AN IN-SITU FIELD ION MICROSCOPE STUDY
OF IRRADIATED TUNGSTEN AND TUNGSTEN ALLOYS:
I. THE VOLUME CHANGE OF MIGRATION OF THE STAGE I
SELF-INTERSTITIAL ATOM

by

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IRRADIATED TUNGSTEN AND TUNGSTEN ALLOYS:

I. THE VOLUME CHANGE OF MIGRATION OF THE STAGE I

SELF-INTERSTITIAL ATOM*

Kenneth L. Wilson^{§†} and David N. Seidman[†]

ABSTRACT

A detailed study has been performed of the effect of the electric field ($\sim 4.75 \text{ VA}^{-1}$) required for field ionization of helium gas atoms, in the field ion microscope (FIM), on the isochronal recovery spectrum of ion (30 keV W^+) irradiated tungsten. All the irradiations were performed in-situ under ultra-high vacuum conditions ($< 10^{-9}$ torr) to a constant dose of $5 \cdot 10^{12}$ ion cm^{-2} . The FIM specimens used were prepared from oriented single crystals of high purity tungsten with a resistivity ratio of $5 \cdot 10^4$. The experimental procedure involved a comparison of the isochronal recovery spectra in the range 18 to 120K of specimens irradiated at 18K in the absence of both the electric field and the imaging gas with identical specimens which were irradiated at 50K and then cooled to 18K in the absence of the electric field. The results of these recovery experiments demonstrated that the field of $\sim 4.75 \text{ VA}^{-1}$ has only a minimal effect on the annealing kinetics of self-interstitial atoms (SIA's) and that the volume change of migration of the Stage I SIA is less than 0.02 atomic volume. This result strengthens our contention that SIA long-range migration occurs at $\sim 38\text{K}$ and that Stage I recovery is certainly completed by $\sim 45\text{K}$ for tungsten. This result is in contradistinction to other investigations which have assigned SIA long-range migration in tungsten to the 70 to 100K range.

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1. INTRODUCTION

The field ion microscope (FIM) technique^(1,2) offers a unique instrument for the direct measurement of the volume change of migration (Δv_{li}^m) of a self-interstitial atom (SIA). The FIM involves the use of an electric field (E) between 4 and 6VÅ⁻¹ (3,4) for the field ionization of the imaging helium gas atoms and for the field evaporation process⁽⁵⁾ required to form the highly perfect surface which characterizes an FIM image. This value of E generates a negative hydrostatic pressure (p) in the tip volume [$\sim(0.1 \text{ to } 1) \cdot 10^{-16} \text{ cm}^3$] which is approximately given by $E^2/8\pi$ ⁽⁶⁾. This negative p dilates the lattice and changes the enthalpy change of migration (Δh_{li}^m) of an SIA by amount that depends on the value of $p\Delta v_{li}^m$.

The relevant thermodynamic relationship between all the physical quantities involved is

$$\Delta h_{li}^m = \Delta u_{li}^m + p\Delta v_{li}^m, \quad (1)$$

where Δu_{li}^m is the internal energy change of migration of an SIA; for Δv_{li}^m positive* and p negative equation (1) becomes

$$\Delta h_{li}^m = \Delta u_{li}^m - |p|\Delta v_{li}^m = \Delta u_{li}^m - \frac{E^2}{8\pi}\Delta v_{li}^m. \quad (2)$$

The physical effect of this decrease in Δh_{li}^m is to increase the diffusivity of an SIA and hence to make the SIA mobile at temperatures where, in the absence of E, it would be immobile. The diffusivity (D_{li}) of an SIA is given by

$$D_{li} = \xi a_o^2 \nu \exp [-\Delta g_{li}^m/kT], \quad (3)$$

*

The assumption of a positive Δv_{li}^m is consistent with both the isochronal resistivity recovery experiments on electron irradiated tungsten and our FIM experiments. See Section 4.2 for a discussion of this point.

where ξ is a number which depends on the geometry of the SIA*, a_0 is the lattice parameter, ν is the frequency with which an SIA at the saddlepoint goes to a new site and Δg_{1i}^m is the Gibbs free energy change of migration. Employing a Grüneisen model and also using the thermodynamic relationship

$$\left[\frac{\partial \Delta g_{1i}^m}{\partial p} \right]_T = -\Delta v_{1i}^m$$

it can be shown that the ratio (η) of the diffusivity of an SIA in a stressed FIM tip ($E \neq 0$) to the diffusivity in an unstressed tip ($E=0$) is given by⁽⁷⁾

$$\eta = \exp \left[\left(\frac{2}{3} - \gamma \right) \chi \frac{E^2}{8\pi} \right] \exp \left[\frac{\Delta v_{1i}^m}{kT} \frac{E^2}{8\pi} \right] = \exp \left[\frac{\Delta v_{1i}^m}{kT} \frac{E^2}{8\pi} \right], \quad (4)$$

where γ is the Grüneisen constant, χ is the lattice expandability and kT has its usual significance. Thus the effect of E is to increase the diffusivity of an SIA and the magnitude of this effect at any T is determined by $|p| \Delta v_{1i}^m$ or alternatively by $E^2 \Delta v_{1i}^m / 8\pi$.

In this paper (Part I of a four part series) experimental results are presented which show that the value of Δv_{1i}^m for the SIA that undergoes long range migration in Stage I of irradiated tungsten is < 0.02 atomic volume**. This result implies that the Δh_{1i}^m value of 0.085eV measured earlier by Scanlan et al.⁽⁷⁾, employing the FIM technique, for the long range migration recovery peak in tungsten only differs from Δu_{1i}^m by < 0.02 eV. This is an important result because it shows that the FIM isochronal recovery spectra may be compared with

* For a three-dimensional SIA ξ is equal to 1/6 while for a crowdion ξ is equal to 1/2. The results of the present paper are independent of the dimensionality of the Stage I SIA.

** Hereafter, an atomic volume is denoted by Ω_a where $\Omega_a = a_0^3/2$ for a b.c.c. metal and $a_0^3/4$ for a f.c.c. metal.

the resistivity isochronal recovery spectra of irradiated tungsten (e.g., see the work of Kunz⁽⁸⁾). It also demonstrates that the p required to produce a significant $p\Delta v_{11}^m$ effect in the case of SIA's is so large that this effect can be neglected in practical situations.

2. EXPERIMENTAL DETAILS

2.1 The irradiation facility and the ultra-high vacuum FIM

The experimental system consisted of a Hill-Nelson sputtered-metal ion source⁽⁹⁾ connected to an FIM by a three-stage differentially pumped flight tube. This system was the one originally employed by Scanlan et al.⁽¹⁰⁾, but several modifications have been made since the time of that publication. First the original ion source was replaced by a commercial Hill-Nelson sputtered metal ion source. This unit produces a flux of $\sim 10^{12}$ W^+ ion $cm^{-2}sec^{-1}$ at the specimen position with an operating pressure of $\sim 8 \cdot 10^{-6}$ torr of argon measured above the diffusion pump which evacuated this source. The ion beam was translated vertically by a parallel plate beam steering assembly and was switched and chemically analyzed magnetically in a horizontal plane by a commercial electromagnet. Second, the flight tube was modified by replacing the second stage titanium sublimation pump (TSP) with a 50 l sec^{-1} ion pump (see Fig. 1 in Scanlan et al.⁽¹⁰⁾). During an irradiation the FIM head was maintained at a pressure of $1 \cdot 10^{-9}$ torr or lower. Third, in the final stage of the flight tube just before the FIM head, a beam raster consisting of two sets of parallel plates was added. Fourth, the differential pumping aperture to the FIM head was removed to allow a more divergent beam to reach the specimen. The horizontal plates of the raster had a 500 V peak-to-peak $5 \cdot 10^3$ Hz sawtooth wave form placed on them, while the vertical plates received a 300V peak-to-peak $5 \cdot 10^2$ Hz sawtooth signal. In this manner an area of $1 cm^2$ centered on the beam axis at the tip received at least 50% of the maximum flux as measured via a Faraday cage.

The Faraday cage, located on the beam axis after the FIM tip, had a 3mm diameter grounded aperture and also a plate biased at -300Vdc with respect to the target to suppress secondary electron emission. This combination of beam raster and Faraday cage gave a more accurate measurement of dose than the arrangement previously used. Finally, an oil diffusion pump vacuum system was added to the FIM head which allowed either dynamic or static imaging conditions. No differences have been observed in the recorded helium ion images between the static and dynamic operating modes. The helium imaging gas was bled into the FIM head at $3 \cdot 10^{-5}$ torr gauge pressure through a Varian UHV leak valve from a glass flask containing ultrapure helium.

The FIM also had a liquid helium cryostat mounted on a bellows to allow for alignment of the specimen with respect to the heavy metal ion beam. Specimen cooling was accomplished by the continuous flow method⁽¹¹⁾, allowing any specimen temperature between ~ 6 and 300K to be achieved. Temperature measurement was made via a miniature platinum resistance thermometer (PRT) mounted at the bottom of the cryostat. The calibration performed by Scanlan et al.⁽¹⁰⁾ was found to be still valid with a ± 0.1 K maximum variation over the range from 15 to 120K.

2.2 Data recording and ciné film analysis

All the FIM images were recorded on 35mm film employing an Automax ciné camera⁽¹²⁾ with the aid of an internal image intensification system based on a Bendix 76mm channel electron multiplier array^(13,14). The ciné film recorded was analyzed with the aid of a Vanguard motion analyzer⁽¹²⁾.

2.3 Electropolishing techniques

The starting material was a single crystal rod of 4-pass zone refined tungsten with a diameter of ~ 1.5 mm and a length of 20 cm. This rod had a resistivity ratio R ($R = \rho_{273K} / \rho_{4.2K}$) of $5 \cdot 10^4$ where ρ represents resistivity. This value of R was uncorrected for any possible specimen size effects. The

first step consisted of dc electropolishing the rod down to an ~ 0.5 mm diameter rod in a solution of 20 gm NaOH electropolishing solution with 9 Vdc on the tungsten specimen with respect to a stainless steel cathode. Polishing was most rapid at the air-electrolyte interface, so that the rod was cut off before it was completely polished away.

The final step employed the drop-off technique⁽¹⁵⁾ to produce an FIM tip. The specimen was suspended vertically in a layer of 1N NaOH solution that was floated on CCl_4 . A 9 Vdc potential was again applied to the tungsten specimen with respect to a stainless steel cathode. The specimen was translated up and down vertically until a slight taper was produced for ~ 2 mm. Then 1 mm of the rod was lowered into the CCl_4 and the entire rod was oscillated slowly through a distance of ~ 0.1 mm. After one minute the rod had a taper with a 1 mm length of an oversized weight on the end; all but 0.2 mm of this weight was removed by dipping it into the electrolyte. The specimen was then immersed for a length of ~ 5 mm into the NaOH solution and the necked region above the weight was allowed to thin. When this neck had almost propagated across the rod completely the DC voltage was switched off and a hand switch was placed in series with a power supply. Voltage pulses with a height of 9 Vdc and 100 msec duration at a repetition rate of 1 Hz were applied to the tip until the weight was observed to drop-off. The tip was then cleaned in distilled water.

The drop-off technique has been criticized by Müller and Tsong⁽¹⁶⁾ because of the high stress that is present in the necked region before the weight drops off. Alternatively, in our experience only about 5 to 10% of the tips produced by the drop-off technique showed evidence of plastic deformation when imaged. It appears as if the back-polishing which occurred during the last 100 msec pulse after drop-off was sufficient to remove any traces of plastic deformation in the tip region.

2.4 Procedure for a low temperature irradiation

The specimen was first field evaporated at room temperature to the desired tip radius, typically 350Å^o; this large tip radius was needed to give a large visible surface area for the isochronal warming experiments. The specimen was then cooled down to the irradiation temperature (T_i) and further field-evaporation was performed in order to produce a true low-temperature end-form. The irradiation was then performed in-situ in the absence of both E and the - imaging gas at a background pressure of $<1 \cdot 10^{-9}$ torr. The voltage was then removed from the specimen for ~20 minutes during the irradiation. After the irradiation the helium imaging gas was reintroduced into the FIM and the specimen was brought back to the Best Image Field (BIF)⁽¹⁷⁾ characteristic of T_i . The side of the tip exposed to the heavy metal ion beam exhibited evidence of sputtering [see Fig. 1(A)], while the other shielded half of the tip's surface remained almost atom-for-atom perfect. Figure 1(A) shows a typical FIM image after an irradiation* to a dose of $5 \cdot 10^{12}$ ion cm⁻² with 30 keV W⁺ ions. The clock, frame counter and film title are also seen in Figs. 1(A) and 1(B). Figure 1(B) shows the same specimen after 5Å^o of tungsten had been removed from the specimen by pulse field evaporation. A specimen that had not been subjected to E for 20 minutes and had not been irradiated exhibited an atomically perfect surface when re-imaged.

After at least 5Å^o of tungsten had been removed the pulse amplitude was slowly diminished to zero and the camera was set to operate at 0.5 frame sec⁻¹. The specimen was then warmed isochronally at a rate ~2 to 2.5K min⁻¹ by decreasing the flow rate of helium gas to the cryostat⁽¹¹⁾. The tip temperature was

* The initial state of damage, after an irradiation at 18K to a dose of $<5 \cdot 10^{12}$ ion cm⁻², consisted of immobile SIA's and a few depleted zones close to the irradiated surface (see Scanlan et al.⁽¹⁰⁾, Beavan et al.⁽¹⁸⁾ and Seidman⁽¹⁹⁾).

monitored as a function of time with the aid of the PRT and this was correlated to each frame of 35mm ciné film by means of the clock and frame counter (Fig. 1). The specimen was maintained at the BIF characteristic of 18K during each isochronal warming experiment. This procedure helped to enhance the contrast effects caused by an SIA.

In the isochronal warming experiments the arrival of an SIA at the surface of an FIM specimen was characterized by the appearance of an extra-bright spot (e.g., see Fig. 4 in Robinson et al.⁽²⁰⁾). This contrast effect was not transient, but usually remained until the specimen temperature was above ~110K. Above 110K very few extra bright spots remained and this is one of the main reasons for the termination of the isochronal recovery experiment at 120K. The other main reason is the poorer image quality at these elevated specimen temperatures[†].

3. EXPERIMENTAL PROCEDURE AND RESULTS

The basic experimental procedure and the results obtained to determine the effect of E on Δh_{11}^m were as follows:

1. A series of FIM tips were electropolished from the four-pass zone-refined tungsten rod and irradiated in-situ with 30keV W^+ ions at $T_i = 18K^*$ to a dose of $\sim 5 \cdot 10^{12} W^+ \text{ ion cm}^{-2}$, in the absence of both E and the imaging gas. The electric

[†] The atomic resolution of an FIM decreases with increasing specimen temperature^(21,22).

* In our initial research^(7,10) on this subject three isochronal anneals were performed for $T_i = 8K$ as well as a large number for $T_i = 15K$. The spectra for these anneals were essentially identical to the one shown in Fig. 2(A). In addition, we have recently (Nielsen and Seidman, unpublished research) performed several more isochronal anneals for T_i equal to both 6 and 8K; again the spectra obtained were essentially identical to the one shown in Fig. 2(A). Hence, we conclude that there is no long-range migration of SIA's between 6 and 18K and that it is sufficient to irradiate at 18K to observe all of the long-range migration peak.

field was then applied to the tip and the surface damage was removed by pulse field evaporation. Each specimen was then warmed isochronally from 18 to 120K and the SIA flux was continuously photographed as a function of temperature.

Figure 2(A) shows a composite plot of the results for the seven irradiations performed. It is a plot of the fraction of the total defect recovery per 5K interval as a function of the specimen temperature. The term defect is employed since some of the contrast effects observed consisted of two or more bright spots. Each multiple bright spot contrast effect was counted as one defect. We have interpreted^(7,10) the large peak centered at 38K as being caused by the arrival of the long-range migration flux of SIA's at the specimens' surfaces (the Stage I SIA). The dominant peak at 38K was followed by further peaks at approximately 50, 65 and 80K. A small amount of additional recovery was detected to as high as 120K. The possible causes of the peaks observed above the 38K peak will be discussed in Part II, but the important point here is that 50.7% of the observed SIA recovery occurred below 50K in the isochronal annealing of the 18K irradiations. The histogram shown in Fig. 2(A) is taken as a nominal standard for the isochronal warming experiments in the presence of the BIF characteristic of 18K.

2. The second type of experiment was similar to the first type except that each 4-pass zone-refined specimen was irradiated at $T_1 = 50K$ in the absence of E and then cooled to 18K. At 18K the electric field was reapplied to the specimen and the first procedure was repeated.

If the long-range migration of an SIA in the absence of E occurred between 70 and 100K, as has been proposed by a number of investigators^(23,24,25,26), then the SIA should be immobile at both $T_1 = 18$ and 50K. The isochronal recovery spectra for the two irradiation temperatures would then be identical if this line of reasoning was correct. Figure 2(B) shows the isochronal annealing data for six irradiations at $T_1 = 50K$. A comparison of Figs. 2(A) and 2(B) shows that the

above argument is not correct. First, the dominant 38K peak observed in the 18K irradiations is absent in the case of the 50K irradiations. Second, only 9.2% of the total recovery took place below 50K. Third, the absolute total number of SIA's observed per run was noticeably less for the 50K irradiations.

4. DISCUSSION

4.1 Discussion of the experimental data

The dramatic decrease in the total amount of isochronal recovery observed between 18 and 50K [see Fig. (2)] for the two different irradiation temperatures employed demonstrates that SIA's are mobile during the 50K irradiation in the absence of E. During the 50K irradiation an appreciable fraction of the SIA's have been annihilated at both the free surface and the depleted zones. A certain fraction of the SIA's have also formed di-SIA's and these subsequently recover above 50K (see Part II for a discussion of this point). The data also indicates that the maximum possible downward temperature shift of the 38K peak was <10K as a result of the $p\Delta v_{11}^m$ effect.

The value of the enthalpy change (Δh) associated with a recovery peak in the isochronal annealing spectrum is taken to be a linear function of temperature. This follows from the equation for the temperature (T_m) at which the maximum occurs for in a recovery peak. This expression⁽¹⁰⁾ is

$$T_m \approx \frac{\Delta h}{k} [\ln \Gamma - 2 \ln \ln \Gamma]^{-1}, \quad (5)$$

where Γ is a dimensionless parameter that can be taken to be independent of temperature to first order. This linear relationship has been verified employing experimental data by Sosin⁽²⁷⁾ for several metals. Sosin's empirical relationship between the activation energy (Q) of a recovery peak and T_m is

$$Q(\text{eV}) = 2.5 \cdot 10^{-3} T_m(\text{K}). \quad (6)$$

In addition, for platinum we⁽²⁸⁾ have shown that a similar linear relationship holds for the existing recovery data in the temperature range 10 to 600K with a constant of proportionality equal to $2.6 \cdot 10^{-3} \text{ eV(K)}^{-1}$. The experimental activation energy obtained for the 38K recovery peak in tungsten was 0.085 eV ⁽¹⁰⁾ and we scaled[†] the activation energies to this value. Thus, a shift of $<10\text{K}$ in T_m corresponds to an energy change of $<0.02 \text{ eV}$ caused by the $p\Delta v_{11}^m$ term. Substitution of $E = 4.75 \text{ V(\AA)}^{-1}$ (or $E^2 / (8\pi) = 1 \cdot 10^{11} \text{ dyne cm}^{-2}$) into equation (2) yields the result that

$$\Delta v_{11}^m < 0.02 \Omega_a.$$

It is this extremely small value of Δv_{11}^m that minimizes the effect of the large electric-field induced pressure that is inherent to the FIM technique. This small value of Δv_{11}^m also indicates that the pulse field evaporation at 18K did not perturb the immobile SIA distribution^{*}. The present experiments demonstrate that the long range migration of the SIA in tungsten must occur below 50K. Electrical resistivity recovery experiments often detect two recovery peaks (sub-stages I_D and I_E)⁽²⁹⁾ due to long range SIA migration. The correlated recovery peak (I_D) is due to recombination of a freely migrating SIA with its own vacancy, while the I_E uncorrelated recovery substage is the recombination of an SIA with some other vacancy. The FIM experiments observe the flux of SIA's at the free

[†] The constant of proportionality was $2.24 \cdot 10^{-3} \text{ eV(K)}^{-1}$.

^{*} For $\Delta v_{11}^m < 0.02 \Omega_a$ and an evaporation field of $\sim 6 \text{ V\AA}^{-1}$ the ratio of the SIA diffusivity while pulse field evaporating at 18K to the diffusivity while imaging at 38K is $\sim 10^{-9}$. In addition, the evaporation field is applied to the tip for a total of only ~ 10 sec during the post-irradiation peel. Thus no significant SIA migration can occur under these experimental conditions.

surface during their long-range migration and the relative diffusion distances are such that the FIM recovery peak should fall between the correlated and uncorrelated electrical resistivity peak temperatures. Hence, any isochronal recovery peaks observed above 50K must belong to Stage II recovery. This conclusion is in contradiction to the earlier work^(23,24,25,26) which claimed that Stage I of tungsten terminated at 100K and that long range migration of the SIA occurred between 70 and 100K.

The first of the stage II peaks occurred at ~50K and we denote this peak substage II_A. The source of the small number of defects observed below the 50K irradiation temperature (see Fig. 2(B)) was probably the leading edge of this substage II_A peak. During an isochronal anneal a small percentage of the immobile substage II_A defects must become mobile before T_m. This slowly rising leading edge in the isochronal recovery spectrum of a thermally activated process has been considered in detail by Scanlan et al.⁽⁷⁾ (also see Part II for a further discussion of the isochronal annealing peak shape).

The small size of Δv_{li}^m allows for a direct comparison of FIM and electrical resistivity isochronal annealing experiments. In Part II⁽³⁰⁾ we make use of this fact in our study of the early Stage II (50 to 120K) recovery in tungsten.

4.2 The sign and magnitude of Δv_{li}^m

In the application of equation (1) it was assumed that Δv_{li}^m is a positive quantity; if Δv_{li}^m is a negative quantity then Δh_{li}^m would be increased as a result of the application of a negative hydrostatic pressure. The physical implication of this last statement is that the long-range migration peak observed at ~38K in the FIM might really be a lower temperature peak which was shifted upward in temperature because of a positive value of $p\Delta v_{li}^m$. This possibility can be eliminated on the basis of the production rate experiments of DiCarlo et al.⁽²⁴⁾ which

were performed at 20 and 30K. These researchers measured a constant defect production rate (as monitored by electrical resistivity) at both 20 and 30K which negates the possibility of long-range SIA motion in this temperature range. The major recovery peak below our 38K peak, for electron irradiated tungsten, is a peak at 30K (see DiCarlo et al., Kunz⁽⁸⁾ and Schultz and co-workers⁽³¹⁾). Since our FIM recovery peak represents long-range migration and it is above this 30K peak we conclude that all of the experiments* are consistent only if Δv_{1i}^m is a positive quantity.

The only theoretical estimates of Δv_{1i}^m for a b.c.c. metal are those of Johnson⁽³³⁾. He calculated that for α -iron the $\langle 110 \rangle$ split SIA has $\Delta v_{1i}^m = 0.1\Omega_a$ and the $\langle 111 \rangle$ crowdion has $\Delta v_{1i}^m = 0.0\Omega_a$. Johnson has performed similar calculations for the f.c.c. metals copper⁽³⁴⁾ and nickel⁽³⁵⁾. He obtained from his calculations, for the $\langle 100 \rangle$ split SIA, Δv_{1i}^m values of $-0.01\Omega_a$ for copper and $0.1\Omega_a$ for nickel. Thus, all the theoretical estimates for Δv_{1i}^m of an SIA in both b.c.c. and f.c.c. metals lie in the range $-0.01\Omega_a$ to $0.1\Omega_a$. On the experimental side the only other measurement of Δv_{1i}^m is from the FIM work of Pétroff and Seidman⁽³⁶⁾ on ion irradiated platinum; they obtained $\Delta v_{1i}^m = (0.02 \text{ to } 0.03)\Omega_a^{**}$. Hence, the existing theory and experiments all point towards a rather small value of Δv_{1i}^m for an SIA in both b.c.c. and f.c.c. metals. This is a physically reasonable conclusion if it is remembered that the volume change per SIA, when an SIA is formed, is very large (e.g., $1.45\Omega_a$ in the case of copper⁽³⁷⁾). Therefore, the nearest neighbor atoms of the SIA are displaced through significant distances from their equilibrium positions when the SIA itself is at an equilibrium position (e.g., see the drawings ...

* The present work in conjunction with our prior research^(7,10) and the DiCarlo et al.⁽²⁶⁾ production rate experiments also exclude Roberts'⁽³²⁾ conclusion that long-range migration of an SIA occurs at 22K in tungsten.

** Wei and Seidman⁽³⁹⁾ have shown that this value is an upper limit as a result of a study of Stage I and Stage II recovery of an ion irradiated alloy of platinum-0.62at.% gold.

of the displacement fields of the [110] split SIA and the [111] split crowdion in Figs. 3 and 4 of Seidman and Lie⁽³⁸⁾). This situation implies that when the SIA goes from an equilibrium position to a saddle-point configuration that the nearest neighbor atoms do not have to alter their positions by very much, since they are already displaced by a significant distance. The result is that volume change of the lattice per SIA when the SIA goes from the equilibrium to the saddlepoint position is small; i.e., Δv_{li}^m is small.

5. SUMMARY

It has been demonstrated experimentally that the value of the volume change of migration (Δv_{li}^m) of the Stage I self-interstitial atom (SIA) in tungsten is less than 0.02 atomic volume. This result implies that the effect of the electric field ($\sim 4.75 \text{ VA}^{-1}$), required for field ionizing helium gas atoms in the field ion microscope, is to reduce the internal change of migration (Δu_{li}^m) of the SIA by less than 0.02 eV. Hence, the enthalpy change of migration (Δh_{li}^m) of 0.085 eV measured by Scanlan et al.^(7,10) for the long-range migration peak at $\sim 38\text{K}$ in tungsten differs from Δu_{li}^m by an upper limit of 0.02 eV. Our results also show that the pressure (p) required to produce a significant $p\Delta v_{li}^m$ effect in more practical situations would be enormous since the field of $\sim 4.75 \text{ VA}^{-1}$ corresponds to $\sim 1 \cdot 10^{11} \text{ dyne cm}^{-2}$ (100 kbar).

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FIGURE CAPTIONS

Fig. 1. (A): An FIM micrograph of a tungsten specimen after irradiation at 18K with 30 keV W^+ ions to a dose of $5 \cdot 10^{12}$ ion cm^{-2} . Extensive surface sputtering is evident on the beam side of the crystal.

(B): The same specimen after $5A$ of metal had been removed by pulse field evaporation. The data chamber can be seen on the right hand side of both images.

Fig. 2. (A): A composite isochronal warming spectrum of seven spectra for four-pass zone-refined tungsten irradiated at 18K with 30 keV W^+ ions and warmed to 120K at a rate of $\sim 2.5K \text{ min}^{-1}$.

(B): A composite isochronal warming spectrum of six spectra for four-pass zone-refined tungsten irradiated at 50K with 30 keV W^+ ions and cooled to 18K in the absence of the electric field. The specimens were then warmed from 18 to 120K at a rate of $\sim 2.5K \text{ min}^{-1}$.

BEAM →
DIRECTION

(A)

(B)

Figure 1

Figure 2