

FIELD ION MICROSCOPY OF DEFECTS IN TUNGSTEN SINGLE CRYSTALS

DUE TO  $\alpha$ -PARTICLE BOMBARDMENT

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Tungsten crystal imperfections due to bombardment with  $\alpha$ -particles of various energies (from 12.7 MeV to 23.4 MeV) are investigated by means of a helium ion projector (field ion microscope) operating at 78°K. The integrated irradiation doses varied between  $1.7 \times 10^{15}$  and  $1.6 \times 10^{16}$  particles/cm<sup>2</sup>. Four main types of imperfections were observed in the irradiated samples: single vacancies, atoms displaced to interstitial positions, vacancy clusters of  $\sim 10 \text{ \AA}$  diameter and small complexes of displaced atoms on the surface.

THE application of the method of field ion microscopy to the study of defects in the crystal lattice of tungsten bombarded by  $\alpha$  particles, was reported in [1-4]. Specimen tips of tungsten, prepared beforehand and mounted inside the ion microscope, were exposed to irradiation by  $\alpha$  particles. In all the cases, polonium sources of  $\alpha$  particles with energies from 5.0 to 5.4 MeV and intensities of  $0.5 \times 10^{-3}$  Curie were used. The sources were mounted inside the microscope in a copper cylinder at a distance of about 1 cm from the tungsten tips. On the average, one  $\alpha$  particle hit the sample every three hours, each hit was visible, this damage being observed only on the side of emergence of the  $\alpha$  particle. About 15 to 30 atoms were displaced within a region of about 50  $\text{\AA}$  in diameter. In half of the observed cases, smaller defects were visible in the region of emergence of the  $\alpha$  particles. For example, tight groups of one to three atoms in interstitial spaces, at distances of up to half the radius of the tip from the place of primary damage.

The purpose of the present work was the study, by means of ion microscopy, of defects produced in tungsten by  $\alpha$ -particle bombardment. Fine tungsten wires were irradiated, rather than previously prepared tips. As in [5], it was established that the character of the distortions observed on the ion image is greatly influenced by the method of irradiation. It was found that this method of irradiation gives more correct information about distortions appearing in the irradiated materials than the method of irradiating previously prepared tips. In addition, irradiation of wires allowed the use of larger integrated doses, thereby substantially increasing the defect concentration, and accordingly the precision of its determination.

Industrial tungsten wires were annealed for one and a half hours in a vacuum of  $10^{-5}$  Torr at 2000°C, and irradiated in the ion channel of the cyclotron of the Institute of Theoretical and Experimental Physics. To do this, the wires were placed on a special movable target, so that a set of three samples could be irradiated in the same run. During irradiation the temperature of the samples did not rise above 60°C. The energies of the  $\alpha$  particles used were  $E = 12.7 \text{ MeV}, 15.7 \text{ MeV}, 18.4 \text{ MeV}$  and  $23.4 \text{ MeV}$  and the integrated irradiation doses  $J_{\Sigma}$  were between  $1.7 \times 10^{15}$  and  $1.6 \times 10^{16}$  particles/cm<sup>2</sup>.

Tips were made out of the irradiated wires by electrochemical etching, and were studied in the helium ion projector [6] at 78°K. The subsequent field evaporation of many surface atomic layers of the tip [7] allowed a volume of the specimen of the order of  $10^{-15}$  to  $10^{-16} \text{ cm}^3$  to be subjected to analysis and thus permitted the volume concentration of the observed defects to be determined.

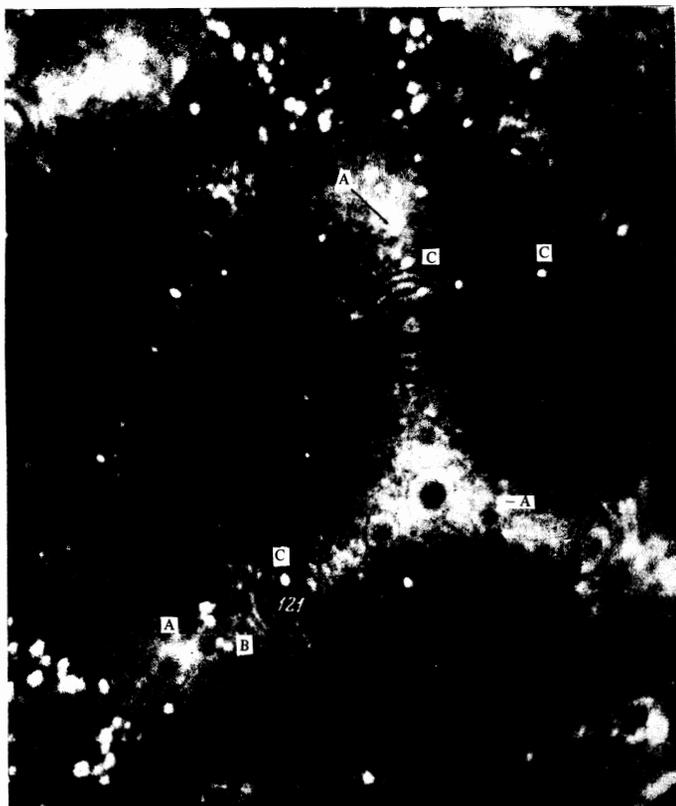
Four types of defects were observed in the crystal lattice of irradiated tungsten: single vacancies, single atoms displaced into interstitial sites, clusters of vacancies with diameter of the order of 10  $\text{\AA}$ , and small complexes of displaced atoms at the surface. While single vacancies and single displaced atoms were observed on the ion image of the  $\alpha$ -bombarded sample at all the energies used, the complexes of displaced atoms at the surface were observed only in the case of  $\alpha$  bombardment at two energies, 18.4 MeV and 23.4 MeV. The clusters of vacancies of 10  $\text{\AA}$  diameter formed by irradiation of the samples, were observed very rarely and only in the samples made of the wires irradiated with 23.4 MeV  $\alpha$  particles.

The concentration  $c$  of the single displaced atoms and the concentration  $c_c$  of the small complexes of displaced atoms were determined from the ion images of the irradiated samples. The data are shown in the table, which lists also the theoretical estimates of the range  $l$  of  $\alpha$  particles with initial energy  $E$  in the tungsten crystal, the number  $n'$  of single displaced atoms corresponding to given integrated doses of irradiation  $J_{\Sigma}$ . The number of displaced atoms appearing on irradiation,  $n'$ , was calculated in accordance with [8, 9] assuming that the threshold displacement energy  $E_d$

E, MeV	$10^{-15} J_{\Sigma}$ , particles/cm <sup>2</sup>	Calculated			Experimental	
		$10^4 l$ , cm	$n'$	$10^{-16} c$ , cm <sup>-3</sup>	$10^{-15} c$ , cm <sup>-3</sup>	$10^{-16} c_c$ , cm <sup>-3</sup>
12.7	1.7	2.85	87	5.2	2	—
15.7	1.85	3.85	97	4.66	>1	—
18.4	13.5	4.85	106	29.5	9	>1
23.4	16.0	6.85	115	26.9	28**	30

\*The calculation was carried out on the assumption of uniform production of displaced atoms as the  $\alpha$ -particles slow down.

\*\*The value shown does not take into account displaced atoms forming complexes; allowance for these atoms leads to the concentration for single displaced atoms equal to  $4 \times 10^{20}$  atoms/cm<sup>3</sup>.



Helium ion image of a tungsten tip made out of wire irradiated with 23.4-MeV  $\alpha$  particles. The arrows point to: A – complexes of displaced atoms at the surface; B – cluster of vacancies of diameter of  $\sim 10$  Å near the (121) face; C – single displaced atom.

for tungsten is 50 eV.<sup>[10]</sup> Good agreement between experimental results and theory was obtained for  $\alpha$  particles with energy 23.4 MeV.

The plate shows a typical ion image of a tip made of tungsten wire irradiated with  $\alpha$  particles of energy 23.4 MeV. Complexes of interstitial atoms on the surface are marked with arrows A, a small cluster of vacancies is marked B, and the single displaced atoms are marked C. The ion image was photographed at a voltage slightly higher than that corresponding to the best representation of the tip surface (i.e., corresponding to the autoionization field of the helium atoms). The presence on the image of the single displaced atoms and the complexes of atoms shows, obviously, strong bonding with the surface, which is higher than the bonding energy to the atoms at the lattice sites. This can be attributed to local strengthening of the electric field in the neighborhood of the displaced atoms, caused by the local decrease in the radius of curvature of the surface, and resulting in an increased contribution to the polarization.

As shown above, the clusters of vacancies of type B were observed only on the images of the samples irradiated with 23.4 MeV  $\alpha$  particles, and they were observed so seldom that their concentration could not be determined accurately; it was only estimated at less than  $5 \times 10^{17}$  clusters/cm<sup>3</sup>.

To determine from the ion images the concentration of single displaced atoms in the irradiated samples, three fundamental factors leading to errors in the calculations were taken into consideration.

1. The presence in the sample of impurity atoms, positioned not at lattice sites and therefore producing points of enhanced intensity (brightness).

2. The process of field evaporation of the atomic layers of the tip, leading to the displacement of some surface atoms of the matrix into metastable positions, which give a higher image brightness.<sup>[11]</sup>

3. The mobility of the displaced atoms of the surface atomic layers at 78°K<sup>[10, 12]</sup> under the influence of great mechanical stresses produced by the electric field inside the microscope.

This mobility of the single displaced atoms at 78°K caused them to appear on the ion image not at the positions of their original formation upon irradiation, thus making a determination of their crystallographic distribution impossible.

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