

SOLUBILITY OF HYDROGEN IN V-4Cr-4Ti AND LITHIUM* J.-H. Park, G. Dragel, R. A. Erck, and D. L. Smith (Argonne National Laboratory) and R. E. Buxbaum (Michigan State University)

SUMMARY

The solubility of hydrogen in V-4Cr-4Ti and liquid lithium was determined at 400–675°C and a hydrogen pressure of 9.09×10^{-4} torr (1.21×10^{-1} Pa). Hydrogen concentration in both materials decreased as temperature increased, and the ratio of the hydrogen concentration in liquid lithium and V-4Cr-4Ti (hydrogen distribution ratio R) increased with temperature, e.g., R was ≈ 17 at 400°C and ≈ 80 at 700°C. Desorption of hydrogen from V-4Cr-4Ti is a thermally activated process and the activation energy of the desorption rate is 0.405 eV.

INTRODUCTION

The ion density in fusion reactor designs is $1.2 \times 10^{20} \cdot \text{m}^{-3}$, which corresponds to a hydrogen pressure of $\approx 10^{-5}$ torr at room temperature. Interaction between hydrogen and first-wall components in a fusion reactor is not likely to produce hydrogen embrittlement of structural materials because the partial pressure of hydrogen in the system is low. However, it is important to determine the solubility of hydrogen in vanadium and liquid lithium and its rate of transport in these materials to optimize the design of a liquid-metal-cooled blanket in a fusion reactor. DeVan et al.¹ measured the solubility of hydrogen in V-5Cr-5Ti at hydrogen pressures of $6 \times 10^{-4} - 3 \times 10^{-2}$ torr by equilibration, followed by fast and slow cooling of specimens that were then analyzed for hydrogen. Cooling rate was found to be an important parameter; the concentration of hydrogen in slow-cooled specimens was higher by factors of 4–7 than that in specimens that were subjected to a fast cooling rate.

EXPERIMENTAL PROCEDURES

Several small tabular specimens of V-4Cr-4Ti ($1.0 \times 12.7 \times 75$ mm and ≈ 10 g, and $1.0 \times 4.0 \times 75$ mm and ≈ 3.4 g) were used in the hydrogen solubility/desorption rate studies. Similar studies were performed on liquid lithium (≈ 1.3 g) in a V-7.5Cr-15Ti tube (7.5 mm diameter, 0.4 mm wall thickness, with a mass of 6.829 g) that was sealed with V-20Ti end plugs (7 mm long, with a combined mass of 2.313 g) by tungsten-inert-gas welding in a helium environment. The sealed cells were heated in a vacuum at 700°C for 24 h to check for leakage of lithium; no leaks occurred. The outside surface of each capsule was sputter-coated with palladium.

Several experimental parameters, such as the volume of the vacuum chamber and steady-state flow rate of hydrogen as a function of pressure, were determined for the apparatus. Hydrogen pressure measured with an ionization gauge (IG) was lower by a factor of 0.44 than the actual pressure. The relationship between hydrogen flow rate and pressure in Fig. 1 is linear to $\approx 10^{-6}$ torr, and the data could be extrapolated with confidence to the 10^{-7} torr range.

The specimens were heated in a tantalum tube furnace (178 mm long, 12.7 mm diameter, 0.5 mm thick), which was installed horizontally in the vacuum chamber. The furnace could be heated to 850°C within 7 min by an AC current of ≈ 125 A. Pure hydrogen was admitted to the system by diffusion through a palladium membrane, and a steady-state pressure of 9.09×10^{-4} torr (IG = 4.0×10^{-4} torr) was maintained by a partially open turbo gate. After attaining the steady state, the pressure was monitored as a function of time with the turbo gate fully open to obtain hydrogen flow rate as a function of pressure in Fig. 1. The solubility of hydrogen in the samples was obtained from these data and by integration of the hydrogen evolution rate as a function of time. However, before measurements could be performed, sources of hydrogen in the system, such as the furnace, chamber, current supplying system, etc., had to be quantified relative to hydrogen contained in the specimens for which experimental data on hydrogen solubility (X_H) and desorption rate (J_H) were obtained. For the transport experiments, net flux of hydrogen from the samples can be written as

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$$J = J_{H, \text{total}} - J_{H, \text{system}} \quad (1)$$

where J is the net hydrogen desorption rate of the specimens and $J_{H, \text{system}}$ is the hydrogen desorption rate of the system, e.g., from the tantalum furnace, chamber wall, etc. The concentration of hydrogen in the specimens was obtained by integrating J between time limits 0 and ∞ , i.e.,

$$X_H = \int J(t) dt. \quad (2)$$

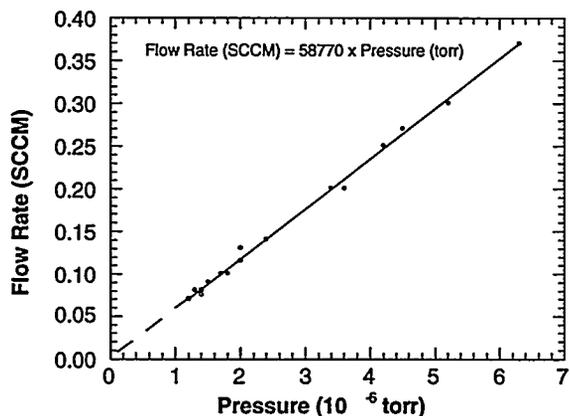


Figure. 1.
Hydrogen flow rate vs. pressure with turbo gate open.

In each experiment, the specimens attain an equilibrium hydrogen concentration at a given temperature. Then, the hydrogen partial pressure in the chamber is lowered by opening the turbo gate for 120 s, during which time most of the hydrogen desorbes at a system pressure of $\approx 10^{-6}$ torr. The remaining hydrogen in the specimens (and the tantalum furnace) desorbs as the furnace is heated to 860°C by an AC current of ≈ 125 A. Figures 2 and 3 are typical plots of temperature and pressure versus time, respectively. Figure 4 shows the amount of hydrogen released from 10 and 32.5 g of the alloy as a function of time during the desorption phase of the experiment. This amount was obtained by integrating hydrogen flux as a function of time. By comparing the results from two tests on specimens of different mass, we can obtain the net amount of hydrogen released by V-4Cr-4Ti. The difference in the amount of hydrogen released from the two sets of specimens is constant after 8.5 min (Fig. 4); however, the initial values are $\approx 1 \text{ cm}^3$ for a hydrogen pressure of 9.09×10^{-4} torr in the chamber. These results indicate that the relative experimental error may increase if this method is used with higher initial hydrogen pressures. Hydrogen solubility in liquid lithium was obtained in a similar manner.

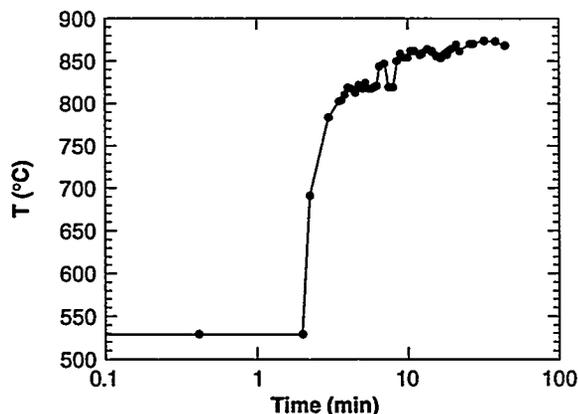


Figure 2.
Furnace temperature vs. time during isothermal and high-heating-rate conditions produced by AC current of 125 A.

Figure 5 shows the effect of temperature on the hydrogen concentration in lithium and V-(4,5)Cr-(4,5)Ti from this investigation and from Ref. 1, along with the predicted dependence on hydrogen in pure vanadium and lithium based on the data in Ref. 2, at a hydrogen pressure of 9.09×10^{-4} torr. Hydrogen concentrations in V-4Cr-4Ti are lower by two orders of magnitude than in pure vanadium. These results were used to calculate the ratio of the hydrogen concentration in lithium and vanadium and V-4Cr-4Ti, i.e., the distribution ratio as a function of temperature, as shown in Fig. 6. The ratios based on our experimental

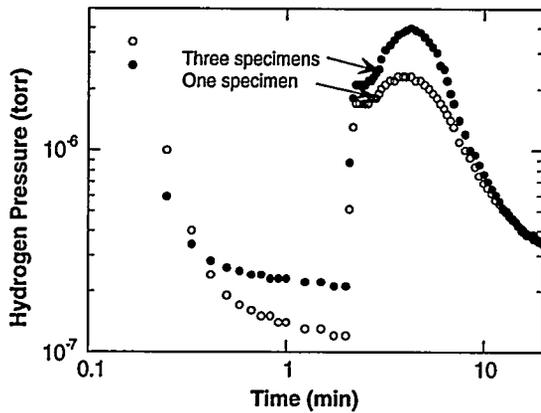


Figure 3.
Typical hydrogen pressure vs. time plot for 10 and 23.5 g of V-4Cr-4Ti.

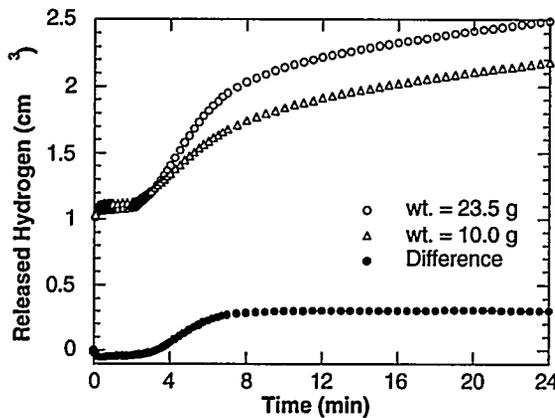


Figure 4.
Amount of hydrogen released vs. time during desorption, from integration of hydrogen flux as a function of time.

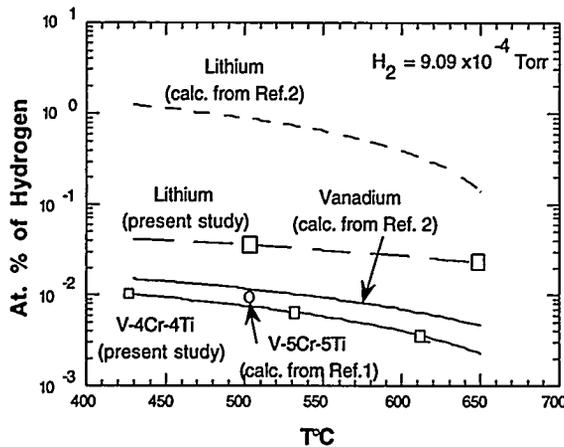


Figure 5.
Atom fraction of hydrogen in lithium, vanadium, and V-4Cr-4Ti vs. temperature at hydrogen pressure of 9.09×10^{-4} torr.

data increase as temperature increases from ≈ 17 at 400°C to ≈ 80 at 700°C . Distribution ratios based on hydrogen solubility data in lithium from Ref. 2 are also shown in the figure; they are higher by a factor of >10 than the present results and the values decrease as temperature increases.

The hydrogen desorption rate for V-4Cr-4Ti specimens, obtained from pressure vs. time plots (e.g., Fig. 3), is shown as function of reciprocal temperature in Fig. 7. Desorption of hydrogen from V-4Cr-4Ti is a thermally activated process with an activation energy of 0.405 eV.

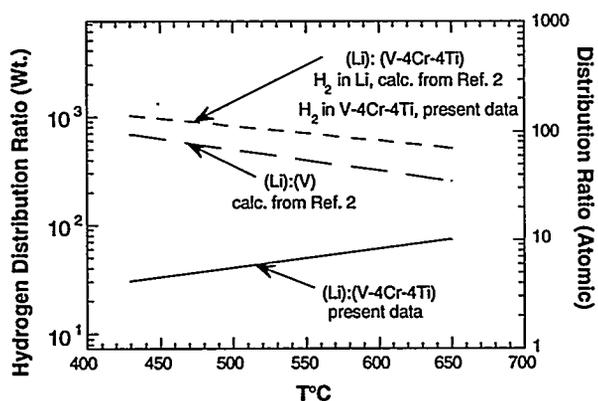


Figure 6. Calculated hydrogen distribution ratio between (a) lithium and V-4Cr-4Ti, from this study; (b) lithium and vanadium, based on data in Ref. 2; and (c) lithium and V-4Cr-4Ti, based on data for lithium in Ref. 2 and experimental data for the vanadium alloy from this study.

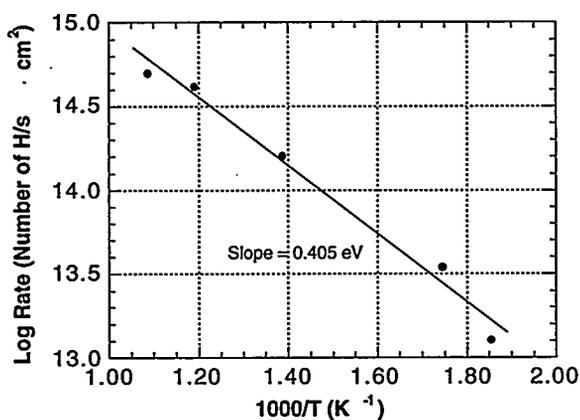


Figure 7. Hydrogen desorption rate for V-4Cr-4Ti vs. reciprocal temperature at hydrogen pressure of 9.09×10^{-4} torr.

CONCLUSIONS

Hydrogen solubility and desorption rate information for V-4Cr-4Ti and liquid lithium were obtained as a function of temperature at a hydrogen pressure of 9.09×10^{-4} torr. Hydrogen distribution ratios between the vanadium alloy and liquid lithium have been determined at 400-700°C at this hydrogen pressure.

ACKNOWLEDGMENTS

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