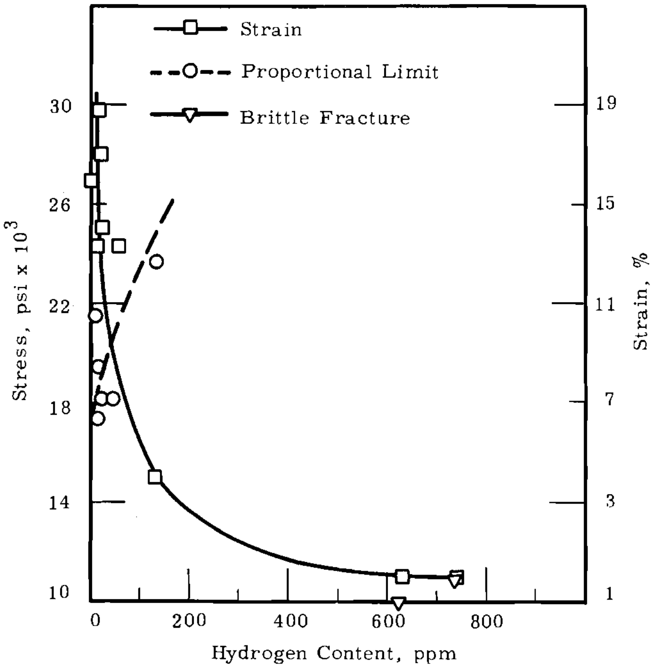

FIGURE 3

Effect of Hydrogen on Room Temperature Tensile Properties of Niobium(19)

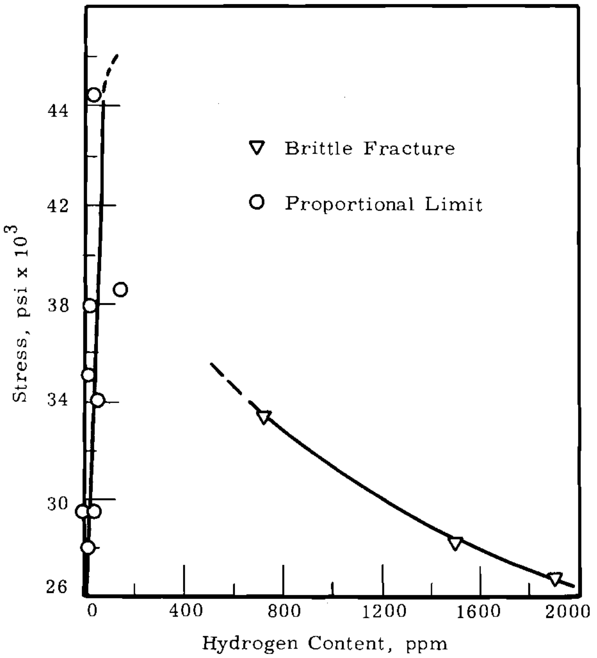

FIGURE 4

Effect of Hydrogen on Room Temperature Bend Properties of Niobium(19) temperature bend properties. These tests on niobium have shown that hydrogen additions up to 200 ppm increases the proportional stress limit.(19) Hydrogen additions greater than 500 ppm have been shown to seriously embrittle niobium.(19)

Creep tests at high temperatures (approximately 1000 C) in atmospheres containing hydrogen showed an increase in the creep rate, but no reduction in ductility was apparent.(19)

Application to the ATR Gas Loop. In the ATR gas loop, refractory metal sample holders will be subjected to a high temperature (approximately 1100 to 1200 C) in high velocity helium gas containing trace impurities (goal purity of the helium is 1 vpm total contaminants). From the data on niobium, it is apparent that a pure niobium or tantalum sample holder will have little affinity for hydrogen and should be in no danger of hydriding at the operating temperatures. Raising the temperature of the loop from room to operating temperatures can be considered a desorption process since the hydride is less stable as the temperature increases. As the temperature decreases, as in a shutdown or cycling procedure, the probability of hydriding the sample holder increases. Thermodynamic, kinetic and mass effects must be evaluated in order to predict the extent of hydriding in the test loop.

The following is typical of ATR gas loop mass-balance relationships. The niobium sample holder in the ATR loop weighs approximately 10 lb. For a mean temperature of 750 C, volume of 90 ft^3 and total pressure of 20 atm, the approximate weight of helium in the system is 5 lb. Assuming a hydrogen contaminant level of 10 vpm (5 ppm by weight), the increase in the hydrogen level in the niobium sample holder, if all the hydrogen is adsorbed and uniformly distributed, will be 2. 5 ppm. Seventy pounds of niobium is a more prototypical weight if a niobium sleeve is used in the ATR loop in-core section. The same contaminant level in 5 lb of helium would increase the hydrogen level in the niobium by about 0. 36 ppm.

Increases of this kind are hardly cause for concern. However, hydrogen could be deliberately added to the ATR loop in an effort to control oxidation of structural materials. [Considering the molybdenum heaters for a moment, Dushman(20) indicates a H_2O/H_2 ratio of 0.524 at 927 C for the reduction reaction: $MoO_2 + 2H_2 \rightleftarrows Mo + 2H_2O$. For an initial oxygen partial pressure of 16 μ, approximately 30.4 μ hydrogen would be required to maintain the above equilibrium. The reduction reaction for chromium, $Cr_2O_3 + 3H_2 \rightleftarrows 2Cr + 3H_2O$, has a H_2O/H_2 ratio of 8.77×10^{-5} at 927 C.(20) An initial oxygen partial pressure of 16 μ in this case would require a minimum hydrogen overpressure of about 126 Torr to reverse the reaction.]

Another way of looking at mass-balance relationships is to consider an initial hydrogen concentration in the gas stream which will uniformly increase the hydrogen in the 70 lb of niobium to the 200 ppm level. This would require an initial hydrogen level of 2800 ppm (5600 vpm) in the helium gas stream or an initial hydrogen partial pressure of about 83 Torr. [The choice of 200 ppm is arbitrary, but is assumed to be approximately the maximum allowable limit of hydrogen in niobium. Beyond this limit, ductility decreases markedly.(19)] Obviously the loop should not and probably will never operate at this high a hydrogen partial pressure.

Granting enough contaminant in the gas stream to obtain the 200 ppm hydrogen in niobium, the effect of temperature on absorptive capacity should be considered. Figure 1 indicates that at about 83 Torr hydrogen and 750 C hydrogen will equilibrate in niobium to the 200 ppm level. Above 750 C, less hydrogen will be held by the metal while below 750 C, more hydrogen can be accomodated. If the niobium is always above 750 C, the 200 ppm level will not be reached even in a partial pressure of 83 Torr hydrogen. Another consideration at this point is the redistribution of hydrogen to low temperature areas. Unfortunately, the migration of hydrogen in niobium induced by a temperature gradient is not known. Since temperature gradients are not large in the niobium section of the loop, hydrogen redistribution will not be discussed further.

The relative rates of absorption should be considered next. Two different situations exist from the standpoint of kinetic effects: the loop start-up and the loop shutdown. During startup, the loop will be allowed to outgas at a low temperature. Assuming a temperature of 300 C, the relative rate of sorption of hydrogen by niobium from a 2800 ppm hydrogen contaminated helium stream can be approximated (Table II). At 300 C and 83 Torr hydrogen, the rate of sorption is 3.5 x 10^{-4} cc/cm^2 sec, and the solubility limit is about 7,000 ppm hydrogen in niobium. Absorption of 200 ppm in the metal at this temperature would require about 2.5 hr. As the temperature increases, the rate of reaction increases but the solubility limit decreases. At 550 C and 67 Torr hydrogen, the rate of sorption is 21 x 10^{-3} cc/cm^2 sec, and the solubility limit is about 500 ppm. Absorption of 200 ppm would require only about 2 min at 550 C under these circumstances. As the temperature increases above 750 C, the rate is fast but the solubility restriction will not allow 200 ppm to be reached in the metal. In startup, the hydrogen will be absorbed and finally desorbed as the operating loop temperatures are reached. The effect of absorption followed by desorption of large quantities of hydrogen on the mechanical properties of niobium is not known. For this reason, it may be advantageous to inhibit sorption of hydrogen while the loop is at the outgas temperature. The relative slow rate at 300 C would be retarded further by a pretarnish of critical niobium pieces (i.e., the sample holder in particular).

TABLE II

INITIAL LINEAR RATE CONSTANTS TO PRODUCE VARIOUS HYDROGEN-NIOBIUM COMPOSITIONS

H/Nb	Temperature, C	Pressure, Torr	Rate Constant 10^{-3}cc $\text{cm}^{-2}\text{sec}^{-1}$
0.05	550	67	21
0.1	450	52	1.7
	500	112	9.1
	550	218	29
0.5	400	145	1.9
	450	450	14
- -	300 (Gulbransen)	21	0.16
0.7	300	83	0.35
	350	460	6.7

When the loop is cycled or shut down, a different situation exists. As the section containing niobium cools, niobium will find hydrogen favorable for hydriding at some time during cooling (i. e. , at a temperature of rapid absorption coupled with moderate to high solubility). Where 2800 ppm hydrogen is in the helium stream as the loop is cooled, the metal could reach 200 ppm almost immediately, once the loop cools below 750 C. However, the proposed purification system should have all impurities at a low value by the time the loop is at operating temperature.

The use of Figure 1, Table II, and the above illustrations of hydrogen sorption imply conditions which should not be approached in the ATR loop. It should be emphasized that the rate constants and solubility limits were arrived at from data obtained at an essentially constant hydrogen overpressure under isothermal conditions. In the actual operation of the loop, the hydrogen partial pressure will continually diminish as absorption occurs because of gas purification as well as solution in the niobium.

The primary factor, then, in determining the degree of hydrogen sorption, especially during shutdown, will be the relative mass ratios of hydrogen to niobium. The gas purification system for the loop will lower and maintain low contaminant levels during startup and, more important, during shutdown. Therefore, as long as the contaminant level (as initially indicated) remains at the projected level of 1 vpm total impurity, no detrimental effects of hydriding should be expected in the niobium.

ACKNOWLEDGEMENTS

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