An atom-probe field-ion microscope study of 200-eV \( ^1\text{H}_2^+ \) ions implanted in tungsten at 29 K

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The atom-probe field-ion microscope (FIM) technique was used to study the low-temperature diffusive behavior of hydrogen (\( ^1\text{H} \)) in tungsten. Tungsten FIM specimens were implanted in situ with 200-eV \( ^1\text{H}_2^+ \) ions, at a specimen temperature of 29 K. The specimens were analyzed chemically on an atomic scale, via the atom-probe FIM technique, during the controlled pulse field evaporation of the (110) planes. No hydrogen events were detected at depths below the surface which corresponded to the calculated mean projected range of either \( ^1\text{H} \) or \( ^1\text{H}_2^+ \). The mean projected ranges were calculated employing a modified version of Biersack’s and Haggmark’s Monte Carlo program entitled TRIM (Transport of Ions in Matter). The experimental results set a lower bound of \( (1-10) \times 10^{-18} \text{ cm}^2 \text{ s}^{-1} \) on the diffusivity of hydrogen (\( ^1\text{H} \)) in tungsten at 29 K. This extremely high diffusivity of hydrogen in tungsten at 29 K, when compared with the value extrapolated from \( \sim 1100 \) K on an Arrhenius plot, suggests strongly that the diffusion of hydrogen in tungsten should be described by a nonclassical model. In addition, experiments were performed which demonstrated the adsorption of hydrogen on the surface of tungsten FIM tips (initially cleaned atomically by field evaporation) from the ambient pressure in the FIM.

I. INTRODUCTION

The interactions of low-energy hydrogen ions with refractory metals are both of fundamental and practical interest. Metals with high melting points are required as materials for the first wall of fusion power reactors. \(^1\) It has been demonstrated that hydrogen recycled between the first wall and the plasma and not the initial gas charge is the important fuel. \(^2\) Characterization of the diffusion and trapping of hydrogen in these materials is thus important. Furthermore, fundamental interest exists in the diffusion of hydrogen in bcc metals because transport may proceed via a nonclassical diffusion mechanism. \(^3\) However, it has been difficult to study the intrinsic lattice diffusion of hydrogen because trapping is often the rate-limiting step. \(^4\) In addition, hydrogen dissolves in the group VIB metals endothermically and thus its solid solubility in these metals is extremely small at room temperature. As a result, measurements of the diffusivity of hydrogen in tungsten have been limited to temperatures greater than \( \sim 1100 \) K. \(^5,7\)

These difficulties can be overcome with the atom-probe field-ion microscope (FIM) because a very small volume (\( \sim 10^{-16} - 10^{-17} \text{ cm}^{-3} \)) of highly perfect metal crystal can be studied and also because the crystal can be both imaged and chemically analyzed directly. Because it has these capabilities, the atom-probe appears to be uniquely capable of yielding information about hydrogen diffusion in the group VIB metals at low temperatures. \(^8\)

In this paper we present the results of experiments concerning the diffusion of hydrogen (\( ^1\text{H} \)) in tungsten. In recent years, the Cornell atom probe has been applied to the study of helium (\( ^3\text{He} \) and \( ^4\text{He} \)) in tungsten. Specifically, tungsten specimens which had been implanted in situ with helium ions have been dissected plane by plane to yield ion-range profiles. \(^9,10\) The enthalpy of migration of both \( ^3\text{He} \) and \( ^4\text{He} \) was determined to be \( \sim 0.28 \) eV. \(^9\) Results for 200-eV \( ^1\text{H}_2^+ \) implantations of tungsten specimens with a (110) orientation, in the same atom probe, are reported in this paper.

The minimum transferred energy necessary to create a stable Frenkel pair in tungsten is \( \sim 43 \) eV. \(^11\) However, the maximum energy that can be imparted to a tungsten atom by a 200-eV \( ^1\text{H}_2^+ \) molecule is only 8 eV in a head-on two-body elastic collision. Therefore, radiation damage could not have been created by any of the implantations made in the present experiments and the present results apply to the perfect, that is, defect-free lattice. This is an extremely important point because hydrogen can be strongly trapped at a lattice defect. If this had occurred, then it would not have been possible to observe the intrinsic diffusivity of hydrogen in tungsten.

As part of the present work, extensive TRIM Monte Carlo simulations of the hydrogen ion implantations were made for the purpose of comparing to our experimental data. \(^12,13\)

In the body of the paper, experimental details are discussed in Sec. II; TRIM code modifications and simulation results are presented and discussed in Sec. III; experimental results are presented in Sec. IV; an analysis to extract a range of values for the diffusivity of hydrogen in tungsten is presented in Sec. V; a discussion is given in Sec. VI; and a summary in Sec. VII.
II. EXPERIMENTAL DETAILS

The experiments were conducted in the Cornell atom probe. The atom probe has a straight flight tube and is computer controlled. Specimens were pulse field evaporated by applying a steady-state voltage plus a voltage pulse; the latter was 0.1 of the steady-state voltage. The field evaporation rate was, typically, a few atoms per second. The temperature of the specimen stage was controlled to within \( \pm 2 \)°. Temperatures were measured with a two-lead Rosemount platinum resistance thermometer. Specimens were prepared from 0.13-mm-diam Materials Research Corp. VP grade polycrystalline wires. These wires had highly elongated grains which were parallel to the [110] direction. The tungsten wires were annealed at 2000 °C for 1 h in ultrahigh vacuum (UHV) \( (10^{-9} \text{ Torr}) \) in order to obtain a grain diameter that was very much greater than the maximum profiling depth. The wires were electroetched into fine needlelike specimens in a solution of 1 M NaOH at 4.3 V ac. After insertion of the specimen, the stainless-steel atom probe was baked at 120 °C for 12 h in order to obtain UHV. Base pressures were \((2-5) \times 10^{-10} \text{ Torr} \) and were read by a Bayard-Alpert ionization gauge calibrated for N\(_2\). Specimens were imaged in \((1-3) \times 10^{-5} \text{ Torr} \) helium bled in from a 1-liter flask of Matheson research purity helium. Specimens were then field evaporated until they had a roughly hemispherical endform. A typical specimen radius was 250 Å. Results are reported here for measurements performed on perfect single-crystal volumes \((10^{-16} - 10^{-15} \text{ cm}^3)\) of the polycrystalline specimens. Only specimens exhibiting perfectly regular field-ionization images were employed. A micrograph of one of the specimens used in the present experiments is shown in Fig. 1. The time-of-flight mass spectrographic results themselves were used for impurity analyses. The number of events not clearly identifiable as either W, H, or He was typically 0.003 of the total number of events observed.

Implantations were made with a differentially pumped ion-beam system that incorporated an analyzing magnet to produce a monoisotopic ion beam. All implantations were made normal \((\pm 5 \degree)\) to the (110) plane. Typically, the gauge pressure in the specimen chamber was \((1-4) \times 10^{-7} \text{ Torr} \) during an implantation. Hydrogen gas was admitted directly into the ion gun from a 1-liter flask of Matheson high-purity hydrogen.

In these experiments, the probe hole was positioned over the field-ionization image of the central [110] pole. For sufficiently blunt specimens a characteristically stepped behavior was observed in plots of the cumulative number of tungsten events versus the cumulative number of field evaporation pulses. This is illustrated in Fig. 2. Depth distributions for hydrogen were obtained by counting the number of \(^1\text{H}\) events field evaporated per plane. The depth of each \(^1\text{H}\) from the irradiated surface was determined from the known 2.2-Å interplanar spacing between the (110) planes. Before presenting our results it is convenient to first discuss the Monte Carlo simulations of hydrogen implantation.

III. TRIM SIMULATIONS

We have used Biersack's and Hagmark's TRIM (Transport of Ions in Matter) code modified to simulate hydrogen implantations of tungsten. The TRIM code simulates ion implantations of solids by following the history of a projectile through its successive collisions with atoms of the target, that is, in the binary collision approximation. An amorphous solid was assumed and, therefore, crystal lattice ef...
ffects were not incorporated. By following a large number of projectile histories, the program determines distributions for both the total path length and the projected range. Particle reflection coefficients were also obtained.

The original TRIM was written to apply to higher values of projectile energy than were of interest in the present experiments. To fulfill the present need, the code was modified so that the distance between collisions was fixed to be the mean interatomic separation reduced by \( p \tan(\theta/2) \), where \( p \) is the impact parameter and \( \theta \) is the polar scattering angle in the center-of-mass frame of reference. The reduction yielded the hard-core value for the time integral of Robinson and Torrens.\(^{17}\) Inelastic energy losses were determined using the electronic energy-loss expression postulated by Oen and Robinson.\(^{18}\) In addition, recursion relations for all three cartesian directions permitted the simulated ion trajectories to be plotted.\(^{13}\) Three trajectories of 100-eV \( ^1\text{H}^+ \) ions impinging on tungsten are exhibited in Fig. 3.

Neither of the two low-energy modifications changed the mean projected ranges by more than 10 Å. The total pathlength (a quantity not accessible experimentally) was, however, found to depend sensitively on which inelastic energy-loss expression was used. Total pathlengths obtained using the Oen and Robinson expression were significantly longer than those obtained using the Lindhard–Scharff electronic energy-loss expression.\(^{19}\) This finding is in agreement with that of Eckstein et al.\(^{20}\) Mean projected range results for the simulation of both 1- and 2-amu projectiles are shown in Fig. 4. Particle reflection coefficients are shown in Fig. 5. Results for both 1- and 2-amu projectiles were obtained to permit estimates of the optimum implantation parameters for our experiments. Two limiting cases can be discerned: (i) the \( \text{H}_2^+ \) molecule did not break up during its collisions with tungsten atoms; and (ii) the molecule broke up at the surface and each hydrogen atom received half of the incident energy. For case (i) the results for mass = 2 amu at 200 eV would apply—55 Å for the mean projected range and 0.60 for the particle reflection coefficient. For case (ii) the results for mass = 1 amu at 100 eV would apply 37 Å for the mean projected range and 0.74 for the particle reflection coefficient.

IV. ATOM-PROBE FIM RESULTS

Results of the plane-by-plane analyses made at 29 K for a specimen implanted to a fluence of \( 3 \times 10^{15} \) ions cm\(^{-2} \) are shown in Fig. 6. A total of 27 (110) planes were removed from the tungsten specimens subsequent to an irradiation normal to the (110) plane, that is, along the axis of the specimen. The total number of events with a mass-to-charge ratio \( (m/n) \) of unity recorded during the field evaporation of each plane are shown versus the plane number in this histogram. Very few \( m/n = 2 \) amu events were detected in these experiments. No inference about the atomic versus molecular state of the hydrogen could be made from this observation since \( \text{H}^+ \) is known to strongly dominate as a field evaporation

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**FIG. 3.** Two computer generated plots of a Monte Carlo simulation. The trajectories of three 100-eV \( ^1\text{H}^+ \) ions in tungsten are shown in (a) as seen along the ion beam, which impinges at normal incidence on the plane surface, and in (b) as seen from the side. All four ions impinged at the point indicated. Three were reflected and one was implanted at a depth of 27 Å.

**FIG. 4.** Monte Carlo simulation results for projected mean ranges and straggling of 1- and 2-amu ions in tungsten. Curves were drawn to guide the eye. Scatter in the simulation results is due to a smaller number of ion histories in some cases than in others.
product.\footnote{We have neglected the occurrence of tungsten-
hydride field evaporation products. These were not resolvable
in the time-of-flight spectra because of energy deficits.
Nevertheless, the copious occurrence of events with $m/n = 1$ amu
indicates that tungsten-hydride ions were dissociated
during the process of field evaporation.}

We note that the TRIM simulations predicted mean pro-
jected ranges of 25 \((110)\) planes for case (i) above and 17 \((110)\)
planes for case (ii). No hydrogen events were observed in or
near these planes.

The hydrogen events detected in planes one through ten
can be explained as arising from the adsorption of hydrogen
from the background gas in the FIM. This is demonstrated in
Fig. 7 which shows results obtained for exposures of atom-
ically clean surfaces (cleaned by field evaporation) to a
background ambient of $6 \times 10^{-10}$ Torr pressure. This back-
ground pressure was typically present before the atom-probe
analyses of implanted specimens were carried out. The null
exposure was performed by decreasing the steady-state dc
voltage and pulse voltage to zero and then immediately in-
creasing these voltages to values which were sufficient to
prevent hydrogen adsorption. Some hydrogen can be ad-
sorbed at nonzero values of the applied voltage, and this
accounts for the nine events observed during the null ex-
pose. The 9 min exposure was conducted with 0 V applied
for this period. The similarity between this histogram (nine min-
utes exposure) and the one in Fig. 6 led us to the conclusion
that adsorption of hydrogen from the background resulted
in the hydrogen events depicted in Fig. 6. If the particle re-
fection coefficient had been <0.75, as predicted by the sim-
ulations, then $>300$ hydrogen atoms should have been im-
planted into the volume of specimen analyzed.

We conclude from these results that hydrogen is mobile
in tungsten at 29 K and that it had all diffused out to the
surface, which is the dominant sink. We have made the same
measurements at lower temperatures, and we then observed
hydrogen events in planes corresponding to the bulk of the
specimen. However, similar results were obtained in control
experiments at lower temperatures in which the ion beam
was blocked from hitting the specimen. These deep events
were due to the field ionization of the ambient hydrogen gas.
The supply of $H_2$ from the background gas was sufficiently
enhanced at temperatures below 29 K to interfere with the
measurement of the implanted depth profile. In a separate

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{fig5}
\caption{Monte Carlo simulation results for the particle reflection coef-
fi cients of 1- and 2-amu ions in tungsten. Curves were drawn to guide the eye.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{fig6}
\caption{Atom-probe FIM data: the number of hydrogen \((^1H)\) events detected
per \((110)\) plane. The \(^1H\) events detected were adsorbed from the back-
ground (see Fig. 7). Implanted ions were not found.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{fig7}
\caption{Atom-probe data: the number of \(^1H\) events detected per \((110)\) plane
for controlled background adsorption. The results of two adsorption
experiments, one of 9-min duration and the other for a null exposure (defined in
the text), are shown.}
\end{figure}
Historically, hydrogen diffusivity values have not compared well from laboratory to laboratory. Trapping of hydrogen at defects and impurity atoms can seriously affect such measurements and are believed to be responsible in part for the discrepancies. This conclusion is supported by the experimental finding that molybdenum and niobium damaged by ion bombardment retained enhanced hydrogen concentrations. Occasional trapping also influenced our data. Infrequently, we did observe a number of hydrogen events in planes corresponding to depths far below the initial specimen surface in experiments conducted at temperatures above 29 K. In one case, an irregularity in the field-ion image was associated with such events; weak specimens were sometimes partly deformed plastically by the mechanical stresses associated with the large electric evaporation field (5.5 V Å⁻¹), and for such a specimen an apparent hydrogen depth profile was observed. Reproducible hydrogen depth profiles were, however, never observed. We attributed apparent depth profiles in those few instances in which deep events were observed at temperatures above 29 K to trapping of hydrogen at defects.

V. THE DIFFUSIVITY OF HYDROGEN IN TUNGSTEN AT 29 K

In this section we make two simple model calculations in order to obtain a range of values for the diffusivity of hydrogen in tungsten at 29 K. The first calculation employs a spherical specimen (radius = R) with an assumed uniform concentration c0(d) at the initial time (t = 0) and subject to the boundary condition that the concentration of hydrogen at the surface is zero for t > 0; the principal term in the solution of the diffusion equation for the hydrogen concentration as a function of time is

\[ \frac{c(t)}{c_0} = \exp\left(-\frac{\pi^2 D_{1\text{H}} t}{R^2}\right). \]

Here \( D_{1\text{H}} \) denotes the diffusivity of hydrogen. We have found that at 29 K no deep hydrogen events were detected for 300 total implanted events, in an experiment that lasted 4 h and 41 min. This is taken to be the isothermal annealing time. The following inequality held at the end of this time:

\[ \frac{c}{c_0} < 1/300. \]

This can be seen to hold since for \( c/c_0 \) greater than or equal to 1/300 we would have detected an event. Since the specimen radius was 275 Å, we obtain from Eqs. (1) and (2):

\[ D_{1\text{H}} > 10^{-17} \text{ cm}^2 \text{ s}^{-1}. \]

The assumption of an initially uniform distribution of hydrogen implies that \( 10^{-17} \text{ cm}^2 \text{ s}^{-1} \) is an approximate lower-bound to \( D_{1\text{H}} \).

A second approximate calculation of \( D_{1\text{H}} \) is based on the fact that the initial distribution of \(^1\text{H}\) is nonuniform—it is most likely a skewed Gaussian distribution, as is the case for low-energy implanted \(^3\text{He}\) and \(^4\text{He}\) ions in tungsten. For the two limiting implantation cases discussed in the last paragraph of Sec. III we calculated mean projected ranges of 37 and 55 Å. The root-mean-squared diffusion distance \((\langle X^2 \rangle)^{1/2}\) is equal to \((6D_{1\text{H}} t)^{1/2}\). Hence, equating \((\langle X^2 \rangle)^{1/2}\) to the mean projected ranges we obtain

\[ D_{1\text{H}} = (1-3) \times 10^{-18} \text{ cm}^2 \text{ s}^{-1}. \]

The value of \( 10^{-18} \text{ cm}^2 \text{ s}^{-1} \) is another approximate lower bound to \( D_{1\text{H}} \).

The two simple model calculations employed above indicate that \( D_{1\text{H}} \) at 29 K is at least \((1-10) \times 10^{-18} \text{ cm}^2 \text{ s}^{-1}\). An extrapolation of the available high-temperature data for the diffusivity of hydrogen in tungsten or molybdenum—on an Arrhenius plot—to 29 K indicates that hydrogen should be completely immobile in tungsten at 29 K. The lowest temperature at which the diffusivity of hydrogen in tungsten had been measured, prior to the present investigation, was \( \sim 1100 \text{ K} \). Thus the present results indicate a nonlinear Arrhenius plot over this large temperature range, with an extremely large value of \( D_{1\text{H}} \) at 29 K.

VI. DISCUSSION

The principal result of the present investigation is the very high mobility of hydrogen in tungsten at 29 K—\( D_{1\text{H}} \) is in the range \( (1-10) \times 10^{-18} \text{ cm}^2 \text{ s}^{-1} \) at this temperature. This result was deduced from the observation that no hydrogen was detected in FIM specimens which had been implanted \( \text{in situ} \) at 29 K with 200-eV \(^1\text{H}_2^+\) molecules. The maximum energy that can be transferred to a tungsten atom by a 200-eV \(^1\text{H}_2^+\) molecule is only 8 eV in a head-on two-body elastic collision. In the case of tungsten the maximum transferred energy required to produce a stable Frenkel pair is 43 eV. Hence, the act of implantation could not have created any radiation damage. The small volume \( \sim 10^{-16}-10^{-17} \text{ cm}^3 \) of a FIM specimen reduced strongly the probability of the existence of any preimplantation lattice imperfections. Thus, once the implanted hydrogen found itself in a defect-free lattice—that is, a perfect lattice—it was able to diffuse to the surface of the FIM specimens, as it could not be trapped by lattice imperfections.

To date, we are not aware of any previously reported atom-probe FIM results concerning \(^1\text{H}_2^+\) implanted in tungsten. However, depth profiles of deuterium implanted into (110)-oriented tungsten at 80 eV were obtained using the imaging atom-probe field-ion microscope. The mean range of the deuterium implanted into specimens held at a temperature of 80 K was determined to be 49 Å. Our results and Panitz's finding that deuterium is immobile in tungsten at 80 K imply that the diffusivity of hydrogen in tungsten is strongly dependent on the isotopic mass, with the lighter \(^1\text{H}\) isotope diffusing faster than the heavier \(^2\text{D}\) isotope. This is consistent with theoretical results obtained by Emin et al.

Picraux and Vook have used ion channeling and nuclear-reaction analyses to show that deuterium implanted in tungsten at 296 K occupies tetrahedral interstitial positions. The \( \text{D}^+ \) implantations were made at an ion energy of 30 keV. At this ion energy the energy transferred to a tungsten atom
is well above the minimum threshold energy of 43 eV required to produce a stable Frenkel pair.\textsuperscript{11} Hence, the implanted deuterium could most certainly have been trapped at vacancies, which are known to be completely immobile in tungsten\textsuperscript{28} at 296 K. At this temperature self-interstitial atoms are highly mobile in tungsten.\textsuperscript{24,29} Thus it is also possible that the highly mobile hydrogen atoms may have formed immobile complexes with the self-interstitial atoms—this possibility cannot be ruled out. In conclusion, the detection of immobile deuterium by Picraux and Vook at 296 K is not inconsistent with our observation that hydrogen is highly mobile at 29 K, as the Picraux and Vook experiments were made in the presence of radiation damage, whereas our experiments were performed in a defect-free lattice.

The extremely high diffusivity of hydrogen in tungsten at 29 K, when compared with the value extrapolated from \(~1100\) K, suggests strongly that the diffusion of hydrogen in tungsten should be described by a nonclassical model. Flynn and Stoneham\textsuperscript{30} have developed a theoretical expression for the phonon-assisted interstitial hopping rate at low temperatures ($T < \frac{1}{2} \theta_D$ where $\theta_D = 310$ K is the Debye temperature of tungsten), which has a $T^7$ temperature dependence. The quantum mechanical calculation which produced this result was based on the theoretical treatment of the hopping rate of self-trapped electrons (small polarons). The theory of small polaron hopping was first developed to describe tunneling of electronic charge carriers in low-mobility semiconductors and insulators.\textsuperscript{31} Emin \textit{et al.}\textsuperscript{26} have shown recently that discrepancies between predictions of the small polaron treatment and measured diffusivities can be reconciled by generalizing the electronic conduction theory, in a way which transcends simplifications of the small polaron treatment which are not appropriate to the diffusion of light interstitials in bcc metals. Specifically, diffusion coefficients for hydrogen and deuterium in niobium, which are consistent with the available data, were obtained.

In view of the fact that we only have a lower limit for the diffusivity of hydrogen in tungsten at one temperature, 29 K, we do not make an explicit comparison with theory. However, we note that both the treatments of Flynn and Stoneham\textsuperscript{30} and Emin \textit{et al.}\textsuperscript{26} predict a high-temperature Arrhenius regime and a low-temperature nonclassical regime.\textsuperscript{32} Our experimental results suggest strongly that the diffusion of hydrogen in tungsten at 29 K is in this low-temperature nonclassical regime.

**VII. SUMMARY**

We have used an atom-probe field-ion microscope to determine a range of values for the diffusivity of hydrogen ($^1\text{H}$) in tungsten at 29 K. This was accomplished by implanting specimens \textit{in situ} with 200-eV $^1\text{H}_2^+$ ions and subsequently dissecting the specimens on an atomic scale via pulse field evaporation. No implanted hydrogen atoms were detected for experiments conducted at 29 K. Hydrogen events were, however, detected during the field evaporation of the first few (110) planes. These events were found to result from the adsorption of the ambient hydrogen in the atom probe onto the specimens. In addition, Monte Carlo simulations of hydrogen ion implantations were carried out. The simulations predicted mean projected ranges between 37 and 55 Å. No hydrogen events were detected at these depths below the implanted surface, and we concluded that all the implanted hydrogen had diffused to the surface of the specimen. These results yield a range of $[1 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}]$ for a lower bound for the diffusivity of hydrogen in \textit{perfect} single crystals of tungsten at 29 K. An extrapolation of the existing data on the diffusivity of hydrogen in tungsten from \(~1100\) to 29 K coupled with the high diffusivity of hydrogen in tungsten at 29 K suggests strongly that the diffusion of hydrogen in tungsten at this temperature should be described by a nonclassical model.

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