NEW INSTRUMENTS AND MEASUREMENT METHODS

Use of high-voltage electron microscopy in solid-state physics

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An analysis is made of the use of high-voltage electron microscopes in solid-state physics research. The most promising fields of application of high-voltage electron microscopes are discussed; these involve mainly research on the mechanism of the action of radiation on solids.

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Transmission electron microscopy occupies one of the leading positions in research on solid-state physics and material science. In addition to the traditional structural studies, this technique finds application also in new fields such as, in particular, study of the domain structure of thin magnetic films, and the electromagnetic properties of superconducting materials.[1]

Many of the advances in solid-state physics research in recent years have been due to use of electron microscopes with accelerating voltages up to 100 kV. However, to obtain images of sufficiently high quality in use of these microscopes, extremely thin objects are required, and in the preparation of these objects the structure can be changed substantially by the action of surface forces.

To improve image quality and avoid undesirable surface effects, there has been a tendency in recent years to use electron microscopes with accelerating voltages above 100 kV. At the present time instruments with accelerating voltage up to 3 MV are rather widely used in Japan, France, England, and the USA. Development of microscopes with accelerating voltage up to 5 MV is being carried on at the Argonne National Laboratory, USA,[1] and up to 10 MV by the Japanese firm JEOL.[2]

Increasing the accelerating voltage leads to an improvement in the quality of the electron microscope image. This is due to the decrease in the electron wavelength and the decrease in the energy loss per unit path length. The change in these parameters of the electrons leads to an increase in resolution, a decrease in chromatic aberration, an increase in image contrast, and a decrease in heating of the objects. A number of other image parameters are also improved, and in addition the techniques of preparing and scanning objects are simplified. The possibilities of direct study of processes due to passage of electrons through solids[3,4] are greatly extended.

An increase in accelerating voltage leads not only to improvement of the electron microscope image. At certain voltages a qualitatively new effect appears–interaction of the electron beam with the crystal lattice–production of radiation-induced defects. In Fig. 1 we have shown the energy dependence of the cross sections for production of radiation defects in electron bombardment for a number of metals.[5] The intersection of the function $\sigma_d(E)$ with the abscissa in Fig. 1 gives the value of the threshold electron energy necessary for formation of radiation-induced defects in the given materials. The value of the threshold electron energy (E_t) can be estimated from the equation

$$E_d = \frac{2E_t(E_t + 2m_0c^2)}{Mc^2}, \qquad (1)$$

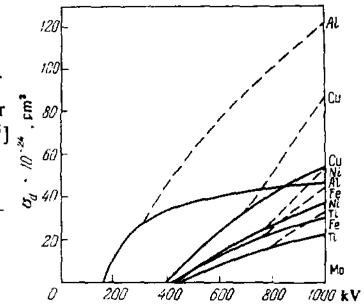

FIG. 1. Energy dependence of cross sections for displacement for electrons in a number of metals.[5] Solid curves–without taking into account the possibility of secondary displacements; dashed curves–with inclusion of the possibility of secondary displacements.

TABLE I

Element	Threshold displacement energy E_d, eV	Threshold energy of electrons E_t, keV	Element	Threshold displacement energy E_d, eV	Threshold energy of electrons E_t, keV
Si	13[6]	150	Fe	24[9]	430
Al	16[7]	170	Ni	24[9]	440
Zn	13.5[8]	310	Mo	30[10]	740
Ge	15[6]	370	Ta	32[11]	1200
Cu	19[7]	390	W	35[7]	1300
Ti	29[9]	430			

where E_d is the threshold energy for displacement of an atom of mass M, m_0 is the electron rest mass, and c is the velocity of light.

In Table I we have given values of E_t for a number of elements, calculated from Eq. (1). It can be seen from the table that, except for Ta and W, study of most materials in electron microscopes with accelerating voltage up to 1 MV is accompanied by formation of radiation defects, and for light elements such as Si and Al, radiation defects are produced at voltages less than 200 kV, which are attained in many microscopes extensively used at the present time.

The electron current density in contemporary electron microscopes operating under normal conditions[12] amounts to 1–10 A/cm², which corresponds to a bombardment intensity of 6×10^{18}–6×10^{19} cm^{-2} sec^{-1}. At these bombardment intensities, the rate of introduction of radiation defects for electron energies of 1 MeV for most of the materials appearing in Table I will amount to 10^{-3}–10^{-2} atomic displacements per second, which is 4–5 orders of magnitude greater than the rate of production of defects achieved ordinarily in experiments on bombardment of materials in electron accelerators with energies of several MeV.

As the electron energy is decreased to a value near threshold, the rate of appearance of radiation defects, although it decreases as the result of decrease in the displacement cross section $\sigma_d(E)$, will amount to a rather large quantity even near threshold.

Let us estimate up to what temperatures radiation effects will be dominant in study of materials in a high-voltage electron microscope. This will obviously be determined by the relation between the thermodynamic equilibrium concentration of point defects and the concentration of defects resulting from bombardment in the high-voltage microscope.

The thermodynamic equilibrium concentration of vacancies can be calculated from the equation

$$c_V = \exp\left(\frac{\Delta S_F^V}{k}\right)\exp\left(-\frac{E_F}{kT}\right), \qquad (2)$$

where ΔS_F^V is the change in entropy in formation of a vacancy, E_F is the vacancy formation energy, k is Boltzmann's constant, and T is the absolute temperature.

Since the rates of appearance of radiation defects in a high-voltage microscope are very high, we can assume that the annealing of radiation vacancies is determined mainly by the mechanism of mutual recombination of defects. For dynamic equilibrium[13]

$$c_V = \sqrt{\frac{k}{\nu_i}}; \qquad (3)$$

$\nu_i = \nu_0 \exp(\Delta S_m^i/k)\exp(E_m^i/kT)$ is the frequency of migration of an interstitial atom, ν_0 is the atomic vibration frequency, ΔS_m^i is the change of entropy on migration of an interstitial atom, and E_m^i is the energy of motion of the interstitial atom.

In Fig. 2 we have shown vacancy concentrations calculated from Eqs. 2 and 3 for copper. The dependence of c_V on bombardment corresponds to a rate of production of uncorrelated defects of 3×10^{-4} cm^{-2} sec^{-1} for a bombardment intensity of 5×10^{19} cm^{-2} sec^{-1} and an electron energy of 1 MeV. It can be seen that up to a temperature 0.65 T_{mp} (the point of intersection of the straight lines on the plot) the concentration of radiation defects will substantially exceed the thermodynamic equilibrium value (T_{mp} is the melting point in °K). At a temperature of 770° K this excess amounts to 1.5 orders of magnitude, and at room temperature—more than 10 orders of magnitude.

It should be noted that in experiments on irradiation of solids in electron accelerators the concentration of radiation defects exceeds the thermodynamic equilibrium concentration of defects up to a temperature no more than 0.4 T_{mp}.

Thus, in electron-microscope studies of solids under conditions in which the accelerating voltage exceeds the threshold value, a very high concentration of radiation defects is produced in the objects studied, over a wide range of temperature. It is evident that under these conditions it is not the original structure of the material which is being studied, but the result of interaction of this structure with radiation defects and its change on occurrence of radiation-stimulated processes (such as radiation enhancement of diffusion).

Therefore in carrying out nonradiation electron-microscope studies it is necessary to use an accelerating voltage below the corresponding threshold values for a given material. However, in this case the advantages of high-voltage electron microscopy and, first of all, the advantages of examination of massive objects are lost to a significant degree. The other method, involving reduction of the beam intensity, is not effective, since the loss in image quality considerably exceeds the modest gain due to reduction of radiation damage.

Even before the advent of high-voltage electron microscopes, transmission electron microscopy was extensively used (and is used at the present time) for investigation of radiation damage and phase transformations associated with radiation-stimulated processes. In this capacity it served only as a method of determining the preliminary radiation effect or the after-effect due to annealing of radiation damage. High-voltage electron microscopy combines simultaneously the production of the effect and its study, which opens new and unique possibilities of its use in radiation physics of the solid state. In radiation studies, in contrast to nonradiation studies, all of the advantages in the quality of the electron microscope image provided by the high accelerating voltage can be utilized practically completely.

One of the most important spheres of application of high-voltage electron microscopy involves the possibility of modeling the action of heavy charged particles and neutrons on solids.

It is well known that the specific features of radiation damage of solids on bombardment by heavy particles and in particular by neutrons consist of the formation, in addition to point defects, of clusters of radiation defects, dislocation loops, and voids. At low electron flux densities in ordinary accelerators with energies of several MeV, it has not been possible until recently to obtain in solids similar defects visible in an electron microscope. Only recently a number of studies have appeared in which clusters of radiation defects have been observed in certain metals (Al[14], Au[15], Ag[16], Ni[17]) after bombardment with high intensity in electron accelerators. Figures 3 and 4 show clusters of radiation defects observed in Al and Ag.

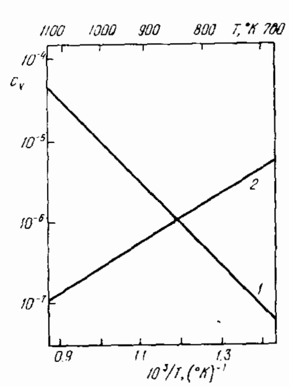

FIG. 2. Temperature dependence of equilibrium concentration of vacancies in Cu (1) and of the dynamic equilibrium concentration of vacancies in Cu in bombardment by electrons with energy 1 MeV and intensity 5×10^{19} cm^{-2} sec^{-1} (2).

FIG. 3. Clusters of radiation defects in Al bombarded at room temperature by 2.3-MeV electrons. [14] The radiation dose is 7.2×10^{18} cm^{-2}.

FIG. 4. Clusters of radiation defects in Ag bombarded (and studied) at a temperature <250° K with 3.0-MeV electrons. [15] The radiation dose is 6.7×10^{18} cm^{-2}.

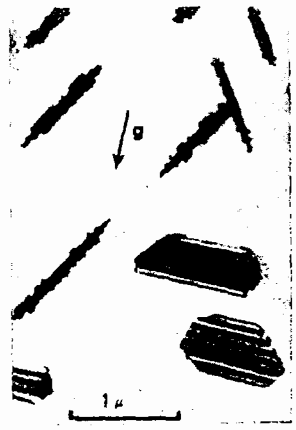

FIG. 5

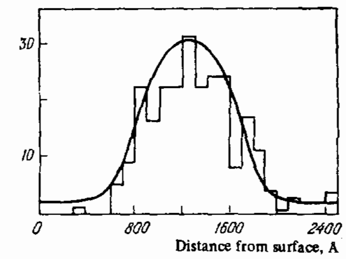

FIG. 6

FIG. 5. Dislocation loops in Cu bombarded in a high-voltage microscope by 600-keV electrons. [18]

FIG. 6. Distribution of dislocation loops in a Cu foil of thickness 2500 Å bombarded in a high-voltage microscope with 600-keV electrons. [18]

However, in carrying out similar investigations with use of accelerators and microscopes with low accelerating voltage, a large loss of radiation defects occurs either through irradiation or through subsequent thinning of the objects. As has been shown in a number of studies, the surface layer in which practically no defects are observed, depending on the material, may amount to more than several hundred angstroms (see for example ref. 18). It is obvious that observation of clusters of radiation defects by means of ordinary electron microscopes is an extremely complex problem.

In use of high-voltage electron microscopes, the conditions for origin, growth, and subsequent study of clusters of radiation defects are immeasurably better. First of all, high bombardment intensities create in a very short space of time clusters and dislocation loops of rather large size. In this case even a very high concentration of impurities–traps for radiation defects–cannot completely suppress the production of clusters, since under conditions of high concentrations of radiation defects, clusters can originate simultaneously at impurities and on encountering defects. Furthermore, under these conditions, by varying the intensity of bombardment or concentration of impurities, it is possible to follow the dominant effect of various cluster formation mechanisms. Very recently, with use of high-voltage microscopes and accelerators, a number of studies have been made in which it has been shown that the role of impurities is so great that even an insignificant change in their concentration has an important effect on the processes of initiation and growth of clusters. Thus, for Au of purity 99.9999% the concentration of clusters after bombardment at 130° K by 3-MeV electrons with doses $> 10^{18}$ cm^{-2} amounts to 8×10^{14} cm^{-3}, and for Au of purity 99.99%–it amounts to[15] 2×10^{16} cm^{-3}. For equal bombardment doses the average size of clusters in gold of purity 99.9999% was higher than in Au of purity 99.999%. Similar results[19] have been obtained for Ni.

The advantages of carrying out such studies directly in high-voltage electron microscopes in comparison even with high-current accelerators are indisputable, since there is no need to prevent changes in the structure in preparation and mounting of the bombarded object for study.

The possibility of examining massive objects permits not only avoiding to a substantial degree undesirable surface effects but also study of the distribution of radiation defects as a function of depth over a rather wide range. In Figs. 5 and 6 we have shown radiation dislocation loops and their distribution in Cu bombarded in a high-voltage electron microscope by 600-keV electrons.[18] As can be seen, in this case the depth of the denuded layer where practically no defects are observed amounts to ~600 Å. When the depth of the denuded layer is comparable with the sample thickness, the kinetics of cluster growth, as shown by Norris,[20] is completely determined by the departure of radiation defects to the surface. Thus, only in samples of thickness substantially greater than the depth of the denuded layer is it possible to model satisfactorily processes of cluster formation in massive crystals.

We can point out a number of promising directions which are made possible by use of high-voltage electron microscopes for production and study of radiation defect clusters and dislocation loops. Among these directions are the study of the spatial distribution of radiation defects along the path of heavy particles accelerated directly in the column of the electron microscope, study of the combined action of electrons and other particles, study of the effectiveness of various types of drainage, investigation of the geometry and nature of clusters and dislocation loops. In the last case definite progress has already been made.

Production of clusters and dislocation loops in bombardment in a high-voltage electron microscope is only one of the aspects of modeling radiation damage in solids under the action of heavy particles and neutrons. The specific behavior of the change of properties on bombardment by heavy particles is determined in many respects also by the formation of voids, which in fact turns out to have a dominant influence on the efficiency of materials used in reactor construction and a number of other fields of technology.

The formation of voids in electron bombardment in high-voltage electron microscopes was observed for the first time in Ni containing argon impurity introduced by previous ion bombardment (Fig. 7).[21, 22] Subsequently it was established that special introduction of gas impurities is not a necessary condition for void formation in bombardment in high-voltage electron microscopes. The gas concentration necessary for stabilization of void nuclei amounts to $< 10^{-8}$ according to some estimates[5] and can be present completely in the form of natural

FIG. 7

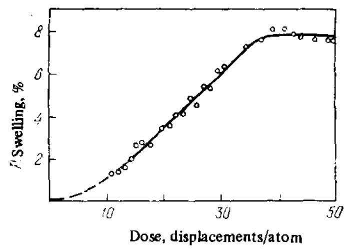

FIG. 8

FIG. 7. Dislocations and voids observed in Ni with argon impurity on bombardment by electrons in a high-voltage microscope. [22]

FIG. 8. Typical dependence of swelling of Ni on bombardment in a high-voltage electron microscope. [24] The rate of atomic displacements is 2 × 10^{-3} sec^{-1}, and the temperature is 450° C.

TABLE II

Element	⟨hkl⟩	E_d, eV	Element	⟨hkl⟩	E_d, eV
Al		16 [28]	Co (hex.)	⟨11$\bar{2}$0⟩	23±0.5 [30]
Au	⟨100⟩	33 [29]	Co (hex.)	⟨1010⟩	30±1.0 [30]
Ni	⟨100⟩	31±1.5 [19]	Co (hex.)	⟨0001⟩	33±1.0 [30]
Ni	⟨110⟩	23±2 [19]	Cu	⟨100⟩	21.6 [31]
Ni	⟨111⟩	28±1.5 [19]	Cu	⟨110⟩	19.2 [31]
Co (FCC)	⟨110⟩	23±0.5 [30]	Cu	⟨110⟩	23.6 [31]
Co (FCC)	⟨100⟩	30±1.0 [30]			

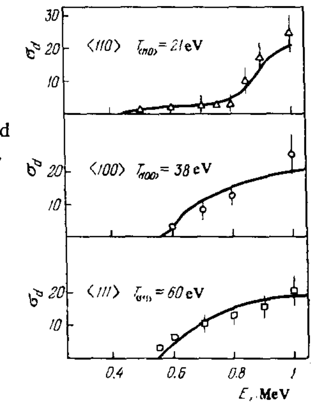

FIG. 9. Theoretical (solid lines) and experimental dependences of the cross sections for displacement for various crystallographic directions in Ni, obtained by high-voltage electron microscopy. [19]

impurities, especially in industrial materials, as has been confirmed in bombardment of stainless steels.[23]

Figure 8 shows a typical dependence of the change in volume (swelling) of nickel as a result of void formation on bombardment in a high-voltage electron microscope with a rate of atomic displacement 2 × 10^{-3} sec^{-1} at a temperature of 450° C.[24]

Except for a small initial period which is associated with the effect of the surface such as drainage for radiation defects, the kinetics of swelling, both for Ni and for industrial reactor materials, corresponds practically completely to the kinetics of swelling of these materials under neutron bombardment.

At the present time a large group of studies have been carried out on the effect of bombardment temperature, surface, dose, impurities, and dislocation structure on the process of void formation in high-voltage electron microscopes.[23, 25, 26] As a result of these studies, more useful information on the void production mechanisms has in fact been obtained than was accumulated during all of the preceding years when information was obtained for the most part only from reactor experiments.

By means of high-voltage electron microscopes it has been possible to show, in particular, that the process of void growth is closely related to the density of dislocations, the change in the dislocation structure on bombardment, and the interaction of dislocations with growing voids. It has been established that the origin and growth of voids in bombardment is observed, as a rule, near dislocations. Dislocations absorb interstitial atoms and enrich the zone near the void with vacancies, thereby facilitating its growth. The proof that the void production mechanism due to the existence of displacement cascades is not dominant, as was previously supposed, is to be considered an important result of these studies.

The possibility of producing resolvable defects directly in bombardment in an electron microscope permits use of high-voltage electron microscopy for determination of threshold displacement energies and for study of the energy dependence of the displacement cross sections.

The method of measuring electrical resistivity is usually used in the radiation physics of solids to determine these quantities in electron bombardment. In measurement of the effective values of E_d and $\sigma_d(E)$ in polycrystalline samples, the electrical-resistivity method permits quite reliable results to be obtained. However, in determination of these quantities for various orientations of single crystals, the reliability of the results obtained is greatly reduced. This is due first of all to the fact that determination of E_d and $\sigma_d(E)$ by the measurement of electrical resistivity is carried out in rather thick samples (10–50 μ), in which the divergence of the primary beam as a result of multiple scattering of electrons is quite large. Thus, for Cu of thickness 12 μ the mean square divergence of an electron beam with energy close to the threshold amounts to[27] 27°. The angles between the ⟨100⟩ and ⟨110⟩, ⟨111⟩and ⟨110⟩, ⟨111⟩ and ⟨100⟩ directions in a face-centered-cubic lattice are respectively 45, 35.3, and 54.7°. Therefore displacements arising as the result of multiple scattering in other directions smear the orientation effects to a significant degree. This explains to a large extent the practically equal values of E_d determined by electrical-resistivity measurement for different directions in single crystals.

In measurement of E_d and $\sigma_d(E)$ in a high-voltage electron microscope it is possible to use samples hundreds of times thinner, which substantially decreases the errors due to multiple scattering.

In Table II we have given values of the threshold displacement energies for a number of metals, measured with electron microscopy, and in Fig. 9 we have shown the energy dependence of the displacement cross sections for the ⟨100⟩, ⟨110⟩, and ⟨111⟩ directions in Ni, obtained also by high-voltage electron microscopy.[19]

Practically all determinations of E_d and $\sigma_d(E)$ by means of high-voltage electron microscopy have been carried out at temperatures where the radiation defects are mobile and can form clusters. Therefore the values of E_d and $\sigma_d(E)$ measured in these experiments, in contrast to electrical-resistivity measurements, refer not to formation of close Frenkel pairs but to the appearance of mobile defects. Recently, however, studies have appeared in which high-voltage electron microscopy is used also to study low-temperature radiation damage. Thus, in Ni, clusters of radiation defects have been observed after bombardment in a high-voltage electron microscope[32,33] at 8°K. Clusters of radiation defects cannot be formed by means of diffusion at 8°K. Therefore their appearance can be the result of defocusing of a sequence of collisions either in impurities or in immobile interstitial atoms. It is also possible that these clusters arose as the result of interaction of the fields of identical defects.

The promise of further work in this field is beyond doubt, since it becomes possible to investigate both the mechanisms of atomic displacements and the specific features of the structure of low-temperature clusters. Other methods of study can hardly compete seriously with high-voltage electron microscopy in this region.

Study of the kinetics of the diffusion growth of clusters, dislocation loops, and voids on bombardment of annealed materials directly in a high-voltage electron microscope permits determination of the diffusion parameters of radiation point defects. Similar information can also be obtained by using samples in which defects of the vacancy type (tetrahedral packing defects, vacancy clusters, and dislocation loops) have been introduced beforehand. On bombardment of such objects in the range of temperatures where diffusion fluxes of interstitial atoms to vacancy drainages will substantially exceed the flux of vacancies, the size and concentration of the defects introduced beforehand will be decreased as the result of annihilation of mobile interstitial atoms in them. An example of investigations of this type is a study[34] of the kinetics of annealing of tetrahedral packing defects in gold on bombardment by argon ions accelerated in the column of a transmission electron microscope in the temperature range from −130 to +70° C. From the data of this study the activation energy for motion of interstitial atoms in Au was found to be 0.75 eV.

Transmission electron microscopy can also be used successfully for study of the annealing of radiation, deformation, and quenching defects. A number of studies of this type have already been carried out for Au, Ag, Cu, and Al, where the kinetics of annealing have been studied both for radiation interstitial loops and for voids produced by previous heating. Values of the activation energy (1.75 eV for Au,[35] 1.85 eV for Ag,[35] 2.05 eV for Cu,[35,36] and 1.31 eV for Al[37]) and the temperature dependence of the self-diffusion coefficients are in good agreement with similar data obtained by the tracer method.

The possibility of obtaining very high concentrations of radiation point defects in a high-voltage electron microscope permits radiation-stimulated processes to be studied over a rather wide range of temperature. In fact, whereas these studies were limited to temperatures up to 0.4 T_{mp} when electron accelerators and reactors were used, the use of high-voltage electron microscopes provides the possibility of extending the temperature range where radiation-stimulated processes will be dominant up to 0.65 T_{mp}. At the present time studies have been made on the intensification of the decay of supersaturated solid solutions[38,39] based on Si and on recrystallization in Cu and stainless steels.[40]

Particularly promising in this, in our opinion, are studies utilizing high-voltage electron microscopes to obtain and investigate phases which because of low thermal diffusion coefficients, are not formed under conditions of thermodynamic equilibrium. Such cases are already known in the radiation physics of solids–here we have in mind the appearance of an ordered phase of the type AuCu after irradiation of Fe-Ni alloy, as the result of radiation enhancement of diffusion.[41]

FIG. 10. Dislocation structure of Al deformed by stretching, bombarded by electrons with a dose of 7.2×10^{17} cm^{-2}.

While radiation-enhanced diffusion facilitates the approach of irradiated systems to thermodynamic equilibrium, sequences of substitutional collisions lead to the reverse effect–disordering of the initial structure. Use of high-voltage electron microscopes can provide useful information on the dynamics of radiation damage also in this area. Such studies are already being carried on as part of the study of the orientation dependence of disordering of ordered alloys on bombardment in a high-voltage electron microscope.[42]

Major possibilities are opened up for the use of high-voltage electron microscopy in study of the interaction of radiation defects with the dislocation structure. As shown by us and in a number of other studies (see for example ref. 43), the dislocation structure undergoes appreciable changes even in the early stages of irradiation, where clusters of radiation defects are not yet observed. The characteristic features of this structure consist of the appearance of a large number of jogs and bends in the dislocation lines (Fig. 10). With increase of the radiation dose the initial dislocation structure may undergo substantial changes as the result of nonconservative motion under conditions of high supersaturation with point defects. In the motion the dislocations interact with each other and with clusters of radiation defects and disappear at the crystal surface.

The possibility of applying mechanical stresses to a sample in the process of study makes the use of high-voltage electron microscopes promising for investigation of such practically important phenomena as radiation creep and stress relaxation. In this case it is possible to analyze the mechanisms of these phenomena more correctly from the change in the dislocation structure. In addition, such investigations permit practically complete modeling of the conditions of operation of materials in nuclear energy plants.

At the present time extensive investigations are being carried on in connection with the development of radiation-stable materials. The role of high-voltage electron microscopes, which in a short period of time can produce high concentrations of radiation defects in materials while simultaneously studying their behavior, cannot be overestimated, particularly if the promise of use of fast neutron reactors is taken into account. It is no accident that at the present time a significant fraction of the studies carried out with high-voltage electron microscopes are associated with just this problem.[44] The advantages of modeling radiation damage by means of high-voltage electron microscopes in comparison with direct reactor experiments are due to their immediate nature, ready interpretation, and economy. Basic problems in such modeling still remain: the problem of the correct calculation of the equivalent number of displacements in electron bombardment and bombardment by heavy particles, and the possibility of reliable correspondence of the results obtained in thin and massive crystals. The second problem, obviously, is not critical because of the prospects for increasing the accelerating voltage of high-voltage electron microscopes.

The analysis which we have made of the use of high-voltage electron microscopes in solid-state physics shows that they are, first of all, an extraordinarily effective tool for study of the mechanism of radiation effects. Their use for ordinary structural studies, when the accompanying radiation damage is taken into account, is extremely limited and consequently ineffective.

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