# IFIELD ION MICROSCOPE STUDIES OF 400 eV ARGON ION BOMBARDMENT DAMAGE IN TUNGSTEN

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Abstract: Field ion microscope has been used to investigate argon ion bombardment of tungsten at 63 K. The bombardment was carried out at a background pressure in the 10<sup>-11</sup> torr range with 400 eV Ar<sup>+</sup> ions. Clusters of vacancies and interstitials have been observed. After field evaporation of a few atomic layers damage could still be observed on the newly exposed surface indicating damage produced inside the specimen. Evidence for an assisted focusing event in the direction of the [100] — zone is presented.

# 1. Introduction

) The field ion microscope <sup>1</sup>) has been extensively used in recent years for the observation of irradiation-induced defects in metals<sup>2-4</sup>). The atomic resolution which is possible with the microscope lends itself to visual examination of atomic displacement phenomena, especially at low bombarding energies where virtually all of the target damage lies within a few atomic layers of the surface.

In an earlier report<sup>5</sup>) the apparatus and experimental techniques used to investigate irradiation-induced damage in tungsten bombarded at 63 K were discussed. The results on specimens irradiated with  $Ar^+$  ions in the 150-450 eV energy range were presented. Diffusion of self-interstitials upon annealing the irradiated specimens from 63 K to 78 K was observed.

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## 2. Results and discussion

Ion bombardment of tungsten specimens was carried out at 63 K. This temperature freezes some of the irradiation-induced interstitials inside the specimen and so they can be revealed either by annealing the specimen or by exposing the interior of the specimen by a field evaporation process. Both techniques were employed in our experiments.



Fig. 1. Tungsten surface after irradiation of 400 eV energy, after field evaporation of several atomic layers.



Fig. 2. The same surface after warming up the specimen to 78 K.

Fig. 1 shows a specimen surface after irradiation by 400 eV energy  $Ar^+$  ions after subsequent field evaporation of a few atomic layers. On annealing this tip to 78 K, (Fig. 2), six interstitials forming a cluster, can be observed on the (001) plane (encircled area in Fig. 2). It is thought that a cluster of interstitials represents a damaged area which had been created by irradiation in the interior of the target. After a few atomic layers were removed by field evaporation, these interstitials, being one or two atomic layers below the surface, diffused to the surface upon annealing. In the annealing process one atom diffused on the (011) plane and anther one on the (101) plane. Two vacancies were created in the annealing process, and are indicated by arrows.



Fig. 3. The surface of a tungsten specimen before bombardment at 63 K.

Another tungsten specimen before bombardment with 400 eV energy  $Ar^+$ ions at 63 K is shown in Fig. 3. The same surface after bombardment is shown in Fig. 4. During the bombardment time of 90 seconds a total ion current of  $1.4 \cdot 10^{-9}$ A was recorded. Due to the target geometry it was impossible accurately specify



Fig. 4. The same surface after bombardment with 400 eV Ar $\pm$  ions at 63 K.

the ion dose per cm<sup>2</sup>. While the imaging voltage was on, specimen contamination was effectively eliminated because the high field was able to ionize residual gas before it reaches the tip. However, during helium gas pumpdown, bombardment and helium gas readmission, the imaging voltage was off for a typical time of 4 minutes. Any gas contamination of the tungsten target area at typical background pressure in the  $10^{-11}$  torr range was thought to be minimal.

The direction of the incident radiation in Fig. 4 is indicated by broad arrows. and was in the direction of the [100] – zone. The encircled areas indicate some of the damage introduced by the bombardment. Four thin arrows indicate an interesting event that took place on the tenth atomic layer below the top (011) plane of the specimen. On the side - the tenth net plane ring towards the ion beam there are two surface vacancies, and on the opposite side, in the direction of the [100] — zone, there are two atoms missing. These vacancies, it is believed, are created by an assisted focusing event in the [100] direction. The number of atoms involved in the focusing sequence is about 30. Assuming the hard sphere condition, Nelson<sup>6)</sup> evaluates a focusing energy for the (100) focusing sequence in tungsten. According to his calculations a sequence starting at E = 200 eV will make approximately 20 collisions before attenuation. At this energy successive collisions occurring before the midpoint between the two interacting atoms consequently only transfer momentum. Because of the potential maximum at the focusing ring, sequences which propagate with successive collisions occurring beyond the midpoint will be of a replacement nature. The focusing event found on the previous micrograph is initiated with 400 eV energy, which corresponds to a transferred energy of 235 eV. This energy would therefore be sufficient to promote a focusing event assuming a head-on collision.

# 3. Conclusion

Bombardment of tungsten field ion m<sup>i</sup>croscope t<sup>i</sup>p spec<sup>i</sup>mens with 400 eV energy  $Ar^+$  ions produced visual damage as observed in the field ion microscope in the form of interstitials, vacancies and their clusters. Field evaporation of irradiated specimens revealed damage in the interior of the specimen. It is suggested that a focusing sequence was responsible for causing vacancies found on the opposite sides of the (011) plane (Fig. 4) after the tip had been iradiated with 400 eV energy  $Ar^+$  ions in the direction of the [100] — zone.

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## ISPITIVANJE ZRAČENOG VOLFRAMA SA ARGON IONIMA 400 eV ENERGIJE U ELEKTROSTATSKOM MIKROSKOPU

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### Sadržaj

Elektrostatki mikroskop je upotrebljen za ispitivanje volframa izloženog zračenju sa argon ionima kod temperature od 63 K. Bombardiranje je vršeno u vakuumu od  $10^{-11}$  mm Hg pozitivnim argon ionima 400 eV energije. Skupine praznina i meduprostornih atoma su nadene na površini zračenih uzoraka.

Nakon odstranjivanja nekoliko površinskih slojeva atoma metodom isparivanja električnim poljem, defekti su prisutni na novo izloženoj površini, što ukazuje da je oštećenje prouzrokovano i u unutrašnjosti materijala.

Iznesena je također mogućnost potpomognutog fokusiranja u smjeru [100] - zone.