SOVIET PHYSICS USPEKHI VOLUME 13, NUMBER 3 NOVEMBER-DECEMBER 1970

548.0:53

FIELD-ION MICROSCOPY OF RADIATION DEFECTS IN SINGLE CRYSTALS

A. L. SUVOROV

Institute of Theoretical and Experimental Physics, Moscow

Usp. Fiz. Nauk 101, 21-52 (May, 1970)

1. INTRODUCTION

THE successful development of atomic power and also the possibility of verifying and explaining important aspects of solid state theory stimulate ever-increasing interest in the problem of radiation damage in solids. An investigation of the laws governing the creation of radiation defects, their distributions, and the nature of their influence on the mechanical and physical properties of the materials which are utilized in the construction of nuclear facilities is of great practical value.

The basic methods which are available for an investigation of radiation damage in solids can only give statistical data, obtained by means of the measurement of macroscopic physical properties which are sensitive to the existence of defects. Such methods include electron- and neutron-diffraction studies, radiography by X rays, methods of measuring the electrical resistivity, internal friction, heat capacity, the modulus of elasticity, the measurement of volume changes, and so forth. However, the indicated indirect methods of investigation are extremely limited. Data obtained with their aid very rarely (new methods in radiography by X rays constitute the only exception) make it possible to reach a conclusion about the relation between physical properties and a definite atomic configuration.

The most promising method is the direct observation of the defects which appear upon irradiation, the establishment of their crystallographic distribution and atomic configuration. The method of electron microscopy of thin foils, which has been developed in recent years, makes it possible to obtain high resolution down to 10 Å. With its aid an investigation of many defects becomes possible, including the observation of small complexes of point defects. An investigation of two thin films, one superimposed on the other, by using the method of the transmission electron microscope makes it possible to obtain, under certain conditions, moiré pictures reflecting the structure of the stresses around the defects.[1] However, even such sensitive methods do not make it possible to observe individual point defects in crystals or configurations of their small complexes. And only the ion projector (field-ion microscope) proposed by Müller[2,3] first placed in the hands of the experimentalists a method which made it possible to directly observe the crystal lattice of metallic samples at the atomic level.

Possessing a resolution of the order of 2 to 3 Å, the ion projector makes it possible to observe individual point defects (vacancies, interstitial atoms, the atoms of impurity elements), linear defects (dislocations), and volume imperfections of different types. In addition, the use of an ion projector turned out to be promising for the investigation of chemical reactions stimulated by a field, processes involving the surface migration of atoms, field ionization and field evaporation, and so forth. With the aid of an ion projector the structures of grain boundaries, the atomic configurations of volume defects, etc. can be investigated.

The atomic resolution of an ion projector makes the study of point defects and an explanation of their crystallographic distribution, and also an investigation of the atomic configurations of imperfections which encompass large many-atom regions the most promising area of application. Among such investigations, at the present time most attention is given to the investigation of radiation damage.

The goal of the present article is a discussion of the existing methods of field-ion microscope investigation of radiation damage and a review of the fundamental articles in this area which have been published up to May 1969. The methods of field-ion microscopy and the theory of the formation of the field-ion image of metallic samples is quite adequately discussed in the review monograph by Müller[3] and will not be touched upon here.

2. CRYSTAL DEFECTS AND THEIR OBSERVATION WITH THE AID OF AN ION PROJECTOR

2.1. Point Defects and Their Complexes

A vacancy pair—a displaced atom (and also an atom of an impurity element)[4,5]—are the simplest defects in a crystalline lattice. The bombardment of metals by different energetic particles in turn leads to the formation in the crystal lattice of precisely these defects and, in a number of cases, of their complexes.

The ion projector of Müller[3] first made it possible to directly observe point defects. Metallic needle-shaped tips with extremely small radii of curvature (100 to 1000 Å), which are placed at distances of several centimeters from a fluorescent screen in an evacuated bulb (see Fig. 1) are used as the samples in an ion projector. After thorough vacuum evacuation, some kind of gas (usually helium or neon at a pressure of the order of 10^{-3} to 10^{-4} Torr) is admitted into the projector system; this gas serves to produce an image of the atomic details of the hemispherical surface of the sample. During the experiment the sample is usually cooled down to the temperature of liquid nitrogen (78° K), liquid hydrogen (21° K), or liquid helium (4.2° K). A high positive potential (up to 30 kV) is applied to the sample, and the fluorescent screen is grounded. Under the influence of the strong electric field the electron gas is somewhat pushed back into the depths of the metal, and the atoms on the surface of the specimen are partially "uncovered."[5] When some atom of the imaging gas approaches an ion on the surface of the specimen, it may

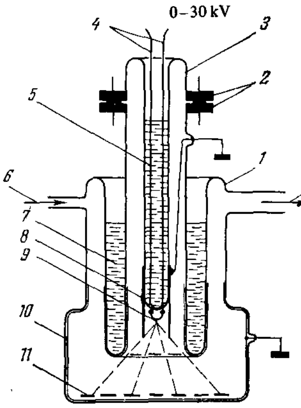

FIG. 1. Schematic diagram of the tube of a demountable ion projector: 1–flask, 2–flanged coupling, 3–cooled stem, 4–metallic leads, 5, 7–cooled liquid, 6–admission of imaging gas, 8–metallic screen, 9–specimen, 10–transparent conducting jacketing, 11–fluorescent screen, 12–to vacuum pumping system.

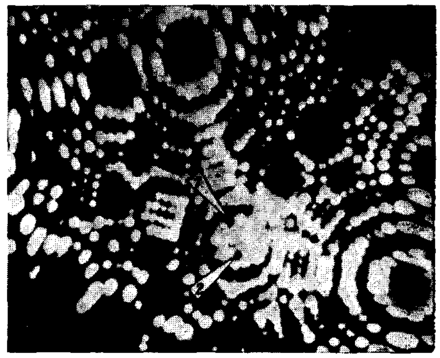

FIG. 2. Helium ion image of platinum showing a single vacancy (arrow 2) and the absence of an atom at the turning point on the edge of a plane (arrow 1). [9]

be ionized in the strong electric field and the electron released by tunneling returns to the metal (the phenomenon of field ionization, discovered by Oppenheimer[6]). The ion which is created is accelerated in a radial direction by the high potential and produces a bright spot at the position of its incidence on the screen. Thus a number of light spots appear on the screen, each of them being produced by ions of the imaging gas which are being created at one and the same ionization center on the surface of the tip. Such ionization centers correspond to the atoms which protrude the most above the surface, especially those which lie on crystal facets with large indices and on the steps which are formed by the edges of the crystal planes.

At a sufficiently high voltage evaporation of the surface atoms from the specimen takes place under the action of the electric field[7, 8]. This process is especially intensive at the edges of the steps formed by the planes of the crystal, since at these points the local field is higher than average. By means of evaporation of the arrays of atoms distributed along the steps of the lattice where the local field is maximal, one can remove the atomic layers in a controlled fashion. Utilization of the process of evaporation of surface atomic layers from the specimen by an electric field makes it possible to investigate the distribution of defects in the volume of the tip, and for defects subtending a larger many-atom region one can ascertain their atomic configurations.

At the present time the identification of single vacancies[9] and of their small complexes in the ionic images of needle-shaped metallic samples is a completely solved and unambiguous problem, although there are certain difficulties in this area which are basically associated with the fact that the field-ion microscope investigates defects on the surface of a crystal, where they are found in peculiar positions.[10]

As is well known, the larger part of the atoms which are visible in the ionic images constitutes the edge of closely-packed net planes; the other part of the atoms constitutes completely built-up resolvable facets. The absence of an atom at the edge of a net plane (for example, the situation indicated by arrow 1 in Fig. 2) may be due to two factors: at this place either there actually is a vacant lattice site or else this is simply a turning point. In such a case it is not possible to find out with the definiteness with which we have to deal. Therefore, in order to eliminate errors the count of the vacancies should be carried out only on completely built-up facets.*) The best crystal faces for searches for vacancies are the most protruding surfaces with a low work function. And although planes with higher indices can be resolved into ion images better, searches for vacancies on them are more difficult because of the fact that such faces contain considerably fewer atoms.

A single vacancy appears on an ion image as a dark spot inside a completely built-up surface (indicated by arrow 2 in Fig. 2), indicating the absence of a single atom in the given lattice plane.

In connection with the observation of annealed and tempered samples in an ion projector, mostly single vacancies are observed in their images, but in rare cases divacancies are observed. In a number of cases the bombardment of samples by heavy charged and neutral particles caused the formation in the crystal lattice of tri-vacancies[12] and small groups of vacancies (vacancy clusters), having linear dimensions of the order of 10 to 15 Å (see, for example, [13]).

It is most convenient to determine the concentration of single vacancies by successively field evaporating them one after the other, photographing many surface atomic layers, and counting the vacancies on any single face, i.e., by analyzing some arbitrarily chosen prismatic volume, containing a known number of atoms, in the lattice of the tip. If the identification and counting of single vacancies and of their small complexes on ion images of the samples does not present any particular difficulties, then the identification of interstitial atoms and especially the development of the reasons for their appearance present considerable difficulty up to the present time, and are represented in different ways by different authors.[13-18]

We recall that the image in an ion projector is created as a result of the ionization of atoms and molecules of the imaging gas above protruding surface atoms of the hemispherical specimen—the tip being at a high positive potential, and by the subsequent acceleration of the created ions along a radial direction to the fluorescent screen. If any atom or group of atoms protrude above the surface of the tip to a greater extent than the other surface atoms, then above it (or above them) there is a local increase in the electric field in-

*One should, however, bear in mind that the absence of a bright spot on the ion image may sometimes be associated with the "invisibility" of certain impurity atoms. [11]

tensity due to the local increase in the curvature of the surface. As a result, at this place the probability of ionization of the atoms or molecules of the imaging gas increases, and this leads to the result that a relatively larger number of ions participate in the image of the indicated atom (or group of atoms). The brightness I_i of the image of each individual atom increases with an increase of the ion current i associated with it and with the energy $E_{i.i}$ of the imaging ions, i.e., with an increase of the positive potential applied to the tip: $I_i = k'E_{i.i}^2$, where k' is a constant determined by the type of scintillator and by the quality of its deposition on the glass screen.

If the atom is located on a lattice site and, in this connection, protrudes above the surface of the tip, then during evaporation of the surface atomic layers by an electric field it will evaporate with greater probability and, thus, will not be observed in the ion image formed by field evaporation of the sample. On the other hand, an atom located at an interstitial position turns out to be more firmly bound to the surface of the tip due to polarization, and in the ion image formed by field evaporation of the surface it is observed as a spot of enhanced brightness. In addition, bright spots in the ion images of metallic samples may correspond to impurity atoms, which in the case of the formation of a solid interstitial solution are also located in interstitial positions.[19-21]

In order to identify the bright spots in the ion images of irradiated specimens it is necessary to take three factors into account. First, the concentration of impurity atoms in the matrix; secondly, the processes of field evaporation of the surface atomic layers (and, in general, the effect of a strong electric field on the surface of the tip), and thirdly, the effect of irradiation directly.

Unfortunately, in an ordinary ion projector it is not possible to distinguish the atom of an impurity element from an interstitial atom, and only a comparison of the ion images of irradiated and unirradiated specimens makes it possible to introduce a correction to estimates of the effects of irradiation. In order to exactly determine whether one or the other bright spot on an ion image corresponds to an impurity atom, the creation of a complicated special projector is necessary, one which is combined with a sensitive mass spectrometer, the type of atom-probe field ion microscope developed by Müller et al.[22]

Now let us consider the second factor. Upon field evaporation of atoms located on kink sites at net plane edges (in the case of a body-centered cubic lattice the atoms at such sites have four nearest neighbors) the displacement of these atoms by a short distance leads to the result that they find themselves in a position with coordination number three (three nearest neighbors).[23] Apparently[24] the deeper penetration of the field in the neighborhood of these metastable atoms leads to an increase of the probability of ionization of the atoms and molecules of the imaging gas, i.e., the brightness of the points on the ion images increases. By precisely this argument one can explain the appearance of a chain of bright spots on the ion images of certain metals, for example, the chain along the [001] zone in ion images of tungsten samples which have been subjected to field

FIG. 3. Helium ion image of a tungsten tip (T = 78°K). [20] The arrows indicate atoms which have been displaced by the forces of the electric field into metastable positions.

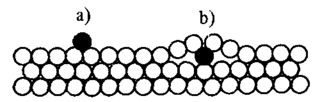

FIG. 4. Schematic diagram of a single atom A on top of a close-packed net plane and of an interstitial atom B below the first surface atomic layer. [3]

evaporation—indicated by arrow 1 in Fig. 3. In the crystal lattice of tungsten metastable positions of the atoms may occur in extremely small quantities and in other regions (for example, the regions indicated by the arrows 2 in Fig. 3).

It should be noted that the sizes of the spots of increased brightness on the ion images of the specimens oscillate from the sizes of the imaging atoms, which are not related to enhancement of the intensity, up to sizes corresponding to two or three times larger diameters. Müller[3] proposed to interpret these spots in the following way (see Fig. 4). The bright spots of larger sizes correspond to "bulges" caused by the presence of an interstitial atom under the first surface atomic layer (B in Fig. 4). The bulging out of the surface layer created by atom B increases the local intensity of the field over a wider range than does a single atom A, thus increasing the influx of atoms of the imaging gas. The atoms forming the bulge are more strongly bound than an individual atom A located on the surface.

The accuracy of a determination, with the aid of an ion projector, of the number of displaced atoms produced upon irradiation (and, in general, the very possibility of their observation) depends on the degree of mobility of these atoms under the operating temperatures of the tip in the presence of a strong electric field. If no electric field is present, the removal of displaced atoms takes place at a temperature of the order of 100° C. However, if an electric field of intensity on the order of several hundred MV/cm acts on the surface of the sample then, due to mechanical stresses caused by the field, interstitial atoms located at a depth of several atomic layers possess considerable mobility at temperatures down to 21° K.[14, 15, 18] At this temperature the sudden appearance of bright spots was observed (see Fig. 5) in ion images of tungsten samples[14] subjected to bombardment by neutral helium atoms with energies of 20 keV during the increase of the electric field to the value corresponding to the best imaging conditions. These bright spots corresponded to displaced atoms lying under the tip's surface at a depth of several atomic layers, which were being extracted from the surface by the field. Their density on the images only increased during the first seconds, but then remained without any change. When the temperature of the tip was increased to 50° K, only a few interstitial atoms remained on the surface, whereas the major part of them disappeared. With a further increase of the temperature to approximately 95° K, the number of bright spots in the image again increased, but a still bigger increase of the temperature now did not lead to any change of the picture. An intensive influx of displaced atoms toward the surface was also observed at the temperature of liquid nitrogen (78°K) in platinum bombarded by neutrons.[16] In addition, at 78°K individual bright spots of small dimensions were observed in the ion images of tungsten tips[13] which were subjected to bombardment by argon atoms.

Thus, one can conclude that in order to determine the concentration and crystallographic distribution of the single displaced atoms which appear in metallic samples upon irradiation, it is necessary to cool them (the samples) down to a temperature below ~20°K in connection with the investigation in an ion projector. In addition, for calculations of the number of single displaced atoms which are produced in the sample material by a single bombarding particle, it is necessary to take into consideration the nonuniformity of the formation of displaced atoms in proportion to its retardation, which will be discussed below.

As both calculations[5] and experimental data[18] show, the mobility of simple complexes of point defects is smaller than that of single defects. The mobility of more stable configurations (containing more than two or three point defects) is apparently limited to one specific direction (similar to a crowdion—see below) or is absent in general.

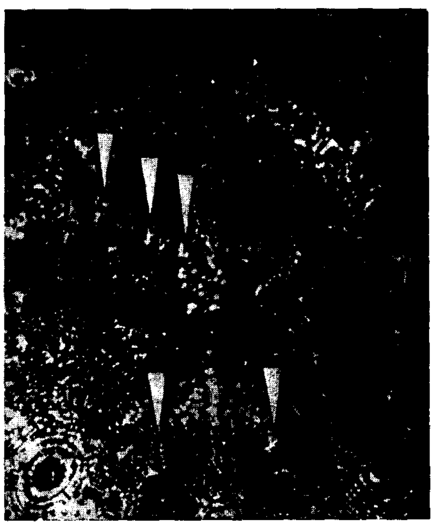

FIG. 5. Helium ion image of a tungsten tip after its bombardment by 10 mercury atoms of 20-keV energy. At least 5 displacement spikes and a number of single displaced atoms are observed. [14]

In any case, one can say that the mobility of complexes containing 5 to 10 displaced atoms in the surface layers of the crystal lattices of metals under the conditions of a strong electric field is already completely absent at 78°K. This makes it possible to observe also, with an ion projector, complexes in precisely those places where they first appeared (during irradiation), i.e., in addition to determining the concentration it is also possible to ascertain their crystallographic distribution.

2.2. Linear Defects

Dislocations are the most important type of defects in a crystal lattice. In many respects their behavior [25-27] determines the mechanical properties of crystalline solids. They play an important role in deformations, crystallization, phase transitions in the solid state, and in a number of other phenomena. In an isotropic continuous medium a dislocation is completely described by its Burgers vector **b** and by the direction **n** of its axis.

In a real crystal as a rule, independently of the care taken in its preparation and the precautions taken with its handling, on the order of 10^6 dislocations/cm^2 are present.

It is customary to assume that in an undeformed crystal the dislocations form a network, since the linear tension tends as far as is possible to reduce their overall length. In addition, a network at the sites of which three or more dislocations occur is not only stable, starting from considerations of elasticity, but it is topologically possible in many crystallographic structures.[4]

In the ion images of almost hemispherical surfaces of needle-shaped samples, dislocations are identified by the appearance of characteristic contrast effects which are produced during the process of field evaporation near the emergence of a dislocation at a surface. The interpretation of the contrast associated with perfect dislocations is based on the ideas of Pashley[28] and Ranganathan.[29] Methods for the interpretation of the contrast caused by the presence of partial dislocations are analyzed in detail in [30-35].

The determination of the Burgers vector of a dislocation is carried out by means of an analysis of the spiral figures which appear on the surface of the sample during the process of its field evaporation.[29, 36] By field evaporation of the surface atoms of the sample one is also able to easily determine the direction **n** of the dislocation line.

A perfect dislocation transforms the initial set of parallel planes (represented on the ion image as a series of concentric rings) into a spiral structure (a spiral on the ion image).

In body-centered cubic or face-centered cubic lattices the perfect dislocations intersecting the (hkl) pole of the tip always form a spiral (with the exception of the case when $\mathbf{n}\cdot\mathbf{b} = 0$), whose height is equal to the total number of interplanar spacings (hkl). If p ($p = hu + kv + lw$) is equal to unity, the appearance of a simple spiral with a height equal to the interplanar spacing is expected. When $p \neq 1$ the height of the spiral is equal to the number of interplanar spacings, and field evaporation leads to the development of p adjacent spirals.

Other types of defects which one can also conditionally relate to linear defects--the so-called focusons and crowdions[37, 38]—were observed in irradiated crystalline samples after the creation of a theory of radiation damage which gave serious attention to crystal structure.

As is well known, crowdion collisions accomplish the transportation of a crowding of atoms, and focusing collisions make it possible to transport along a closely-packed chain of atoms the impact energy received by one of the atoms of this chain.

The utilization of an ion projector also makes it possible to indirectly observe the focusing of atomic collisions in crystals. Thus, for example, by bombarding tungsten tips directly in a projector during observation of their ion images Sinha and Müller[14] observed the sudden formation of single vacancies on the surface of the specimen. These vacancies were interpreted as the result of field evaporation of a single surface atom at that moment when a packet of focused energy reaches the surface. In other words, under a voltage potential corresponding to the conditions for the best imaging, if a focused packet of energy reaches the surface then a surface atom is evaporated in the form of an ion (it is facilitated, so to speak, by the evaporation) and a surface vacancy is produced.

2.3. Surface and Volume Defects

According to the accepted classification of defects, their classification is usually carried out according to the number of dimensions in which an inelastically stressed region is macroscopic, whereas in the remaining dimensions it is microscopic (i.e., of the order of several interatomic spacings). Then point defects—single vacancies, atoms in interstitial positions, and the atoms of impurity elements—are zero-dimensional; linear defects—dislocations and lines of point defects—are one-dimensional; surface defects—grain boundaries, stacking faults, twin boundaries, etc.—are two-dimensional; and the various vacancy clusters, spikes, displacement events, etc. pertain to three-dimensional defects and bear the name of volume defects.

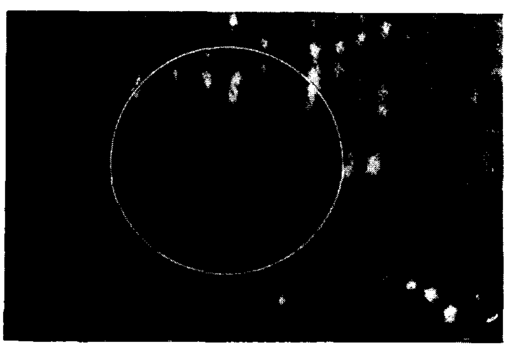

FIG. 6. Ion image of a tip of neutron-irradiated iridium, containing a compact vacancy cluster consisting of 200 single vacancies. [35]

Small voids containing several single vacancies have already been mentioned.

Fortes and Ralph,[35] investigating with the aid of an ion projector the defects which appear in an iridium crystal lattice upon neutron irradiation, observed three types of vacancy clusters which they classified in the following way. Large clusters of the first type contained approximately 200 single vacancies, compactly distributed in a nearly spherical form. A section through such a cluster is shown in Fig. 6. The second type of cluster corresponds to larger clusters which collapse to give dislocation loops, and consequently give enhanced contrast of the ion image. One such cluster is shown in Fig. 7 a, and the observed form of the contrast is schematically illustrated in Fig. 7 b. The authors[35] qualitatively explain it (the form of the contrast) in terms of the stress field of the dislocation loop. The distortion of the lattice plane rings is a maximum near the points A and B where the dislocation intersects the surface. This distortion falls off towards the center of the dislocation loop. Similar loops were observed in iridium on planes of the type $\{111\}$, and it is conjectured that they correspond to Frank sessile dislocation loops. The size of the loop suggests that they arise from the collapse of clusters containing about 250 single vacancies.

An example of the first two types of clusters in iridium is shown in Fig. 8. A section through a cluster of the first type is outlined by the rectangle A, and a section through a dislocation loop by B. Since these clusters are located at a distance of 20 Å from each other, it is statistically improbable that they arise from different causes. It is apparently more likely that the original displacement event was very energetic and produced a very large number of vacant sites which, due to self-annealing in the tip, collapsed to form two separate clusters.

In the third configuration of vacancy clusters, the single vacancies forming it are dispersed through a small volume of the crystal. In a particular case a dispersed cluster was observed containing about 30 to 40%

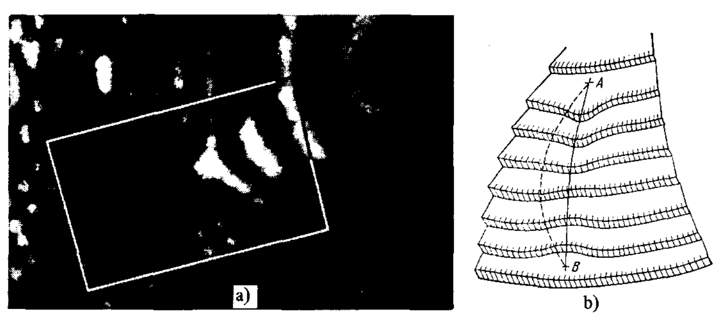

FIG. 7a. Form of the ion image produced by the intersection of a dislocation loop approximately 30Å in diameter with the surface of the iridium specimen.

FIG. 7b. A schematic diagram illustrating the distortion of the crystal planes due to the presence of a dislocation loop intersecting the specimen surface at the points A and B. [35]

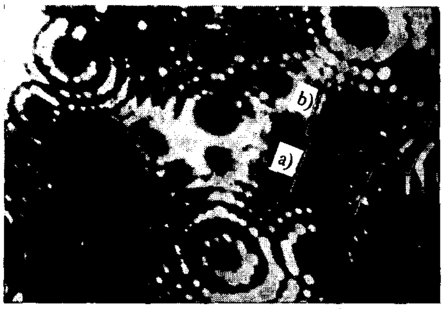

FIG. 8. Closely distributed compact cluster (A) and dislocation loop (B). [35]

FIG. 9. Helium ion image of a tungsten tip (T = 78°K). Neutron irradiation led to the formation of a large dispersed cluster. [39]

vacant sites in a cylindrical volume of length 20 Å, disposed along a [100] axis. The total number of vacant sites in this cluster is about 100.

In addition, in connection with the field-ion microscope study of neutron irradiated tungsten, Kukavadze et al.[39] observed the formation of dispersed clusters of considerably larger dimensions in comparison with the clusters observed by Fortes and Ralph in irradiated iridium.[35] The linear dimensions of the observed clusters amounted to several hundred Angstroms. Step-by-step field evaporation of the tips made it possible to expose the atomic configurations of such clusters, and it was established that they correspond to "porous" regions of the material being studied, in which on the order of 40% of the atoms in the lattice are missing. One of the ion images, containing a section through a large dispersed cluster, is shown in Fig. 9.

In view of the fact that the irradiation of polycrystalline samples may lead to the occurrence of boundary porosity of materials,[40] a field-ion microscope study of the structure of grain boundaries is of considerable interest. Thus, with the aid of an ion projector Garber et al.[41] observed screw dislocations near the conjunction of the identical planes of two grain boundaries, in the region of a small-angle boundary. A field-ion microscope study of large-angle grain boundaries in tungsten[42] exhibited the stepwise structure of the boundaries lying along close-packed planes of the sublattice and made it possible to determine the Burgers vectors of twinning dislocations at boundaries.

We also note that the extension of the techniques of the ion projector to an investigation of the disintegration of dislocations and stacking faults in crystals[43,44] is extremely important, since the extent of disintegration of the dislocations and the energy of the stacking faults to a considerable extent determine the kinetics of flow for stages IV and V of annealing in materials subjected to irradiation.

3. PASSAGE OF RADIATION THROUGH MATTER

A theoretical investigation of the processes which take place during the passage of different kinds of radiation through matter, and also a summary of the extensive data concerning experimental investigations (by different methods than the field-ion microscope) of these processes does not appear in the present article since these topics are contained in extensive reviews and monographs, for example, [40,45-52].

4. EXPERIMENTAL ARRANGEMENT AND THE CHOICE OF CERTAIN PARAMETERS

4.1. The Choice of Material for Investigation

The usefulness of one or the other pure metal for study in an ion projector is basically determined by the ratio of two factors.

One is, in the first place, the value of the electric field intensity F_{im} at which field ionization of the atoms of the imaging gas being used occurs above the hemispherical surface of the sample. It only depends on the type of gas and may be approximately estimated according to the formula[23]

$$F_{im} = 3{,}7\, U_i^{3/2}, \tag{4.1}$$

where F_{im} is in MV/cm and the ionization potential U_i is in eV.

Calculated values of the image field for a number of gases, which appear to be promising from the point of view of their use as imaging gases in an ion projector, are given in the review [23].

The second factor is the value of the electric field intensity on the surface of the tip, at which evaporation of the surface atoms in the form of ions begins. This field characterizes only the metal itself and is reached in that case[23] when the energy of the electrical forces acting on the ions plus a small thermal activation energy begin to exceed the binding energy, to which a polarization term should also be added. The theory of field evaporation, using the concept of image forces,[9,53] gives the following expression for the field strength for evaporation, D_{evap}:

$$D_{\mathrm{evap}} = n^{-3}e^{-3}\left[\Lambda + \sum^{n} U_n - n\varphi + {}^1/_2\,(\alpha_0 - \alpha_i)\,D_{\mathrm{add}} - kT\ln(\tau/\tau_0)\right]^2, \tag{4.2}$$

where ne is the single or multiple charge of the ion (n = 1 or 2), Λ is the vaporization energy of the metal atom, φ is the work function of the specific crystallographic plane, α_0 is the polarizability of the metal atom located at the surface lattice site, α_i is the polarizability of the free metal ion, τ is the time required for evaporation, and τ_0 is the vibration period of the bound surface atom.

In the case when the imaging gas and the metal are chosen such that

$$F_{im} \geqslant D_{add},$$

it is impossible to observe a stable ion image of the surface of the specimen in an ion projector since field evaporation of the surface atomic layers of the tip begins earlier (the > sign) or simultaneously with the start of imaging of this surface. In order for it to be possible to investigate a given metal in an ion projector, it is necessary to fulfill the condition $F_{im} < D_{evap}$.

As experiment showed (for example,[23, 54]), the use of different gaseous mixtures in order to image the surface of needle-shaped samples makes it possible to investigate a rather broad circle of metals and a number of their alloys. However, from the point of view of the investigation of point defects in irradiated metals, as yet only a few refractory metals give satisfactory results; among these it is apparently most advisable to choose the following: from metals with a body-centered cubic lattice—tungsten and tantalum, from metals with a face-centered cubic lattice—iridium and platinum, and from metals with an hexagonal close-packed structure—rhenium. The values of the evaporation fields for these metals substantially exceed the field required for field ionization of helium (450 MV/cm), whose utilization, as is well known, makes it possible to achieve the best resolution in an ion projector.

4.2. Choice of the Kind of Irradiation

The choice of the type of particles which it is proposed to use for irradiation of the samples for a field-ion microscope study of radiation damage in crystal lattices is largely determined by the goals of the experiment to be carried out.

Investigations using an ion projector of the radiation effects due to neutron irradiation are of special interest in order to clarify the mechanism of change of properties of the construction materials used in nuclear facilities. In this sense a parallel investigation by different methods of the effect of neutrons on materials (the field-ion microscope, roentgenography, methods of measuring different mechanical and physical properties) is extremely useful. In addition, there is a great deal of interest in the study of the damage to a crystal lattice associated with the passage of fission fragments through it. Field-ion microscope investigation of the defects in materials bombarded by electrons and their irradiation by heavy charged particles apparently can give useful information with regard to the development of a general theory of radiation damage. The use of an ion projector probably also can facilitate the detailed study of cathode sputtering processes in materials.[55]

4.3. Method of Irradiating the Samples

The method of irradiating the material being studied may introduce important differences in the results of field-ion microscope studies of radiation damage. All experiments involving an investigation of radiation damage with the aid of an ion projector may in this sense be divided into two basic groups:

4.3.1. To the first group belong experiments in which the investigated material is exposed to radiation in the form of fine wire, after which tips are prepared from it (by means of electrochemical etching). In this case a tip prepared from an unirradiated wire or from a wire which has been subjected to thorough annealing may serve as a standard.

4.3.2. To the second group of experiments belong those in which tips prepared beforehand are subjected to irradiation. Here several variations are possible:

4.3.2a. Tips prepared beforehand are placed in the projector, and after field evaporation of several tens of surface atomic layers which have been contaminated by adsorbed gases, the ion images of the cleaned surfaces are photographed. Then the tips are removed from the projector, fastened to an experimental target, and placed in a beam of the radiation source being used, after which their repeated investigation in the projector is carried out. *Here the requirement, that the time for* transfer of the tip under the beam of irradiating particles and back to the projector should be minimal, is extremely important. In addition, repeated investigation of irradiated tips is an ion projector may be undertaken every time only after removal of contaminated surface atomic layers by the field. Upon investigation of irradiated tips in a projector, the step-by-step field evaporation of many surface atomic layers one after the other and the photographing of their ion images makes it possible to ascertain the volume distribution of the defects which appear upon irradiation. A comparison of the ion images of one and the same tip obtained before and after irradiation makes it possible to separate the defects due to irradiation from the defects present in the specimen material before irradiation.

4.3.2b. Tips prepared beforehand are irradiated directly in the projector, where the electric field is reduced at the time of irradiation. A few ion images of the surface cleaned by the field are photographed before irradiation and a whole series after irradiation. The beam of irradiating particles may be introduced into the projector directly from an accelerator or from another source. The formation of the beam of bombarding particles, their introduction into the projector, and the measurement of the beam intensity are, in each concrete case, independent technical problems.

4.3.2c. In this case tips prepared beforehand are irradiated directly in the projector while observing their ion images. In this connection it is possible, by using a comparator technique,[56, 57] to obtain three-color ion images of the surface of the tip on which points of one color correspond to the surface atoms which disappear after irradiation, points of another color correspond to atoms which are not observed on the ion image of a given surface before irradiation and appear on it as a consequence of irradiation. And finally, points of the third color (representing the addition of the first and second colors) correspond to surface atoms which are unchanged as a result of irradiation.

In addition, step-by-step field evaporation of the surface atomic layers of the tip after its irradiation makes it possible to ascertain the volume distribution of defects, and in the case of damage of considerable dimensions—to determine their atomic configuration.

Each of the indicated methods has its own advantages and shortcomings in one or the other specific case. For example, it is practically impossible to carry out an investigation of neutron irradiation with the aid of an ion projector according to methods 4.3.2b and 4.3.2c. This is explained by the fact that a noticeable effect in crystal lattices associated with their neutron irradiation is achieved in that case when the integrated neutron flux exceeds 10^{12} to 10^{13} neutrons/cm^2, but it is not possible to obtain such fluxes with the aid of natural sources (during a comparatively short time interval). Therefore, in all neutron experiments it is necessary to irradiate the samples in the experimental channels of nuclear reactors.

In the case of an investigation of the damage which arises in the crystal lattices of metals upon their irradiation by heavy charged particles with energies up to several MeV, the irradiation of fine wires out of which tips are then prepared and the irradiation of previously prepared tips (according to the method 4.3.2a) give strikingly different results. Thus, Suvorov and Kukavadze[58] showed that if the irradiation of previously prepared tips[59] (method 4.3.2a) by deuterons with an energy of 12 MeV (a total flux of 1.9×10^{11} deuterons/cm^2) leads to the appearance of considerable (encompassing large many-atom regions) damage in the crystal, then irradiation of a fine wire by the same source, with subsequent preparation of tips from it makes it possible to observe only individual point defects, dislocations, and in rare cases small vacancy clusters of diameter up to 10 Å. In this case apparently the irradiation of fine wires gives results which more correctly describe the nature of the defects which arise in large volumes of material. However, in such cases it is advisable to carry out a parallel irradiation of the samples by the two indicated methods and to then compare the results obtained. Ion images of tips prepared from irradiated fine wires give an idea about the nature of the defects which arise, their distribution and quantity, whereas the images of irradiated tips, upon comparison with their images obtained before irradiation, assist one to eliminate from the first estimates of the defects due to causes other than irradiation (see Sec. 5).

Upon using methods 4.3.2b and 4.3.2c for the irradiation of samples in connection with a field-ion microscope investigation of radiation damage, one should keep the following in mind. As Müller showed,[24] the presence of a strong electric field at the surface of the tip causes an expansion of the lattice of the specimen by up to 10%. This may have an important effect on the nature of the damage on the surface of the irradiated samples, and sometimes also in their volume, which considerably hinders a determination of the nature of the defects observed in the ion images.

Thus, in order to eliminate ambiguities associated with the interpretation of the ion images of tips which have been irradiated directly in the projector, it is advisable to carry out irradiation without applying a strong electric field to the sample. However, in this connection, in order to obtain the possibility of a direct comparison of the ion images of one and the same tip surface before and after irradiation, it is necessary that no changes at all occur on this surface except for changes caused directly by the bombardment by particles, i.e., during the irradiation process (in which the strong electric field on the surface of the tip is reduced) contamination and corrosion of the surface are not permitted. This becomes feasible upon the creation of an ultrahigh vacuum in the projector and upon the provision for an intense beam of bombarding particles (in order to achieve maximum reduction of the irradiation time).

Such a version of investigation was implemented by Petroff and Washburn[60, 61] in order to investigate the defects which appear in the crystal lattice of iridium upon its irradiation by protons with an energy of 10 MeV. Before irradiation a curve showing the dependence of the rate of contamination and corrosion of the specimen surface on the time was plotted without applying an electric field to the specimen. For a vacuum of 10^{-9} Torr in the chamber of the projector, changes on the specimen surface were first observed after eight minutes. A cyclotron, however, can provide a pulse of protons with a high density of particles and a time which is considerably smaller than the time during which the surface of the tip would remain without changes under the given conditions.

The authors of [60, 61] observed that upon irradiation of the tips in the presence of a strong electric field on their surfaces (necessary for field ionization of the imaging gas) the observable surface damage is more extensive than upon irradiation of the samples without a field.

4.4. Energy of the Bombarding Particles and Integrated Radiation Fluxes

In connection with an investigation with the aid of an ion projector of the defects which arise in the crystal lattices of samples upon their irradiation, the choice of the energy of the particles and the necessary total flux per unit surface area are basically determined by the following factors: a) the type of bombarding particles; b) the method used to irradiate the sample; c) the properties of the material being studied; d) the objectives of the investigation.

In connection with the irradiation of samples by heavy charged particles (protons, deuterons, α-particles) the use of high energies may turn out to be advisable only in the case of irradiation according to the method 4.3.1. In this connection the integrated fluxes of radiation must be sufficiently high (10^{12} to 10^{15} particles/cm^2) so that their particles will pass straight through the irradiated fine wires, and only a small fraction of the energy lost by them to the material of the sample will be expended in the formation of stable defects (vacancies, displaced atoms, etc.). The major fraction of the energy of the particles will be expended in the ionization of atoms in the crystal lattice of the sample, which are rapidly neutralized by conduction electrons and cannot be observed. For smaller total fluxes of irradiating particles the concentration of defects which appear turns out to be so low that its determination becomes extremely difficult.

With a lowering of the energy of the bombarding particles its share, which is expended on the formation of stable lattice defects (on elastic interactions) during passage through the sample material, increases and a perceptible (with the aid of an ion projector) concentration of defects is obtained for smaller integrated fluxes.

Variation of the energy of the bombarding particles and of the integrated radiation fluxes within definite limits may enable one to establish the dependence of the nature of the defects, their distribution, and concentration on the different parameters of the beam.

However, for the irradiation of samples by heavy charged particles (method 4.3.1) their energy must not be less than a certain energy E_{min}, which corresponds to the range of the particles in the sample material, equal to or somewhat larger than one-half the diameter of the fine wire being irradiated. It is established that upon etching of the tips their points fit, with a certain spread, on the axes of the original wires.

For the irradiation of samples by electrons, the energy of the electrons must be chosen to correspond to relativistic velocities, since only in this case are the electrons able to cause displacements of atoms in the lattice. If tips prepared beforehand are subjected to irradiation by heavy charged particles, then the energy of the particles should be chosen to be smaller than in the case of irradiation of fine wires, since sputtering of the material in the tips at the place where the beam is incident and also possible thermal heating[59] may lead to its becoming blunted, as a result of which its investigation in an ion projector becomes impossible.

It is most convenient to carry out neutron irradiation of samples in the form of fine wires, with subsequent preparation of the tips. In this connection the energy of the neutrons may be chosen to be arbitrary (thermal neutrons, fission spectrum), and the necessary integrated fluxes must lie within the limits from 10^{13} to 10^{20} neutrons/cm^2. As Fortes and Ralph[35] showed, for neutron irradiated iridium with integrated fluxes of 10^{13} and 10^{14} neutrons/cm^2, the imperfections which appeared could be identified in the ion projector.

It is interesting to perform the irradiation by neutral atoms of previously prepared tips directly in the projector, choosing their energies within the limits between 10 and 100 keV.[13, 14] In this case the fraction of the energy expended in elastic collisions is so substantial that even the incidence of individual atoms on the tip is reflected by changes on the ion images. At the same time such energies are sufficient in order to be able to pass right through the tip, so that one can study the damage (effects) at the places where the bombarding particles enter the crystalline lattice of the sample and at the places where they emerge from it.

We also note that in connection with the choice of integrated radiation fluxes it is necessary to be guided by the type and quality of the material being investigated, i.e., it is necessary to consider the possible presence of defects in the sample material prior to irradiation, and also the physical and especially the mechanical properties of the sample material in the sense that the defects which appear upon irradiation do not make the samples unsuitable for field-ion microscope analysis (see Sec. 5.3 below).

In addition, the method of irradiation, in some way determining the energy of the bombarding particles and their integrated flux, depends on the goals of the investigation. Thus, for example, if the objective of the investigation is a study of individual stages of the annealing of different kinds of defects, then it is necessary to irradiate fine wires because prolonged tempering of irradiated tips (for the purpose of annealing the defects which arise) inevitably leads to such a blunting of the tip that its subsequent analysis in an ion projector becomes impossible.

5. THE INFLUENCE OF SECONDARY FACTORS ON THE NATURE OF THE DAMAGE OBSERVED IN IRRADIATED SAMPLES

The ion projector is an ideal device for investigating the effects of radiation, because almost all kinds of possible changes in the crystalline lattice of the sample can be identified at an atomic level on the ion images of the irradiated samples obtained with its aid. However, one must be extremely careful in interpreting the imperfections observed in the ion images of irradiated samples, because these imperfections may be caused by certain other causes, different from irradiation.

5.1. Defects Present in the Sample Material before Irradiation

Such defects may be divided into two groups:

a) Defects which were formed during the preparation of the fine wires (foils or rods), which are intended for the preparation of the tips.

b) Defects which are formed during the preparation of the tips (for example, because of the asymmetric nature of the electrochemical etching process)—in the case of irradiation of tips prepared beforehand.

In order to separate the defects which are related to the asymmetric nature of the etching process and the defects which are always present with a certain equilibrium concentration in unirradiated and thoroughly annealed samples, it is necessary before irradiation to carefully scan the ion images of all samples and to carefully compare them with the images obtained after irradiation. In the case of irradiation of samples in the form of fine wires, ion images of tips prepared from initial batches of unirradiated wires serve as the standard, but in the case of irradiation of tips prepared beforehand—ion images of these tips, obtained before irradiation, serve as the standard.

5.2. Oxidizing Processes

In the case of tips prepared beforehand, which are being irradiated outside of the projector, in order to interpret their ion images it is necessary to consider the possible oxidizing action of the atmosphere during the time taken to transport the tip from the projector to the chamber for irradiation and back.[62]

An ion image of a tungsten tip (see Fig. 10) irradiated by deuterons with an energy of 12 MeV[59, 62] may be cited as an example. Irradiation was carried out according to method 4.3.1.

It would apparently be incorrect to unequivocally attribute the observed, heavily damaged cross-shaped region in the neighborhood of the {112} type faces and the central (011) plane to the result of radiation effects. One can designate oxidation of the tip of the specimen as the most probable reason for its creation because rather similar defects were sometimes observed in unirradiated tips which had been exposed to the air for a long time.

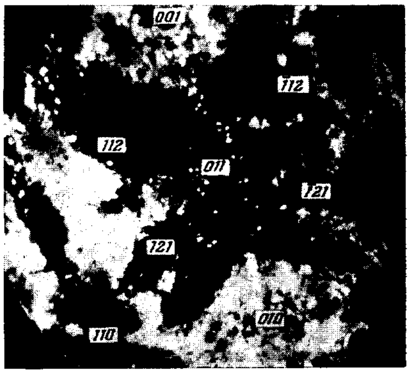

FIG. 10. Helium ion image of the surface of a tungsten tip which is undergoing oxidation. [62]

The field-ion microscope investigation of the interaction of oxygen with tungsten carried out by Sugata et al.[63] showed that exposure of tungsten at 550 °K to oxygen at a pressure of 4×10^{-3} Torr for a period of five minutes leads to a penetration of the oxygen atoms into the crystal lattice of the tungsten to a depth equal to 70 atom layers. Therefore, for the purpose of eliminating the influence of oxidizing processes it is necessary to reduce the time of exposure of the tips to the air to a minimum, and each analysis of an irradiated tip in an ion projector begins only after the removal by the field of several tens of surface atom layers which have been distorted by oxidation and contaminated by adsorbed gases.

5.3. Mechanical Stresses (Effect of the Electric Field)

The fact that tips in an ion projector both during the formation of their ion images and during the field evaporation of surface atomic layers experience enormous mechanical stresses[64] (of the order of 1000 kilograms/mm^2) on the part of the applied electric field is responsible for the main errors in the identification of imperfections in the ion images of irradiated samples. These stresses by themselves may be the sources of different kinds of defects which, for the irradiated specimens, are combined with the radiation effects.

The formation of point defects under the influence of a strong applied electric field is analyzed in article [23]. There the possibility of cracks developing in the samples is indicated.

The presence of a strong electric field at the surface of the tip may lead to the occurrence of plastic deformation in its material and to its partial or complete destruction.[65]

It should be noted that the sizes and nature of the imperfections caused by the field to a considerable extent depend on the defects which appear as a result of irradiation. Thus, in certain cases the radiation defects may, as it were, facilitate destruction of the tip by the forces associated with the electric field. Shearing processes[39, 59] may occur in it, and the ejection of substantial regions of the tip material.[61] Therefore, from the form of the imperfections which are produced during observation of the ion images of irradiated samples, one can qualitatively estimate the nature of the radiation defects, which facilitate destruction of the tip by the electric field forces.

In order to separate the imperfections associated with plastic deformation, occurring in the tip material due to the influence of the field forces, from the radiation defects it is necessary to continuously observe the ion images and the pictures of field evaporation of the surface atom layers of the tip during the entire procedure of its investigation in an ion projector.[61] The occurrence of plastic deformation is noted as a sudden change in the form of the image. In connection with field evaporation of a tip, the distribution of the brightness of the smeared regions of the image changes suddenly as a consequence of plastic deformation.

6. EXPERIMENTAL RESULTS

6.1. Neutron Irradiation

At the present time field-ion microscopy has already given a certain amount of direct information about the nature of the defects which appear in tungsten,[12, 13; 39, 66–70] platinum,[16, 17, 71] iridium,[35] and molybdenum[15] upon neutron irradiation. The results of these investigations are briefly summarized in Table I. Certain results are analyzed in the review article by Ralph.[72] In all of these investigations neutron irradiation led to the formation of single displaced atoms, vacancies, and small (10 to 15 Å in diameter) vacancy clusters.

Attardo and Galligan[67] investigated the accumulation of voids at or around dislocations in irradiated tungsten samples. In general the accumulation of voids may occur in two different ways.[73] First, one can expect the extent of the damages at dislocations to be higher because of defocusing of a primary knock-on atom, and second, in connection with the random diffusion of vacancies one can expect that they will be "trapped" by a dislocation. The authors of [67] showed that the formation of the voids which are observed at dislocations in irradiated samples excludes their accumulation through thermally activated processes because the vacancies introduced by neutron bombardment were removed at a temperature of 800° C.[12] The temperature of irradiation of the samples in the reactor did not exceed 70°C. In addition, a proof of the radiation nature of the observed voids is the fact that such voids were not observed at dislocations in the annealed tungsten used in the indicated experiments.

In addition to the defects already observed, (in neutron irradiated tungsten) Kukavadze et al.[39] observed large dispersed clusters, and with the aid of field evaporation of many surface atom layers, volume models of these clusters were constructed. It should, however, be noted that diagrams of clusters constructed according to ion images do not describe the form of the imperfections produced by neutron irradiation with sufficient accuracy. The process of electrochemical etching, which is used in order to prepare the tips, turns out to have some influence on the form of the defect. Thus, if a fine wire is being etched at a place where such extensive damage formed as the result of irradiation is located, it is necessarily made worse (the edges of the cluster are etched, a certain amount of the etching penetrates inside the defect).

Table I. Experimental Data on Field-Ion Microscope Investigation of Radiation Defects in Metals

Bombarding particles, their energies	Integrated radiation dose, particles/cm^2	Material investigated	Method of irradiation	Temperature of investigation	Observed defects	Additional investigations, remarks	References
Neutrons	2×10^{18}	Tungsten	Irradiation of fine wires in a reactor with subsequent preparation of tips	78° K	Single vacancies–concentration 10^{-4}, vacancy clusters–up to 10 single vacancies in each cluster–density 10^{15} to 10^{16} clusters/cm^3		13
»	5×10^{17}	Tungsten, tungsten + 5% rhenium	The same	78° K	Single vacancies–concentration 1.2×10^{-3}, vacancy clusters of diameter up to 10Å–concentration 10^{-4}, divacancies–concentration 10^{-4}; vacancy clusters at grain boundaries–up to 50 single vacancies in a volume of diameter 15Å	Defects at grain boundaries	66
Neutrons, E > 1 MeV	10^{18}	Tungsten	» »	78° K	Single vacancies–concentration 10^{-2}, vacancy clusters of diameter up to 15 Å	Annealing of defects	12
Neutrons, E > 1.45 MeV	10^{18}	»	» »	78° K	Voids at dislocations		67
Neutrons	10^{17}	»	» »		Single vacancies–concentration 10^{-4}, vacancy clusters containing up to 100 single vacancies in a spherical volume–concentration 10^{-5}		69
Neutrons of the fission spectrum	1.5×10^{19}	»	» »	78° K	Single vacancies, single displaced atoms, dislocations, vacancy clusters of diameter up to 15 Å, dispersed clusters with linear dimensions up to 500 Å		39
Neutrons		Tungsten	» »	78° K	Single vacancies, chains of atoms in the region of {016} faces	Relaxation of microstresses, X-ray investigations	70
»	10^{18}	Platinum	» »		Complexes of several interstitial atoms, small vacancy clusters		16
Neutrons E > 1.4 MeV	10^{18}	»	» »		Regions of violation of the crystalline structure of the type of the depletion bands of Seeger [75]–concentration of 6×10^{15} regions/cm^3		71
Neutrons, E > 1.45 MeV	10^{16}, 10^{17}, 10^{18}, 5×10^{19}	»	» »		Dislocation loops of dimension 40 to 60 Å in the {110} planes–concentration 3×10^{14} loops/cm^3, depletion zones of size from 10 to 40 Å in diameter, single interstitial atoms, single vacancies	Annealing of defects in stages III and IV	17
Neutrons	10^{13}, 10^{14}	Iridium	» »		1) Clusters containing up to 200 single vacancies, compactly distributed in a spherical volume. 2) Regions obtained by the collapse of dislocation loops. 3) Dispersed clusters containing 30 to 40 single vacancies in a cylindrical volume of length 20 Å along the [100] axis		35
Deuterons 12 MeV	2×10^{11}	Tungsten	Irradiation outside the projector of tips prepared beforehand	53—63° K, 78° K	Removal of material from the tip at places where the deuterons enter the specimen, regions with a frozen-in thermal disordered nature, single vacancies, single displaced atoms, dispersed clusters, displaced zones, removal of material from the tip at the places where the particles exit from the sample		59, 76
α-particles, 5.4 MeV	Individual hits on the tip	»	Irradiation inside the projector of tips prepared beforehand		From 15 to 30 single displaced atoms, complexes of 2 to 3 displaced atoms		78
α-particles, 5.0 MeV	The same	»	The same	78° K	Single vacancies, single displaced atoms		77, 80
α-particles, 12.7; 15.7; 18.4; 23.4 MeV	1.6×10^{16}, 1.4×10^{16}, 1.9×10^{15}, 1.7×10^{15}	»	Irradiation of fine wires with subsequent preparation of tips	78° K	Single vacancies, single displaced atoms–density within the limits from 2×10^{19} to 3×10^{20} atoms/cm^3 for energies of 18.4 and 23.4 MeV, complexes of displaced atoms–density 10^{18} and 3×10^{19} complexes/cm^3 respectively, vacancy clusters of diameter 10 to 15 Å		82
Deuterons, 12 MeV	2×10^{11}	»	The same	78° K	Single vacancies, atoms in interstitial positions, dislocations and vacancy clusters of diameter up to 10 Å		58

Table I. (cont.)

Bombarding particles, their energies	Integrated radiation dose particles/cm^2	Material investigated	Method of irradiation	Temperature of irradiation	Observed defects	Additional investigations, remarks	References
Deuterons, 4, 5, 8, 10, and 12 MeV	2×10^{11} to 10^{13}	Tungsten	Irradiation of fine wires with subsequent preparation of tips and irradiation outside the projector of tips prepared beforehand	78° K	Individual displaced atoms, single vacancies, small complexes of displaced atoms, dislocations, and in a number of cases—vacancy clusters (see text)		18
Deuterons, 0.1 MeV	1.2×10^{12}	»	Irradiation outside the projector of tips prepared beforehand	78° K	Single displaced atoms, 6 to 8 displaced atoms per single bombarding deuteron, single vacancies, complexes of 5 to 8 displaced atoms of diameter of the order of 15 Å in the neighborhood of {112} type faces, and in rare cases dislocations		62
Protons, 10 MeV	3×10^{13}	Iridium	Irradiation in the projector without a field of tips prepared beforehand and during observation of their ion images	10° K, 78° K	Single vacancies and interstitial atoms, deposited on certain planes of atomic layers		60, 61
Electrons 1 MeV		Tungsten	The same	78° K	Single vacancies, single displaced atoms		3
Electrons, 240 MeV	10^{16}	»	Irradiation of fine wires with subsequent preparation of tips	78° K	Single displaced atoms—density 5×10^{17} atoms/cm^3, vacancy clusters of diameter up to 10 Å		90

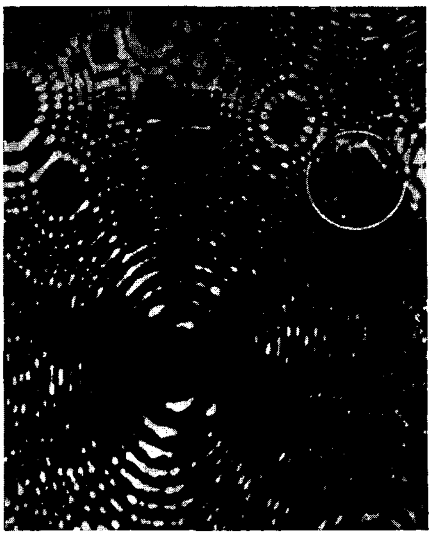

FIG. 11. Helium ion image of a neutron-irradiated platinum tip. The disturbed region which appears is surrounded by a small circle. [71]

The strong electric field which exists inside an ion projector plays an even more important role. Possible displacements in the crystal lattice were mentioned above. In addition, the enormous mechanical stresses caused by the field facilitate the removal of material from the tip in the case of the formation of "porous" regions.

With the aid of an ion projector Attardo and Galligan[16] were able to prove directly that complexes of interstitial atoms and vacancy clusters are present in platinum irradiated by neutrons at a temperature below stage III of the recovery spectrum[74] (the irradiation was carried out at 75°C). It is interesting that single isolated vacancies and divacancies were generally not observed in the experiment under consideration. Complexes of displaced atoms turned out to be more weakly bound than individual surface atoms, located at normal lattice sites. The observed shapes of the complexes of displaced atoms—lines and triangles—were not observed on either pictures of field evaporation of annealed platinum or in heavily damaged regions from which large volumes of material are removed by the field forces.

Imperfections similar to Seeger's depleted zones[75] were observed in irradiated platinum.[71] One of the obtained ion images of irradiated platinum is shown in Fig. 11. The region of damage which appears as a result of the irradiation is enclosed by a small circle. Diagrams are presented in the article for the spatial distribution of point defects—displaced atoms and vacancies—in the disturbed regions which arise upon irradiation. The sizes of such regions range from 10 to 30 Å, where the largest number of disturbed regions observed had sizes of the order of 10 Å.

Attardo and Galligan investigated recovery processes associated with the annealing of crystal lattices of tungsten and platinum[17] which had been stressed as a result of irradiation.

In the case of tungsten it was shown that the resulting vacancy clusters, whose sizes reached 15 Å in diameter, may increase due to the ability of certain defects to move at temperatures below 70°C. The concentration of single vacancies is annealed (at 1500°C for a period of three hours) tungsten was equal to 10^{-5}, whereas in irradiated samples it was equal to 10^{-2}. Recovery of the regular structure upon annealing took place at the expense of the removal of the vacancies, which is most effective at temperatures above 700°C.

In connection with the investigation of the annealing of defects in platinum,[17] by using different radiation doses of neutrons at 100 and 340°K the creation of a significant number of depleted zones was observed,

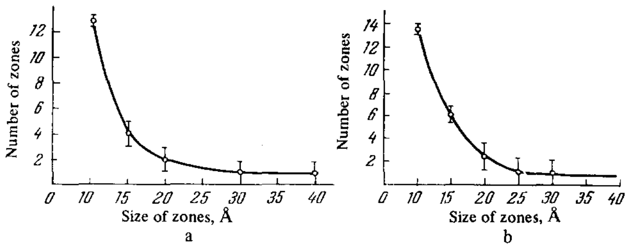

FIG. 12 a) Size distribution of the depleted zones: the platinum samples were irradiated by fast neutrons (E > 1.45 MeV) at 100°K and annealed at 280°K; b) size distribution of depleted zones after annealing at a temperature corresponding to stage III. [17]

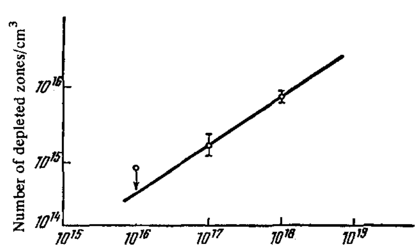

FIG. 13. Integrated flux dependence of the total number of depleted zones in neutron irradiated platinum (E > 1.45 MeV). [17]

whose sizes ranged from 10 to 40 Å in diameter. Annealing at 280 and 373°K of platinum samples irradiated at 100°K made it possible to observe strong changes in the size distribution of the depleted zones, where the new distribution (Fig. 12) was consistent with the distribution existing for samples irradiated at 340°K. However, annealing at 773°K led to an increase in the number of depleted zones having intermediate sizes, whereas small size zones were completely removed. An analysis of the change in the nature of the size distribution of the depleted zones in stage IV of the annealing of irradiated platinum permits one to conclude that vacancies possessing appreciable mobility are responsible for these changes. It was shown that the number of depleted zones increases linearly with an increase of the integrated radiation flux (Fig. 13).

Single interstitial atoms produced as a result of irradiation of the samples were removed from the platinum at a temperature corresponding to stage III of annealing.

Annealing irradiated platinum above 873°K removes all vestiges of damage introduced at lower temperatures.

In order to establish the presence of a partial relaxation of microstresses, Kirienko and Potapov[70] used an ion projector to investigate tungsten irradiated by neutrons (at 27°C) and subjected to annealing at 200, 250, and 350°C. In addition to the radiation defects usually observed (single vacancies, chains of atoms), the authors of [70] photometrically determined the change in the width of the interference line (from Debye powder diagrams, obtained by Cu-irradiation of the same sample after a series of successive annealings).

6.2. Bombardment by Neutral Atoms

Brandon et al.,[13] by modifying an argon ion gun, were able to extract argon ions with energies up to 30 eV and, by subjecting them to charge exchange, were able to produce a beam of low-energy neutral atoms. A beam of these atoms was introduced into the ion projector (operating at a temperature of the tips of 78°K), and the tungsten tips were directly bombarded while observing their ion images. Charge exchange took place in appreciable amounts directly inside the projector bulb under an argon pressure of 10^{-5} Torr. Operation under an argon pressure of 10^{-5} Torr and a helium pressure of 5×10^{-3} Torr did not create any difficulties with regard to the helium ion imaging.

Following a five-second bombardment of a tungsten tip by argon atoms with energies of 150 eV, several displaced atoms appeared on the surface opposite to the beam, whereas on the source side a large number of atoms were completely removed.

Bombardment for fifteen seconds caused the formation in the crystal lattice of the tip of damages encompassing more extensive regions. Interstitial atoms were mainly observed in the region of the central (011) plane and type {112} faces. A preliminary investigation of the threshold energy for passage right through the samples show that it lies between 120 and 140 eV, corresponding to the threshold for displacements of the atoms, 70 eV $< E_d <$ 85 eV. Sputtering of the tungsten atoms from the side of the source of the neutral argon atoms was observed even at appreciably smaller energies.

Sinha and Müller[14] bombarded tungsten tips, set up in an ion projector, with neutral atoms of helium having an energy of 20 keV and with mercury atoms. The atomic beam was generated by producing a beam of ions (from a Penning discharge tube) in a side arm of the microscope and by its acceleration into the system using two lenses. Then the ions were deflected by a magnetic field, and the neutral atoms generated as a result of charge exchange were directed toward the tip. The intensity of the beam guaranteed, on the average, one collision per second. The tips were irradiated both during observation of their ion images and upon removal of the electric field. Three types of defects were observed on the surfaces of the irradiated tips: single vacancies, interstitials and their clusters (see Fig. 5), and disordered regions of diameter from 50 to 100 Å. Defects were observed on both sides of the irradiated tips; however, their densities were different.

The observed and calculated values for the number of displaced atoms produced during a five second bombardment by neutral helium atoms and for different pressures of the helium imaging gas are given in Table II. It should be noted that in the case of bombardment of the tips in the presence of a strong electric field, the defect density produced on the surface is higher than in the case when the field is not present. This may be explained by the fact that, in addition to the neutral atoms, the helium ions in the beam also bombarded the tip. During the use of mercury atoms as the bombarding particles, their separation from the ions was not carried out. The mercury was condensed in a liquid-nitrogen trap below the Penning discharge tube, and when the tip was to be bombarded, the trap was gradually warmed up. Immediately after bombardment two

Table II

Helium gas pressure, 10^{-3} torr	Number of displaced atoms counted on the surface of a bombarded tip	Theoretical value in the volume of tip
1.5	4	8
1.8	5	11
2.5	22	16
3.0	44	31
3.5	52	72

basic types of defects were observed on the ion images. First, a large number of vacancy clusters in which a complete disordering of the structure occurred. Such regions were uniformly distributed over the entire ion image. And secondly, field evaporation removed entire pieces from the surface. This was explained by the fact that mercury atoms impart an energy to the tungsten atoms which considerably exceeds the energy transferred by bombarding helium atoms. Field evaporation of a region of diameter up to 200 Å, containing approximately 5×10^3 atoms, was observed.

6.3. Bombardment by Heavy Charged Particles

The field-ion microscope investigation of the results of bombarding metallic samples by heavy charged particles (protons,[60, 61] deuterons,[18, 58, 59, 62, 76] and α-particles[77-82]) has been carried out for various materials using different methods for bombarding the samples and particles of different energies. The basic results of these investigations are collected in Table I.

Müller[78] and shortly afterwards Brandon and Wald[77, 80] published the results of observation of point defects in the structure of tungsten tips which were bombarded by α-particles having an energy of 5.4 MeV. In both cases polonium sources of α-particles of an intensity from 0.5 to 1.0 millicurie, which were fastened in a copper cylinder at a distance of 1 cm from the tip, were used for irradiation. On the average one α-particle was incident on the tip every three hours[78] and every incidence was seen, and also the damage to the lattice was observed only from the side of emergence of the particle from the sample material. Displacements of 15 to 30 atoms took place inside regions approximately 50 Å in diameter. Two-thirds of the displaced atoms recalled atoms in interstitial positions just below the surface (see Sec. 2.1) whereas one-third of the displaced atoms vanished from the surface. This might represent the end of a displacement spike at the surface.[85] In half of the cases smaller defects were seen in the neighborhood of emergence of the particle, for example, from one to three distributed in a close group of atoms in interstitials, which appeared to the side away from the site of the primary damage at distances up to half the radius of the tip.

An investigation of the dependence of the nature of the defects in single crystals of tungsten on the energy of the bombarding particles and on the method of irradiation was carried out by Suvorov and Kukavadze.[18] For deuterons with energies of 4, 5, 8, 10, and 12 MeV and integrated radiation fluxes lying within the limits from 2×10^{11} to 10^{13} deuterons/cm^2, single vacancies (concentration equal to 5×10^{-5} for energies of 4, 5, 8, and 10 MeV) and individual interstitial atoms were observed in all specimens, independently of whether wires or previously-prepared tips were irradiated.

Small vacancy clusters of diameter up to 10 Å were observed on the ion images of samples irradiated by deuterons with energies of 8, 10, and 12 MeV. In the case of direct irradiation of the tips, vacancy clusters of small sizes were also observed in the specimens which were irradiated by deuterons with energies of 4 and 5 MeV, but for energies of 8, 10, and 12 MeV, the formation of substantial vacancy clusters encompassing large many-atom regions was observed in which almost half of the atoms in the lattice were removed.

Small complexes containing up to 10 displaced atoms were observed in the ion images of the surfaces of tips which had been irradiated by deuterons of all energies. Usually they were located in the neighborhood of $\{112\}$ type faces, and they were observed so rarely under the fluxes used that it turned out to be impossible to reach any conclusions about how their concentrations and sizes depend on the energy of the bombarding particles.

In addition, dislocations were observed on the ion images of the samples irradiated by deuterons. The obtained images of unirradiated specimens in general did not indicate the presence of spiral edges of the planes, and only in rare cases was it possible to observe the edge of a half-plane. It was most often possible to observe dislocations on faces or in the neighborhood of a type $\{112\}$ face.

6.4. Ion Bombardment

Stayer et al.[83] carried out a preliminary investigation of the surface defects in tungsten, cleaned by field evaporation, associated with its bombardment by low-energy xenon ions (Xe^+). The energies of the ions were between 100 and 1300 eV, and the tips set up in the projector were irradiated without applying a strong electric field to them. In order to decrease contamination of the surface during irradiation, the projector was evacuated to an ultrahigh vacuum.

Hudson et al.[84] investigated the nature of the damages appearing in the crystal lattice of iridium upon its bombardment by Ar^+ ions with an energy of 100 keV. The ion fluxes used were in the range from 10^{11} to 10^{16} ions/cm^2, and irradiation was carried out at room and liquid nitrogen temperatures (78°K) directly inside the projector, under both high-vacuum conditions without the application of an electric field and under conditions corresponding to field ionization of the imaging gas.

In the case when irradiation was carried out at 78° K with low doses (of the order of 10^{11} ions/cm^2), the ion images only showed the formation of isolated single vacancies. Applying the technique of a three-dimensional model developed by the authors, they were able to ascertain that the observed single vacancies form regions of the type of dispersed clusters.

Upon irradiation of the samples by argon ions (60 keV) at room temperature using higher doses (integrated flux of 5×10^{13} ions/cm^2), the concentration of single vacancies turned out to be lower. The majority of the damage was present as vacancy clusters and as

FIG. 14. Helium ion micrograph of an iridium tip after its irradiation with 60 keV Ar^+ ions (integrated dose of radiation–5×10^{13} ions/cm^2) at room temperature. [84]

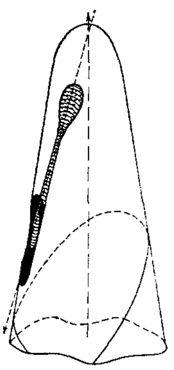

FIG. 15. A schematic representation of the track of the damage induced by the passage of a single fission fragment through a tungsten tip. [87]

dislocation loops (Fig. 14). A parallel analysis, carried out with an electron microscope, of iridium foils irradiated under the same conditions as the tips in the field-ion microscope showed that the density of "black spots" on the electron micrographs is approximately equal to the density of clusters plus the density of dislocation loops seen in the field ion micrographs.

6.5. Bombardment with Fission Fragments

Bowkett et al.[86, 87] investigated the damage in tungsten caused by the passage of fission fragments through its crystal lattice. Tips prepared beforehand were mounted in a special evacuated aluminum capsule at a distance of 6 mm from a small piece of enriched uranium foil (90% U^{235}) and were placed in the channel of a nuclear reactor. The use of rather thick uranium foil (its thickness appreciably exceeded the mean free path of the fission fragments in uranium) resulted in the energy spectrum of the fission fragments having a single peak near 30 MeV.[88] The total neutron flux on the samples amounted to 5×10^{14} neutrons/cm^2. The flux of fission fragments was monitored with the aid of the technique described in [89], and during irradiation it corresponded to the incidence of one fission fragment on each tip. The geometry of the irradiation was such that[87] the inclination of the fragments to the ([011]) axis of the samples amounted to $20 \mp 7°$.

The first ion images of specimens irradiated in this manner showed a rather normal structure with a dark region in the center, which indicated the presence of a surface depression. Field evaporation of several surface atom layers made it possible to observe a cavern of diameter around 50 Å below the surface. Further field evaporation of the tip exposed a light track of damage, and for the case of non-axial bombardment the fragment track emerges from the side surface of the tip, where removal of part of the material has occurred (see Fig. 15). In actual fact this would not occur in bulk material where atoms knocked off lattice sites must sit on interstitial sites, agglomerate, or effectively annihilate themselves on other lattice defects. A separate investigation of neutron irradiation of tungsten wire using comparable doses[66] showed that it was precisely the passage of fission fragments into the specimen material which was responsible for the formation of the large damages which were observed.

6.6. Irradiation by Electrons. Cathode Sputtering

The first observation of damage to a crystal lattice due to the action of electron bombardment was made by Müller[3] in a helium ion projector. Electrons with energies of the order of 1 MeV were obtained owing to ionization of gas atoms in the space above the tip and, being accelerated by the electric field, they bombarded the surface of the tungsten tip. As a result it was confirmed that electrons of such energies create single vacancies and interstitial atoms in the crystal lattice.

Garber et al.[90] carried out a field-ion microscope investigation of defects in tungsten, subjected to irradiation by electrons with an energy of 240 MeV. The integrated radiation flux amounted to 10^{16} electrons/cm^2, and the temperature of irradiation did not exceed 40°C. The ion images of the irradiated material showed the formation of single atoms displaced into interstitial positions (density equal to 5×10^{17} atoms/cm^3) and small vacancy clusters of diameter up to 10 Å.

One of the promising applications of the ion projector is its use to investigate the process of cathode sputtering of the surface of the needle-shaped samples. A simple version of an experiment on cathode sputtering is the bombardment of the surface of the tip by ions of the imaging gas directly in the projector. For this purpose, after preliminary purification of the surface of the tip by the field, the polarity of the high voltage field is changed, as a result of which the tip begins to function as a field emitter. The field-emission electrons accelerated in the direction of the fluorescent screen produce ionization of the imaging gas, and the resulting ions, being accelerated in the reverse field, bombard the tip. A similar technique was first used by Müller[1, 78] in order to sharpen the specimens in an ion projector by means of the removal of surface atom layers. The results obtained in [78] concerning cathode sputtering of tungsten by helium ions were in good agreement with the data known from other experiments. Apparently neon and other heavier gas ions will produce larger damages in the structure of the surface owing to more effective energy transfer.

Working with a helium ion projector, Waclawski and Müller[91] observed, during observation of its ion images, that the surface of the tip undergoes pronounced

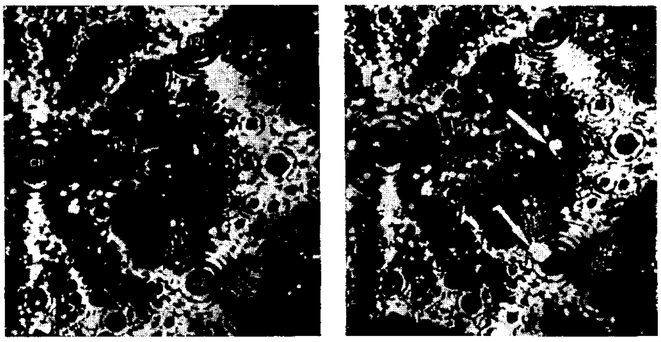

FIG. 16. Helium ion image of a tungsten surface cleaned by field evaporation after its bombardment by aluminum ions. The arrows indicate clusters of interstitial atoms on the surface. [91]

changes. Clusters of interstitial atoms and other damage to the crystal structure was observed (Fig. 16) resembling the effects of cathode sputtering. In this work a field ion microscope with a dynamic gas supply was used, for which purpose the sample was surrounded by a cylindrical accelerating electrode made of aluminum or stainless steel, at the bottom of which, opposite the emitter tip, there was a small aperture (of radius 0.15 mm).

The appearance of the observed damage on the surface of the tips was explained as being caused by the release of negative ions from the accelerating electrode as a result of its bombardment by the imaging gas ions. The negative ions formed in this way were accelerated toward the tip, gaining almost the energy corresponding to the applied high voltage. Approaching the tip these ions were neutralized by electron tunneling, after which their positive ionization occurred in the strong electric field. However, as a result of inertia the particles still continued to move toward the tip, bombarding its surface.

7. CONCLUSION

The utilization of the methods of the ion projector in order to investigate radiation defects in solids—this is an extremely new direction in field-ion microscopy. Nevertheless, it is not an exaggeration to say that the information about defects in crystalline structure, their nature and distribution, which this direction may be able to provide and which it has already partially provided, enable one to reach a whole series of interesting conclusions about the nature of the phenomena taking place in solids upon irradiation, and, perhaps, it forces us to take another look at certain theoretical ideas which are accepted at the present time.

In addition, the field-ion microscope study of the processes of recovery of the proper structure in irradiated samples during different stages of annealing may be of practical value in regard to improving the quality and properties of construction materials.

[1] Direct Observation of Imperfections in Crystals, edited by J. B. Newkirk and J. H. Wernick, Interscience Publishers, 1962 (Russ. Transl., Metallurgizdat, M. 1964).

[2] E. W. Müller, Z. Physik 131, 136 (1951).

[3] E. W. Müller, Adv. in Electronics and Electron Physics 13, 83 (1960).

[4] H. G. van Bueren, Imperfections in Crystals, North-Holland Publishing Co., 1960 (Russ. Transl., IL, M, 1962).

[5] A. C. Damask and G. J. Dienes, Point Defects in Metals, Gordon and Breach, 1964 (Russ. Transl., Mir, M. 1966).

[6] J. R. Oppenheimer, Phys. Rev. 31, 66 (1928).

[7] Erwin W. Müller, Phys. Rev. 102, 618 (1956).

[8] D. G. Brandon, Phil. Mag. 14, 803 (1966).

[9] Erwin W. Müller, Z. Physik 156, 399 (1959).

[10] E. W. Müller, International Conference on Vacancies and Interstitials in Metals (Jülich, September 23, 1968). Preprint, Vol. 2, Jülich (1968), p. 482.

[11] B. Ralph, in Field-Ion Microscopy, edited by John J. Hren and S. Ranganathan, Plenum Press, 1968, pp. 157–182.

[12] M. Attardo and J. M. Galligan, Phys. Stat. Sol. 16, 449 (1966).

[13] D. G. Brandon, M. Wald, M. J. Southon, and B. Ralph, J. Phys. Soc. Japan 18, Supplement 2, 324 (1963).

[14] M. K. Sinha and E. W. Müller, J. Appl. Phys. 35, 1256 (1964).

[15] E. W. Müller, J. Phys. Soc. Japan 18, Supplement 2, 1 (1963).

[16] M. J. Attardo and J. M. Galligan, Phys. Rev. Lett. 14, 641 (1965).

[17] M. J. Attardo and J. M. Galligan, Phys. Rev. 161, 558 (1967).

[18] A. L. Suvorov and G. M. Kukavadze, FMM 28, 238 (1969).

[19] M. J. Attardo, Phys. Lett. 25A, 184 (1967).

[20] A. L. Suvorov and V. A. Kuznetsov, FMM 27, 566 (1969).

[21] V. A. Kuznetsov, G. M. Kukavadze, and A. L. Suvorov, v sb. "Renii v novoi tekhnike" (in the collection: Rhenium in the New Technology), Nauka, M. 1969.

[22] Erwin W. Müller, John A. Panitz, and S. Brooks McLane, Rev. Sci. Instr. 39, 83 (1968).

[23] E. W. Müller, Science 149, 591 (1965) (Russian Transl. in Usp. Fiz. Nauk 92, 293 (1967)).

[24] E. W. Müller, Surface Science 2, 484 (1964).

[25] W. T. Read, Dislocations in Crystals, McGraw-Hill, 1953 (Russ. Transl., Metallurgizdat, M. 1957).

[26] A. H. Cottrell, Dislocations and Plastic Flow in Crystals, Oxford University Press, 1953 (Russ. Transl., Metallurgizdat, M. 1958).

[27] J. Friedel, Dislocations, Addison-Wesley, 1964 (Russ. Transl., Mir, M. 1967).

[28] D. W. Pashley, Reports on Progress in Physics 28, 291 (1965).

[29] S. Ranganathan, J. Appl. Phys. 37, 4346 (1966).

[30] D. A. Smith, M. A. Fortes, A. Kelly, and B. Ralph, Phil. Mag. 17, 1065 (1968).

[31] S. Ranganathan, in Field-Ion Microscopy, edited by John J. Hren and S. Ranganathan, Plenum Press, New York, 1968, pp. 120–156.

[32] M. A. Fortes, D. A. Smith, and B. Ralph, Phil. Mag. 17, 169 (1968).

[33] M. A. Fortes and B. Ralph, Phil. Mag. 19, 181 (1969).

[34] J. J. Hren, in Field-Ion Microscopy, edited by John

J. Hren and S. Ranganathan, Plenum Press, New York, 1968, pp. 102–119.
[35] M. A. Fortes and B. Ralph, Phil. Mag. **14**, 189 (1966).
[36] M. I. Mikhaĭlovskiĭ, Candidate's dissertation, Kiev, 1969.
[37] R. I. Garber and A. I. Fedorenko, Usp. Fiz. Nauk **83**, 385 (1964).[Sov. Phys.-Uspekhi **7**, 479 (1965)].
[38] R. H. Silsbee, J. Appl. Phys. **28**, 1246 (1957).
[39] G. M. Kukavadze, A. L. Suvorov, and B. V. Sharov, Fiz. Metal. Metalloved. **27**, 797 (1969).
[40] S. T. Konobeevskiĭ, Deĭstvie ioniziruyushchikh izlucheniĭ na metally i splavy (The Effect of Ionizing Radiation on Metals and Alloys), Atomizdat, M. 1967.
[41] R. I. Garber, I. M. Mikhaĭlovskiĭ, Zh. I. Dranova, and V. I. Afanas'ev, Fiz. metal. metalloved. **23**, 345 (1967) [The Physics of Metals and Metallography **23**, No. 2, 161 (1967)].
[42] R. I. Garber, D. I. Dranova, and I. M. Mikhaĭlovskiĭ, Zh. Eksp. Teor. Fiz. **54**, 714 (1968) [Sov. Phys.-JETP **27**, 381 (1968)].
[43] H. F. Ryan and J. Suiter, J. Less-Common Metals **9**, 258 (1965).
[44] R. I. Garber, Zh. I. Dranova, and I. M. Mikhaĭlovskiĭ, Fiz. Tverd. Tela **10**, 1012 (1968) [Sov. Phys.-Solid State **10**, 799 (1968)].
[45] Hans A. Bethe and Julius Ashkin, in Experimental Nuclear Physics, edited by E. Segrè, John Wiley & Sons, Inc., 1953, Vol. I, pp. 166–357 (Russ. Transl., IL, M. 1955, p. 241).
[46] Niels Bohr, The Penetration of Atomic Particles Through Matter, Det Kgl. Danske Videnskabernes Selskab. Mathematisk-fysiske Meddelelser **XVIII**, No. 8 (1948) (Russ. Transl., IL, M. 1950).
[47] G. J. Dienes and G. H. Vineyard, Radiation Effects in Solids, Interscience Publishers, 1957 (Russ. Transl., IL, M. 1960).
[48] G. Kinchin and R. Piz, Usp. Fiz. Nauk **60**, 590 (1956).
[49] J. Glen, Usp. Fiz. Nauk **60**, 445 (1956).
[50] J. Slater, Usp. Fiz. Nauk **47**, 51 (1952).
[51] R. Sternheimer, in the collection: Principles and Methods of Detection of Elementary Particles (Russ. Transl., IL, M. 1963, p. 9).
[52] A. M. Shalaev, Deĭstvie ioniziruyushchikh izlucheniĭ na metally i splavy (The Effect of Ionizing Radiation on Metals and Alloys), Atomizdat, M. 1967.
[53] Robert Gomer and Lynn. W. Swanson, J. Chem. Phys. **38**, 1613 (1963).
[54] E. W. Müller, in Field-Ion Microscopy, edited by John J. Hren and S. Ranganathan, Plenum Press, New York, 1968, pp. 88–101.
[55] N. D. Morgulis, Usp. Fiz. Nauk **28**, 202 (1946).
[56] E. W. Müller, Umschau **57**, 579 (1957).
[57] Gert Erlich and F. G. Hudda, J. Chem. Phys. **44**, 1039 (1966).
[58] A. L. Suvorov and G. M. Kukavadze, Fiz. Metal. Metalloved. **27**, 347 (1969).
[59] V. A. Kuznetsov, G. M. Kukavadze, B. M. Stasevich, and A. L. Suvorov, Atomnaya énergiya **26**, 22 (1969).
[60] Pierre Petroff and Jack Washburn, Rev. Sci. Instr. **39**, 317 (1968).
[61] P. Petroff and J. Washburn, phys. stat. sol. **32**, 427 (1969).
[62] A. L. Suvorov and G. M. Kukavadze, Fiz. Metal. Metalloved. **27**, 72 (1969) [The Physics of Metals and Metallography **27**, No. 1, 76 (1969)].
[63] E. Sugata, H. Kim, K. Kasagi, K. Izeki, and S. Kobiyama, in Proceedings of the Sixth International Congress for Electron Microscopy, Kyoto (1966), edited by Ryozi Uyeda, Maruzen Co., Ltd., 1966, Vol. 1, p. 247.
[64] D. G. Brandon, in Field-Ion Microscopy, edited by John J. Hren and S. Ranganathan, Plenum Press, New York, 1968, pp. 53–68.
[65] A. L. Suvorov and G. M. Kukavadze, Fiz. Metal. Metalloved. **27**, 345 (1969).
[66] K. M. Bowkett, J. Hren, and B. Ralph, in Proceedings of the Fifth European Conference on Electron Spectroscopy, Vol. A, Prague, 1965, p. 191.
[67] M. J. Attardo and J. M. Galligan, J. Appl. Phys. **38**, 418 (1967).
[68] M. Wald, Ph.D. Thesis, Cambridge University, 1963.
[69] K. M. Bowkett, Ph.D. Thesis, Cambridge University, 1966.
[70] V. I. Kirienko and L. P. Potapov, Dokl. Akad. Nauk SSSR **186**, 1309 (1969) [Sov. Phys.-Doklady **14**, 607 (1969)].
[71] M. J. Attardo and J. M. Galligan, Phys. Rev. Lett. **17**, 191 (1966).
[72] B. Ralph, in Field-Ion Microscopy, edited by John J. Hren and S. Ranganathan, Plenum Press, New York, 1968, p. 171.
[73] G. Leibfried, in The Interaction of Radiation with Solids, edited by R. Strumane, J. Nihoul, R. Gevers, and S. Amelinckx, North-Holland Publishing Co., Amsterdam, 1964.
[74] G. R. Piercy, Phil. Mag. **5**, 201 (1960).
[75] A. K. Seeger, in Proceedings of the Second United Nations International Conference on Peaceful Uses of Atomic Energy (United Nations, Geneva, Switzerland, 1958), Vol. 6, p. 250.
[76] V. A. Kuznetsov, G. M. Kukavadze, B. M. Stasevich, and A. L. Suvorov, v sb. "Monokristally tugoplavkikh i redkikh metallov" (in the collection: Single Crystals of Refractory and Rare Metals), No. 1, Nauka, M. 1969, p. 71.
[77] D. G. Brandon and M. Wald, Phil. Mag. **6**, 1035 (1961).
[78] E. W. Müller, in Proceedings of the Fourth International Symposium on the Reactivity of Solids (1960), Elsevier Publishing Co., Amsterdam, 1960, p. 862.
[79] D. G. Brandon, M. J. Southon, and M. Wald, Properties of Reactor Materials and the Effects of Radiation Damage, Butterworths, London, 1962, p. 113.
[80] D. G. Brandon and M. Wald, Discussions Faraday Soc. **31**, 73 (1961).
[81] A. H. Cottrell, Mem. Sci. Rev. Metallurge **63**, 237 (1966).
[82] A. L. Suvorov, G. M. Kukavadze, and A. F. Bobkov, Zh. Eksp. Teor. Fiz. **58**, 85 (1970) [Sov. Phys.-JETP **31**, 47 (1970)].
[83] R. W. Stayer, E. C. Cooper, and L. W. Swanson, Proceedings of the 12th Field-Emission Symposium, Pennsylvania State University, University Park, Pa., 1963.
[84] J. A. Hudson, R. S. Nelson, and B. Ralph, Phil. Mag. **18**, 839 (1968).
[85] John A. Brinkman, J. Appl. Phys. **25**, 961 (1954).

[86] K. M. Bowkett, L. T. Chadderton, H. Norden, and B. Ralph, Phil. Mag. **11**, 651 (1965).
[87] K. M. Bowkett, L. T. Chadderton, H. Norden, and B. Ralph, Phil. Mag. **15**, 415 (1967).
[88] R. F. Redmond, R. W. Klingensmith, and J. N. Anno, J. Appl. Phys. **33**, 3383 (1962).
[89] F. P. Bowden and L. T. Chadderton, Proc. Roy. Soc. (London) **A269**, 143 (1962).
[90] R. I. Garber, Zh. I. Dranova, I. M. Mikhaĭlovskiĭ, and V. A. Stratienko, Zh. Eksp. Teor. Fiz. **54**, 1025 (1968). [Sov. Phys.-JETP 27, 545 (1968)].
[91] B. J. Waclawski and E. W. Müller, J. Appl. Phys. **32**, 1472 (1961).

Translated by H. H. Nickle