



Department of Energy  
Chicago Operations and Regional Office  
9800 South Cass Avenue  
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August 21, 1980

**MASTER**

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CONTRACT DE-AC02-76ER03158 (FORMERLY CONTRACT NO. EY-76-S-02-3158)  
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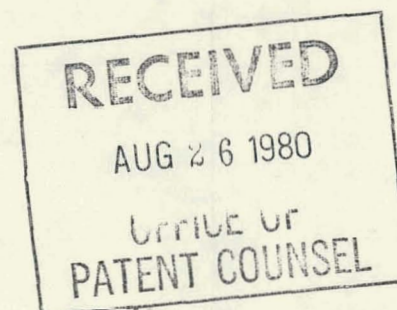
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CONF-800744--6

## DIRECT OBSERVATION OF THE PRIMARY STATE OF RADIATION

## DAMAGE OF ION-IRRADIATED TUNGSTEN AND PLATINUM

by

D.N. Seidman, M.I. Current, D. Pramanik and C.-Y. Wei

July 1980

Report #4278

Issued by

The Materials Science Center

Prepared for

THE U.S. DEPARTMENT OF ENERGY under Contract No. DE-AC02-76ER03158.

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Invited paper presented at the Second International Ion Beam Modification of Materials Conference, 1980, July 14-18, State University of New York at Albany, New York. To appear in Nuclear Instruments and Methods (1981).

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DIRECT OBSERVATIONS OF THE PRIMARY STATE OF RADIATION

DAMAGE OF ION-IRRADIATED TUNGSTEN AND PLANTINUM<sup>†</sup>

by

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ABSTRACT

A brief summary was presented of all the Cornell work on the primary state of radiation damage in ion-irradiated tungsten and platinum. The primary research tool for all this research was the field-ion microscope (FIM); the FIM was ideally suited for this research because of its excellent atomic resolution and the ability to examine the interior of the specimens, as a result of the field-evaporation effect. This paper summarized, in outline form, the following items: (1) the principal experimental quantities determined from the analyses performed on all the individual depleted zones (DZs) observed; (2) the main experimental programs; (3) a number of the more important results and conclusions concerning the vacancy structure of DZs; and (4) the three-dimensional spatial distribution of self-interstitial atoms around DZs in tungsten which had been irradiated and examined in situ at 10 K.

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This research was supported by the U.S. Department of Energy under Contract No. DE-AS02-76ER03158. Additional support was received from the National Science Foundation through the use of the technical facilities of the Materials Science Center at Cornell University.

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The field-ion microscope (FIM) with its excellent atomic resolution and the ability to examine the interior of specimens--as a result of the field-evaporation effect [1,2]--is ideally suited for the study of the properties of point defects [3-5]. As early as 1971 we examined the point-defect structure of two depleted zones (DZs) created in tungsten, at <15K, by 20 keV W<sup>+</sup> ions and measured the range of 25 self-interstitial atoms (SIAs) which had been produced as a result of a series of replacement collision sequences (RCSs) [6]. This work continued with the detailed observation, in 1973, of the vacancy structure of a DZ created in tungsten at 473 K by a single 20 keV W<sup>+</sup> ion. More recently we determined the vacancy structure of a (220) platelet which had been created by a single 30 keV W<sup>+</sup> ion in a platinum-4 at.% gold alloy [8]. These early studies served as background experience for our more recent and rather extensive systematic studies of the primary state of damage of ion-irradiated tungsten [9-15] and platinum [15].

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"In general, it has been found possible to classify the vacancy portion of the primary state of radiation damage into three morphological states: (1) depleted zones; (2) compound vacancy clusters or voids; and (3) dislocation loops. The SIAs were observed within and around isolated DZs [6,13], that had been produced in tungsten specimens maintained at  $<15$  K. The values of the ion fluences employed were small enough such that each DZ was created by a single projectile ion. In the case of tungsten, the temperature of each specimen both during and after an irradiation was well below the onset of the long-range migration of SIAs [16]. Whereas, for platinum the temperature of irradiation was above the SIA long-range migration stage, that is, Stage I. All specimens were irradiated in situ under ultra-high vacuum conditions, to a dose of less than  $10^{13}$  ions  $\text{cm}^{-2}$ , and then examined in situ employing the pulse field evaporation technique [3-5].

The principal experimental quantities determined from the analyses performed on all the individual DZs observed were as follows:

- (1) The direct determination of the absolute number of vacancies contained within each DZ ( $v$ ) that was created by a single projectile ion of mass ( $M_1$ ) and initial energy ( $E_1$ );
- (2) Visual representations of the positions of the vacancies included within every DZ--these were made employing the OR TEP program [17];
- (3) The measurement of the average diameter of an individual DZ ( $\langle \lambda \rangle$ ) and the orientation  $[hkl]$  of the DZ with respect to the ion beam and the crystal lattice;
- (4) A calculation of the average vacancy concentration within each DZ ( $\langle c_v \rangle$ ) based on  $v$  and the actual volume filled by the vacancies;
- (5) A calculation of two different types of radial distribution functions [9,10,12,14,15] for the vacancies comprising each DZ;
- (6) The determination of the fraction of first-nearest neighbor vacancies--within each DZ--in clusters of size  $n$ , where  $n$  ranged from one to five to approximately four hundred;

- (7) The depth (L) from the irradiated surface--measured along a direction parallel to the incident ion beam--at which the DZ was detected and the direction of elongation ([hkl]) of the DZ; and
- (8) The measurement of the number ( $v_{ns}$ ) and concentration ( $\langle c_v \rangle_{ns}$ ) of vacancies which were created in the near-surface region ( $< 5 \text{ \AA}$  thick) of ion-irradiated tungsten specimens [11,12].

For each experimental program listed below the physical quantities outlined in the preceding paragraph were determined. The main experimental programs were as follows:

- (1) The determination of the effect of  $M_1$  on the point-defect structure of DZs, in tungsten, at constant  $E_1$ --the projectiles  $W^+$ ,  $Mo^+$ ,  $Kr^+$ ,  $Cu^+$ ,  $Cr^+$  and  $Ar^+$  were employed for  $E_1 = 30 \text{ keV}$ ;
- (2) The determination of the effect of  $E_1$ , at constant  $M_1$ , on the point-defect structure of DZs in tungsten--15, 30, 45, 60 and 70 keV Kr ions were used as well as 20, 30, 45, and 60 keV W ions;
- (3) The effect of monomer versus dimer irradiations on the point-defect structure of DZs in tungsten--the projectiles employed were 20 keV  $Ag^+$  and  $W^+$ , and 40 keV  $Ag_2^+$  and  $W_2^+$  ions; and
- (4) In the case of tungsten the three-dimensional spatial distributions of SIAs around DZs was determined;
- (5) The point-defect structure of DZs in platinum which had been irradiated with 20 keV  $Kr^+$  ions;
- (6) The sign (vacancy or SIA) and habit plane of the dislocation loops detected in both the ion-irradiated platinum and tungsten specimens.

Within the context of this precis of our work it is impossible to give all the results and conclusions reached. Instead we have simply listed a number of the more important ones; the reader is referred to the references for further details. For the tungsten specimens<sup>†</sup> the following was observed and concluded:

- (1) To first order the value of  $v$  was independent of  $M_1$  and equal to approximately 172 vacancies per DZ for  $E_1 = 30 \text{ keV}$ ;

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<sup>†</sup> The results that were characteristic of platinum are listed under numbers (11) and (12).



- (2) This value of  $\nu$  was in good agreement with the Kinchin-Pease [18] expression as modified by Robinson and Torrens [19];
- (3) The value of  $\langle \lambda \rangle$  increased as  $M_1$  was decreased at constant  $E_1$  (30 keV);
- (4) The value of  $\langle c_v \rangle$  decreased from  $\approx 16$  to 2 at.% as  $M_1$  was decreased from 183.85 amu (W) to 39.948 amu (Ar) at an  $E_1$  of 30 keV;
- (5) For both Kr and W the value of  $\nu$  increased linearly as  $E_1$  was increased from 15 to 70 keV;
- (6) For both Kr and W ions, with energies between 15 and 70 keV, the values of  $\langle \lambda \rangle$  did not follow the trend of increasing  $\langle \lambda \rangle$  predicted by the linear-cascade model;
- (7) The spatial arrangement of the vacancies within the DZs was a strong function of  $M_1$  at constant  $E_1$  (30 keV)--in general, the number of subcascades within each DZ increased as  $M_1$  was decreased;
- (8) The fraction of monovacancies contained within each DZ increased as  $M_1$  was decreased at constant  $E_1$  (30 keV);
- (9) The value of  $\langle \nu \rangle$  per projectile ion for DZs produced by dimers was  $\approx 1.5$  times greater than the value of  $\langle \nu \rangle$  for the DZs created by monomers--at a constant energy per projectile ion;
- (10) Statement (9) indicated the existence of strong nonlinear effects in the production of dense collision cascades;
- (11) In the case of platinum, for  $E_1 = 20$  keV, examples of all the three morphological states were found--this was not the case for tungsten irradiated with 20 and 30 keV ions;
- (12) For  $E_1 = 20$  keV Kr<sup>+</sup> ions, in the case of platinum, the DZs collapsed more easily to dislocation loops than in the case of tungsten--even though the average size and point-defect structure of the DZs in the two metals were similar; and
- (13) The fraction of DZs that collapsed into dislocation loops in tungsten, irradiated with W ions, increased as  $E_1$  was increased from 20 to 60 keV.

Finally, the three-dimensional spatial distribution of SIAs around DZs, in ion-irradiated tungsten, was determined [13]. Tungsten FIM specimens were irradiated in situ with 30 keV  $\text{Cr}^+$  or 18 keV  $\text{Au}^+$  ions along the  $[\bar{7}41]$  direction, at 10 K, and examined at this temperature by the pulse field evaporation technique. At 10 K the SIAs, in tungsten, were completely immobile. The distances were measured, along the close-packed crystallographic directions--that is, the  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$ -type direction--between each SIA and the DZs. Distance measurements were also made between each SIA and the irradiated surface of the specimen. The set of distances employed for analysis corresponded to the minimum measured distances; thus, the histograms of the distances presented represented a lower bound (denoted by  $R_{\min}$ ) to the actual propagation distances. For the 30 keV  $\text{Cr}^+$  ion-irradiated specimen a total of 97 SIAs were detected and  $\langle R_{\min} \rangle$  was  $175 \pm 110 \text{ \AA}$ ; the  $\pm$  values for  $\langle R_{\min} \rangle$  represent one standard deviation. In the case of the 18 keV  $\text{Au}^+$  irradiated specimen the quantity  $\langle R_{\min} \rangle$  was  $175 \pm 130 \text{ \AA}$ ; 33 SIAs were detected. A composite distribution of  $R_{\min}$  values was obtained by combining our earlier measurements [6] with the present results to obtain  $\langle R_{\min} \rangle \approx 160 \pm 120 \text{ \AA}$ . We suggested that this value may have represented an overestimate of the mean range of replacement collision sequences (RCSs) in tungsten. Nevertheless, the results constituted very direct evidence for the existence of RCSs in tungsten. These results are consistent with our observation that the values of  $\langle v \rangle$  were in agreement with the modified Kinchin-Pease expression [18,19]. That is, the RCSs provided an efficient mechanism for the separation of SIAs from the DZs.

All of the experimental results given above for DZs have been compared, in detail, with both the analytical and Monte Carlo computer simulation models of deposited energy; the reader is referred, once again, to the publications for details.

#### ACKNOWLEDGEMENT

We wish to thank Mr. Robert Whitmarsh for his enthusiastic technical assistance in the experimental program.

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