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Standard Practice for
Investigating the Effects of Neutron Radiation Damage Using Charged-Particle Irradiation[1]

INTRODUCTION

This practice is intended to provide the nuclear research community with recommended procedures for using charged-particle irradiation to investigate neutron radiation damage mechanisms as a surrogate for neutron irradiation. It recognizes the diversity of energetic-ion producing devices, the complexities in experimental procedures, and the difficulties in correlating the experimental results with those produced by reactor neutron irradiation. Such results may be used to estimate density changes and the changes in microstructure that would be caused by neutron irradiation. The information can also be useful in elucidating fundamental mechanisms of radiation damage in reactor materials.

1. Scope

1.1 This practice provides guidance on performing charged-particle irradiations of metals and alloys, although many of the methods may also be applied to ceramic materials. It is generally confined to studies of microstructural and microchemical changes induced by ions of low-penetrating power that come to rest in the specimen. Density changes can be measured directly and changes in other properties can be inferred. This information can be used to estimate similar changes that would result from neutron irradiation. More generally, this information is of value in deducing the fundamental mechanisms of radiation damage for a wide range of materials and irradiation conditions.

1.2 Where it appears, the word "simulation" should be understood to imply an approximation of the relevant neutron irradiation environment for the purpose of elucidating damage mechanisms. The degree of conformity can range from poor to nearly exact. The intent is to produce a correspondence between one or more aspects of the neutron and charged particle irradiations such that fundamental relationships are established between irradiation or material parameters and the material response.

1.3 The practice appears as follows:

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials

E170 Terminology Relating to Radiation Measurements and Dosimetry

E821 Practice for Measurement of Mechanical Properties During Charged-Particle Irradiation

E910 Test Method for Application and Analysis of Helium Accumulation Fluence Monitors for Reactor Vessel Surveillance, E706 (IIIC)

E942 Guide for Simulation of Helium Effects in Irradiated Metals

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[1] This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.08 on Procedures for Neutron Radiation Damage Simulation.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 Descriptions of relevant terms are found in Terminology C859 and Terminology E170.

3.2 *Definitions:*

3.2.1 *damage energy, n*—that portion of the energy lost by an ion moving through a solid that is transferred as kinetic energy to atoms of the medium; strictly speaking, the energy transfer in a single encounter must exceed the energy required to displace an atom from its lattice site.

3.2.2 *displacement, n*—the process of dislodging an atom from its normal site in the lattice.

3.2.3 *path length, n*—the total length of path measured along the actual path of the particle.

3.2.4 *penetration depth, n*—a projection of the range along the normal to the entry face of the target.

3.2.5 *projected range, n*—the projection of the range along the direction of the incidence ion prior to entering the target.

3.2.6 *range, n*—the distance from the point of entry at the surface of the target to the point at which the particle comes to rest.

3.2.7 *stopping power (or stopping cross section), n*—the energy lost per unit path length due to a particular process; usually expressed in differential form as $-dE/dx$.

3.2.8 *straggling, n*—the statistical fluctuation due to atomic or electronic scattering of some quantity such as particle range or particle energy at a given depth.

3.3 *Symbols:*

3.3.1 A_1, Z_1—the atomic weight and the number of the bombarding ion.

A_2, Z_2—the atomic weight and number of the atoms of the medium undergoing irradiation.

depa—damage energy per atom; a unit of radiation exposure. It can be expressed as the product of $\bar{\sigma}_{de}$ and the fluence.

dpa—displacements per atom; a unit of radiation exposure giving the mean number of times an atom is displaced from its lattice site. It can be expressed as the product of $\bar{\sigma}_d$ and the fluence.

heavy ion—used here to denote an ion of mass >4.

light ion—an arbitrary designation used here for convenience to denote an ion of mass ≤4.

T_d—an effective value of the energy required to displace an atom from its lattice site.

$\sigma_d(E)$—an energy-dependent displacement cross section; $\bar{\sigma}_d$ denotes a spectrum-averaged value. Usual unit is barns.

$\sigma_{de}(E)$—an energy-dependent damage energy cross section; $\bar{\sigma}_{de}$ denotes a spectrum-averaged value. Usual unit is barns-eV or barns-keV.

4. Significance and Use

4.1 A characteristic advantage of charged-particle irradiation experiments is precise, individual, control over most of the important irradiation conditions such as dose, dose rate, temperature, and quantity of gases present. Additional attributes are the lack of induced radioactivation of specimens and, in general, a substantial compression of irradiation time, from years to hours, to achieve comparable damage as measured in displacements per atom (dpa). An important application of such experiments is the investigation of radiation effects that may be obtained in environments which do not currently exist, such as fusion reactors.

4.2 The primary shortcoming of ion bombardments stems from the damage rate, or temperature dependences of the microstructural evolutionary processes in complex alloys, or both. It cannot be assumed that the time scale for damage evolution can be comparably compressed for all processes by increasing the displacement rate, even with a corresponding shift in irradiation temperature. In addition, the confinement of damage production to a thin layer just (often ~1 μm) below the irradiated surface can present substantial complications. It must be emphasized, therefore, that these experiments and this practice are intended for research purposes and not for the certification or the qualification of materials.

4.3 This practice relates to the generation of irradiation-induced changes in the microstructure of metals and alloys using charged particles. The investigation of mechanical behavior using charged particles is covered in Practice E821.

5. Apparatus

5.1 *Accelerator*—The major item is the accelerator, which in size and complexity dwarfs any associated equipment. Therefore, it is most likely that irradiations will be performed at a limited number of sites where accelerators are available (a 1-MeV electron microscope may also be considered an accelerator).

5.2 *Fixtures* for holding specimens during irradiation are generally custom-made as are devices to measure and control particle energy, particle flux (fluence rate), and specimen temperature. Decisions regarding apparatus are therefore left to individual workers with the request that accurate data on the performance of their equipment be reported with their results.

6. Composition of Specimen

6.1 An elemental analysis of stock from which specimens are fabricated should be known. The manufacturer's heat number and analysis are usually sufficient in the case of commercally produced metals. Additional analysis should be performed after other steps in the experimental procedure if there is cause to believe that the composition of the specimen may have been altered. It is desirable that uncertainties in the analyses be stated and that an atomic basis be reported in addition to a weight basis.

7. Preirradiation Heat Treatment of Specimen

7.1 Temperature and time of heat treatments should be well controlled and reported. This applies to intermediate anneals during fabrication, especially if a metal specimen is to be irradiated in the cold-worked condition, and it also applies to operations where specimens are bonded to metal holders by diffusion or by brazing. The cooling rate between annealing steps and between the final annealing temperature and room temperature should also be controlled and reported.

7.2 The environment of the specimen during heat treatment should be reported. This includes description of container, measure of vacuum, presence of gases (flowing or steady), and the presence of impurity absorbers such as metal sponge. Any discoloration of specimens following an anneal should be reported.

7.3 High-temperature annealing of metals and alloys from Groups IV, V, and VI frequently results in changes, both positive and negative, in their interstitial impurity content. Since the impurity content may have a significant influence on void formation, an analysis of the specimen or of a companion piece prior to irradiation should be performed. Other situations, such as selective vaporization of alloy constituents during annealing, would also require a final analysis.

7.4 The need for care with regard to alterations in composition is magnified by the nature of the specimens. They are usually very thin with a high exposed surface-to-volume ratio. Information is obtained from regions whose distance from the surface may be small relative to atomic diffusion distances.

8. Plastic Deformation of Specimen

8.1 When plastic deformation is a variable in radiation damage, care must be taken in the geometrical measurements used to compute the degree of deformation. The variations in dimensions of the larger piece from which specimens are cut should be measured and reported to such a precision that a standard deviation in the degree of plastic deformation can be assigned to the specimens. A measuring device more accurate and precise than the common hand micrometer will probably be necessary due to the thinness of specimens commonly irradiated.

8.2 The term *cold-worked* should not stand alone as a description of state of deformation. Every effort should be made to characterize completely the deformation. The parameters which should be stated are: *(1)* deformation process (for example, simple tension or compression, swaging, rolling, rolling with applied tension); *(2)* total extent of deformation, expressed in terms of the principal orthogonal natural strain components (ε_1, ε_2, ε_3) or the geometric shape changes that will allow the reader to compute the strains; *(3)* procedure used to reach the total strain level (for example, number of rolling passes and reductions in each); *(4)* strain rate; and *(5)* deformation temperature, including an estimate of temperature changes caused by adiabatic work.

8.2.1 Many commonly used deformation processes (for example, rolling and swaging) tend to be nonhomogeneous. In such cases the strain for each pass can be best stated by the dimensions in the principal working directions before and after each pass. The strain rate can then be specified sufficiently by stating the deformation time of each pass.

9. Preirradiation Metallography of Specimen

9.1 A general examination by light microscopy and transmission-electron microscopy should be performed on the specimen in the condition in which it will be irradiated. In some cases, this means that the examination should be done on specimens that were mounted for irradiation and then unmounted without being irradiated. The microstructure should be described in terms of grain size, phases, precipitates, dislocations, and inclusions.

9.2 A section of a representative specimen cut parallel to the particle beam should be examined by light microscopy. Attention should be devoted to the microstructure within a distance from the incident surface equal to the range of the particle, as well as to the flatness of the surface.

10. Surface Condition of Specimen

10.1 The surface of the specimen should be clean and flat. Details of its preparation should be reported. Electropolishing of metallic specimens is a convenient way of achieving these objectives in a single operation. The possibility that hydrogen is absorbed by the specimen during electropolishing should be investigated by analyses of polished and nonpolished specimens. Deviations in the surface from the perfect-planar condition should not exceed, in dimension perpendicular to the plane, 10 % of the expected particle range in the specimen.

10.2 The specimen may be irradiated in a mechanically polished condition provided damage produced by polishing does not extend into the region of postirradiation examination.

11. Dimension of Specimen Parallel to Particle Beam

11.1 Specimens without support should be thick enough to resist deformation during handling. If a disk having a diameter of 3 mm is used, its thickness should be greater than 0.1 mm.

11.2 Supported specimens may be considerably thinner than unsupported specimens. The minimum thickness should be at least fourfold greater than the distance below any surface from which significant amounts of radiation-produced defects could escape. This distance can sometimes be observed as a void-free zone near the free surface of an irradiated specimen.

12. Helium

12.1 *Injection:*

12.1.1 Alpha-particle irradiation is frequently used to inject helium into specimens to simulate the production of helium during neutron irradiations where helium is produced by transmutation reactions. Helium injection may be completed before particle irradiation begins. It may also proceed incrementally during interruptions in the particle irradiation or it may proceed simultaneously with particle irradiation. The last case is the most desirable as it gives the closest simulation to neutron irradiation. Some techniques for introducing helium are set forth in Guide E942.

12.1.2 The influence of implantation temperature on how helium is distributed in the material (that is, whether helium is dispersed in the lattice, in small clusters, in bubbles, etc.) is known to be important. The consequences of the choice of injection temperature on the simulation should be evaluated and reported.

12.2 *Analysis and Distribution:*

12.2.1 Analysis of the concentration of helium injected into the specimens should be performed by mass spectrometry. Using this technique, the helium content is determined by vaporizing a helium-containing specimen under vacuum, adding a known quantity of ^{3}He, and measuring the ^{4}He/^{3}He ratio.

This information, along with the specimen weight, will give the average helium content in the specimen. The low-level ^{2}He addition is obtained by successive expansion through calibrated volumes. The mass spectrometer is repeatedly calibrated for mass fractionation during each series of runs by analyzing known mixtures of ^{3}He and ^{4}He. Other methods of measurement, such as the nondestructive α-α scattering technique, may be employed, but their results should be correlated with mass spectrometric results to ensure accuracy. Refer to Test Method E910 and Guide E942 for additional details.

12.2.2 In many experiments, attempts are made to achieve uniformity of helium content within the damage region by varying the incident energy of the alpha-particle beam and by avoiding fluence variations on the specimen surface. The success of these attempts should be measured by analyzing separate sections of the specimen for helium. It may be necessary to use several companion specimens for this purpose. Variation of helium concentration through the thickness of the specimen as well as variations across the specimen can also be nondestructively measured with the α-α scattering technique.

12.3 *Alpha-Particle Damage*—Alpha-particle irradiation produces some displacement damage in the specimen. This damage, which changes as the specimen is heated for irradiation by other particles, may influence the radiation effects subsequently produced. Therefore, in those cases where helium injection precedes the particle irradiation, a specimen should be brought to the irradiation temperature in the same manner as if it were going to be irradiated and then examined by transmission-electron microscopy at ambient temperature to characterize the microstructure.

13. Irradiation Procedure

13.1 *Quality of Vacuum*—Contamination of the specimen surface by oxidation or deposition of foreign matter and diffusion of impurities into the specimen must be avoided. A vacuum of 133 μPa (10^{-6} torr) or smaller should be maintained during irradiation for most nonreactive metals. High-temperature irradiation of metals from Groups IV, V, or VI should be done in a vacuum of 1.33 μPa (10^{-8} torr) or smaller. Oil-diffusion pumps should be cold-trapped to restrict the passage of hydrocarbons into the target chamber and beam tube. The visual appearance of the specimen after irradiation and the vacuum maintained during irradiation should be reported.

13.2 *Specimen Temperature:*

13.2.1 The temperature of the specimen should not be allowed to vary by more than ±10°C. It should be controlled, measured, and recorded continuously during irradiation. Infrared sensors offer a direct method of measuring actual temperature of the specimen surface. If thermocouples are used, they should be placed directly on the specimen to avoid temperature gradients and interfaces between the thermocouple and the specimen, which will produce a difference between the thermocouple reading and the actual temperature of the specimen volume being irradiated. A thermocouple should not be exposed to the particle beam because spurious signals may be generated.

13.2.2 Beam heating should be minimized relative to non-beam heating to minimize temperature fluctuations of the specimen due to fluctuations in beam flux (fluence rate) and energy. If a direct measurement of specimen temperature during irradiation cannot be made, then the specimen temperature should be calculated. Details of the calculation should be fully reported.

13.3 *Choice of Particle*—Since the accelerated particles usually come to rest within the specimen, the possibility of significant alterations in specimen composition exists with concomitant effects on radiation damage. If metallic ions are used, they should be of the major constituents of the specimen. Electron irradiation poses no problems in this regard.

13.4 *Choice of Particle Energy:*

13.4.1 Three criteria should be considered in the choice of particle energy:

(1) The range of the particle should be large enough to ensure that the region to be examined possesses a preirradiation microstructure that is unperturbed by its proximity to the surface.

(2) The point defect concentration during irradiation in the observed volume should not differ substantially from that expected of irradiated volumes located far from free surfaces.

(3) The energy deposition gradient parallel to the beam across the volume chosen for observation should be small over a distance that is large compared to typical diffusion distances of defects at the temperature of interest. The best measure of surface influence is the observation of denuded zones for the microstructural feature of interest. The width of denuded zones for voids can be significantly larger or smaller than those observed for dislocations. The volume of the specimen to be examined should lie well beyond the denuded zone because steep concentration gradients of point defects may exist on the boundary of such zones. Gradients in the deposited energy can be reduced by rocking the specimen (varying the angle between the beam and the specimen surface), but local time-dependent flux variations will exist.

13.4.2 The nominal energy of the accelerated particle should be verified periodically by calibration experiments. These experiments should be reported and an uncertainty assigned to the energy.

13.5 *Purity of Beam:*

13.5.1 The use of a bending magnet is an effective way of selecting a particular ion for transit through the beam tube to the specimen. However, it is possible that the selected ions will interact with foreign atoms in the beam tube, causing foreign atoms to strike the specimen also and altering the charge and energy on the selected ion.

13.5.2 A good vacuum in the beam tube will eliminate the significance of these effects, and therefore this vacuum should be monitored during irradiation. A discoloration of the specimen surface could indicate a problem in this regard even though a satisfactory vacuum exists in the vicinity of the specimen.

13.6 *Flux (fluence rate):*

13.6.1 The particle flux (fluence rate) on the specimen should be recorded continuously during irradiation and integrated with time to give the fluence. This is particularly important since most accelerators do not produce a constant flux. Flux and fluence should be reported as particles/$m^2 \cdot s$ and particles/m^2. For the case where the particle comes to rest within the specimen, the specimen holder assembly should be designed as a Faraday cup. The flux measured this way should be checked with a true Faraday cup that can be moved in and out of the beam. If the particles are transmitted through the specimen, a Faraday cup can be positioned on the exit side for flux measurement. Variations in flux during the irradiation should be reported.

13.6.2 It is desirable that the flux be the same everywhere on the specimen surface. The actual flux variation in a plane parallel to the specimen surface should be measured and considered when interpreting results of postirradiation examination. A beam profile monitor is recommended for this purpose. It is possible to mitigate the effects of a spatially nonhomogeneous beam by moving the beam over the surface of the specimen during irradiation. A defocused beam should be used; the maximum translation should be less than the beam half-width.

13.6.3 Rastering (periodic scanning) of a focused beam over the specimen will subject the specimen to periodic local flux variations. It is recommended that a rastered beam be avoided for the simulation of a constant neutron flux, although it may be appropriate for the simulation of a pulsed neutron flux. Radiation-induced defect structures that evolve under such pulsed conditions can differ substantially from those that evolve in a constant flux. Recent work has identified conditions in which significant microstructural differences are observed when a rastered beam is used (**1**, **2**)[3]. It should be noted that pulsed operation is an inherent characteristic of many accelerators.

14. Damage Calculations

14.1 *Scope*—This section covers methods and problems of determining displacement rates for ions and electrons in the energy ranges most likely to be employed in simulations of fission and fusion reactor radiation effects. These are 0.1 to 70 MeV for ions and 0.2 to 10 MeV for electrons, although not all energies within these ranges are treated with equal precision. To provide the basis for subsequent descriptions of neutron-charged particle correlations, the calculation of displacement rates in neutron irradiations is also treated.

14.2 *Energy Dissipation by Neutrons and Charged Particles*—See Appendix X1.

14.3 *Particle Ranges*—Ions suffer negligible deflections in encounters with electrons; hence, if electron losses dominate, differences between range, projected range, and path length will be small. Furthermore, energy dissipation in this case is by a large number of low-energy-exchange events, so range straggling will be small and, at a given depth (except near end of range), energy straggling will be small. These conditions apply to light ions for energies down to the tens of keV range, but only at much higher energies for heavy ions such as nickel.

14.3.1 *Light Ions:*

14.3.1.1 Stopping powers of light ions are easiest to calculate in the range of several MeV to several tens of MeV, but these calculations cannot be done accurately from first principles. At lower energies, heavy reliance must be placed on the few experimental measurements of stopping powers. Several tabulations of stopping powers and the path lengths deduced from them exist (**3-7**). A modern Monte Carlo code, SRIM, can also be easily used to compute the required ranges and stopping powers (**8**).

14.3.1.2 Although the work by Janni (**6**) appears to be the most comprehensive one for protons, experimental range data (**9**) have been produced that are in disagreement with his tables for 1-MeV protons incident on steel. In view of the better agreement of the tables of Williamson et al. (**4**) with these data, it was recommended (**10**) that the latter tables be used for the path length of protons in iron and nickel and their alloys. Ranges can be obtained from these path length values by subtracting a correction for multiple scattering as given by Janni, but this correction is only – 2.2 % at 0.1 MeV, decreasing to – 0.8 % at 5 MeV for protons incident on iron. Ranges for iron should be valid also for steels and nickel-base alloys to within the accuracy of the tables (several percent). The referenced tables should be consulted for data on proton ranges in other metals (the distinction between path length and range is generally ignored) and for deuteron and alpha ranges (**7**). Range estimates can conveniently be made for deuterons and alphas in terms of those for protons for energies at which the stopping power is primarily electronic by employing the following equations:

$$R^{\alpha}(E) \cong R^{p}(E/4) \tag{1}$$

$$R^{d}(E) \cong 2\, R^{p}(E/2) \tag{2}$$

These approximations agree with tabulated values to within better than 5 % for alpha energies >8 MeV and deuteron energies >2 MeV, the accuracy increasing with increasing energy.

14.3.2 *Heavy Ions:*

14.3.2.1 Heavy ions suffer increasing range straggling as the energy is decreased—the spread in range is a large fraction of the mean range at 1 MeV. This corresponds to an increasing fraction of energy lost as kinetic energy imparted to atoms (nuclear stopping) as opposed to excitation and ionization of electrons (electronic stopping).

14.3.2.2 Ranges of heavy ions in the low MeV range cannot be calculated with high accuracy. A semi-empirical tabulation of ranges by Northcliffe and Schilling is available (**3**), and a more recent tabulation of range distributions and stopping powers is contained in a series of books edited by Ziegler and coworkers (**7**). Note that the ranges in Ref (**3**) (actually path lengths) have been corrected for nuclear stopping, whereas their tabulated stopping powers are for electronic stopping only.

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[3] The boldface numbers in parentheses refer to the list of references appended to this practice.

14.3.2.3 Ranges are generally tabulated as areal densities, for example, mg/cm^2; as such they are invariant to changes in mass density. In particular, they apply to material containing voids. The linear range is obtained by dividing the areal density by the mass density—the latter must of course be the actual density, including a correction for void volume if present. An increase in range straggling and energy straggling is caused by the production of voids during an irradiation (**11**).

14.3.2.4 Ranges can be computed with a code developed by Johnson and Gibbons (**12**). It is included as a subroutine in the E-DEP-1 Code (see 14.4.3.1). It permits evaluations of projected ranges and range straggling as well. More recently, the SRIM code (**8**) has been used for such calculations.

14.3.3 *Electrons:*

14.3.3.1 Electrons are subject to many large-angle scattering events; hence range straggling is severe. In radiation damage studies, however, the primary concern is with the passage of electrons through relatively thin targets in which the fractional energy loss is small. This loss can be estimated for many purposes using the following general prescription. The principal loss mechanisms are ionization and radiation. If x is the projected range and N and Z are the atomic density and atomic number of the target, respectively:

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{ion}}\ \alpha\ NZ \tag{3}$$

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{rad}}\ \alpha\ NZ^2\ E \tag{4}$$

for $E > 1$ MeV. Hence, given values for some reference material, energy dissipation for any other material can be estimated. A convenient reference material is lead, in which both mechanisms contribute approximately equally at 10 MeV:

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{ion}} \cong \mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{rad}} \cong 16\ \mathrm{MeV/cm} \tag{5}$$

$$\cdot(\text{or } 1.6\ \mathrm{keV}/\mu\mathrm{m})\ 10\ \mathrm{MeV\ in\ Pb}$$

Using this relation to evaluate the proportionality factors for a second material with atomic number Z_2 and atomic mass A_2 yields:

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{ion}} \cong 0.357\ p_0 Z_2/A_2 \mathrm{keV}/\mu\mathrm{m} \tag{6}$$

or:

$$3.57\ p_0 Z_2/A_2 \mathrm{MeV/cm}$$

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{rad}} \cong 0.000435\ E(\mathrm{MeV})\ p_0 Z_2{}^2/A_2 \mathrm{keV}/\mu\mathrm{m}$$

or:

$$0.00435\ E(\mathrm{MeV})\ p_0 Z_2{}^2/A_2\ \mathrm{MeV/cm} \tag{7}$$

where p_0 is the mass density. For example, these relations give:

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{ion}} \cong 13\ \mathrm{MeV/cm}$$

and:

$$\mathrm{d}E/\mathrm{d}x\,\big|_{\mathrm{rad}} \cong 4\ \mathrm{MeV/cm}$$

for 10-MeV electrons in iron. For 1-MeV electrons in iron, this procedure overestimates the radiation loss by a factor of 3 but at this energy the ionization loss accounts for over 90 % of the energy loss.

14.4 *Damage Energy Calculations:*

14.4.1 *Damage Energy*—A necessary (but not sufficient) condition for consistency between displacement damage estimates for neutrons and charged particles is that the same energy partition model be used in calculating the damage energy. The currently recommended model (**10, 13, 14**) is due to Lindhard et al. (**15**); the expression for the damage energy T_{dam} lost by a knock-on of initial kinetic energy T is:

$$T_{\mathrm{dam}} = T\left[1 + kg(\varepsilon)\right]^{-1} \tag{8}$$

$$k = 0.1337\ Z_1{}^{2/3}/A_1{}^{1/2}$$

$$= T/\left(0.08693\ Z_1{}^{7/3}\right)$$

Following Robinson and coworkers (**16, 17**):

$$g(\varepsilon) = \varepsilon + 0.40244\varepsilon^{3/4} + 3.4008\varepsilon^{1/6} \tag{9}$$

$$\varepsilon = \frac{A_2 T}{(A_1 + A_2)}\,\frac{a}{Z_1 Z_2 e^{\,2}} \tag{10}$$

$$a = a_o\left(\frac{9\pi^{\,2}}{128}\right)^{1/3}\left(Z_1{}^{2/3} + Z_2{}^{2/3}\right)^{-1/2} \tag{11}$$

where a_o is the Bohr radius (5.292×10^{-9} cm), e is the electronic charge (4.803×10^{-10} statcoulomb), and the subscripts 1 and 2 on the atomic numbers (Z) and atomic masses (A) denote the incident ion and the target atoms, respectively. These units require that the kinetic energy, T, in Eq 10 be expressed in ergs.

14.4.1.1 Strictly speaking, this energy partitioning model can only be applied to monatomic systems, that is, $Z_1 = Z_2$. However, it can reasonably be applied as long as these two values are sufficiently close (**16**). In the case of alloy targets, an effective Z should be calculated by weighting the alloy constituents by their respective atomic fractions. In addition, the Lindhard model is limited to energies T less than about $25 \cdot Z_1{}^{4/3} \cdot A_1$ (in keV) (**16**).

14.4.2 *Neutrons:*

14.4.2.1 The calculation of damage energy for neutron irradiations is most conveniently expressed in terms of an energy-dependent damage energy cross section, $\sigma_{de}(E)$. This expresses the damage energy per atom per unit neutron fluence; a convenient unit is eV-barns. In calculating this cross section, all possible reactions that can transfer sufficient energy to an atom of the medium to displace it must be considered. These include elastic scattering, inelastic scattering, neutron multiplication reactions [for example, (n,2n)], charged-particle-out reactions [for example, (n,p)], and absorption reactions (n,γ). Most of the necessary data are included in the ENDF/B files (**18**), and it is recommended that these be used in damage calculations.

14.4.2.2 The treatment of the kinematics for these reactions has been documented (**19-21**); the result is a cross section $\mathrm{d}\sigma(T,E)$ for the production, by all possible reactions, of a primary knock-on atom (PKA) of energy T by a neutron of energy E. The damage energy cross section is then simply the integral of the product of this primary cross section and the damage energy, T_{dam}, associated with a PKA of energy T:

$$\sigma_{de}(E) = \int_0^{T_m} T_{\mathrm{dam}}\left[\mathrm{d}\sigma(T,E)/\mathrm{d}T\right]\mathrm{d}T)\ (\mathrm{eV} - \mathrm{barns}) \tag{12}$$

The upper limit of the integral, T_m, is the maximum possible PKA energy; in the absence of charged particle emission, it results from a head-on elastic collision and is given by:

$$T_m = 4A_2/(A_2+1)^2E \tag{13}$$

where the atomic weight is expressed in terms of neutron masses, as in ENDF/B notation. Higher values of T_m are possible in some charged-particle-out reactions that are exoergic. The lower limit in Eq 12 was sometimes assumed to be T_d, an effective displacement energy. When E exceeds several keV, the difference between using T_d and 0 is small.

14.4.2.3 To determine the damage energy density in a neutron-irradiated material, the neutron flux-spectrum $\varphi(E)$ must be known. The damage energy deposition per atom (depa) per second is then:

$$\text{depa/s} = \int_0^\infty \varphi\,(E)\sigma_{de}\,(E)\,\mathrm{d}E \tag{14}$$

This can be converted to damage energy per cubic centimeter per second by multiplying by N, the atom density. The cumulative damage energy density is obtained by integrating over the irradiation time.

14.4.2.4 Since, for most reactor spectra, the damage energy contributed by neutrons of energy less than a few keV is negligible, the depa for neutron irradiations is generally independent of T_d (see further discussion under 13.6.2).

14.4.3 *Heavy Ions:*

14.4.3.1 In general, the damage energy depends on the ion energy so it will vary with penetration. A simple computer code, E-DEP-1 (**22**), was developed and extensively applied for calculating damage energy versus depth distributions for heavy ions. It made the simplifying assumption of approximating energy straggling by using the range straggling theory of Lindhard et al. (**23**). Also implicit is the additional assumption that the ranges of knock-on atoms are negligible; that is, all damage energy is deposited in the immediate vicinity of the point at which the incident ion produces the knock-on atom (energy transport is neglected). Beeler (**24**) has performed computer experiments and Winterbon (**25**) has made analytical calculations to estimate the effect of this assumption on the shape of the damage energy-depth profile. The effect is not large for experiments that effectively integrate over macroscopic intervals (for example, 50 nm) of the profile. The more modern Monte Carlo code SRIM (**8, 26, 27**) is now most commonly used to perform these calculations. The use of SRIM permits more sophisticated analyses to be performed than does EDEP-1. SRIM is relatively fast and can be used for both light- and heavy-ion irradiations as long as nuclear reactions are not involved.

14.4.3.2 The damage-energy density increases with depth, reaches a peak, and then drops rapidly to zero. In the vicinity of the peak, the uncertainty in the E-DEP-1 calculation must be assumed large—perhaps 25 to 50 % (**10**). Nearer the specimen surface where the gradient and damage energy is less, the uncertainty is perhaps 20 %. The uncertainty in SRIM calculations may be lower. Measurements of observed damage versus depth are highly recommended if the intent is to make damage observations in the peak damage region.

14.4.3.3 In applying E-DEP-1, the user has the option of describing electronic stopping of the incident ion using the expression for k given by Lindhard et al. (**23**), or reading in some other value. k is the proportionality factor between the electronic stopping power and the ion velocity. SRIM includes a more modern description of electronic stopping. Lindhard et al. gives the approximate expression:

$$k = 0.0793\,Z_1^{1/6}(Z_1Z_2/Z)^{1/2}A_2/A_0^{3/2} \tag{15}$$

in which:

$$Z^{2/3} = Z_1^{2/3}+Z_2^{2/3},\ A_0 = A_1A_2/(A_1+A_2) \tag{16}$$

It is suggested that better k values may be determined directly from the tabulated stopping powers of Northcliffe and Schilling (**3**).

14.4.4 *Light Ions:*

14.4.4.1 Damage energy estimates for light ions at low energies can be made in a more straightforward manner. The mean energy, E_x, at depth x is first determined from tables as follows. Let E_0 be the incident ion energy and $R(E)$ the mean range of an ion of energy E. Assume range and energy straggling are negligible. Then the residual range of an ion at x is $R(E_x) = R(E_0) - x$. Given E_0 and x, one can find $R(E_0)$ in the range-energy tables, calculate $R(E_x)$, and thus determine E_x from the tables. A knowledge of E_x permits application of the Rutherford scattering cross section, $d\sigma_R(T,E_x)$, which gives the approximate number of knock-ons in the interval $\mathrm{d}T$ at knock-on energy T that is produced by an ion of energy E_x (**28**):

$$\mathrm{d}\sigma_R(T,E_x) = (B\gamma^2/E_x)(\mathrm{d}T/T^2) \tag{17}$$

where:

B = $4\pi a_0^2\,E_R^2(A_1/A_2)Z_1^2Z_2^2$,
$\gamma_1 Z_1$ = effective charge of the moving ion,
a_0 = 0.053 nm, and
E_R = 13.6 eV.

A convenient expression for γ given by Bichsel (**29**) is $\gamma = 1 - \exp\,(-1.316\ y + 0.1112\ y^2 - 0.0650\ y^3)$; $y = 100\beta/Z_1^{2/3}$ where $\beta(<< 1)$ is the ratio of the particle velocity to that of light. Expressed as a function of particle energy, $y = (4.63/Z_1^{2/3})\ [E_x(\mathrm{MeV})/A_1]^{1/2}$. The damage energy cross section is given by integrating over the product of the number of events producing a knock-on of energy T [$\mathrm{d}\sigma_R(\mathrm{T,E}_x)$] and the damage energy associated with the knock-on, T_{dam}:

$$\sigma_{de}\,(E_x) = (B\gamma^2/E_x)\int_{T_d}^{T_m} T_{\mathrm{dam}}\,(\mathrm{d}T/T^2) \tag{18}$$

Unlike Eq 12, the lower limit of this integral which includes an explicit form for the cross section is the mean energy required to displace an atom, T_d, and the upper limit is the maximum possible energy transferred to an atom given by:

$$T_m = 4A_1A_2/(A_1+A_2)^2E_x. \tag{19}$$

Then depa/s is the product of the particle flux φ and σ_{de}. If the atom density is N and the irradiation time is t, the damage energy density (eV/cm^3) is given by $\varphi t N\sigma_{de}$.

14.4.4.2 The Rutherford scattering cross section describes only coulomb scattering. Another source of elastic scattering for light ions above several MeV is nuclear potential scattering.

Large-angle coulomb scattering is rare and hence large-angle elastic scattering will be dominated by potential scattering above several MeV, as discussed by Logan et al. (**30**) for niobium. To calculate correctly the elastic scattering contribution to the displacement cross section, experimental data on angular differential cross sections or optical model code computations of these cross sections must be used. The results for medium Z materials are generally lower than obtained, assuming coulomb scattering. However, in the same energy range, nonelastic scattering begins to become significant. Rigorous calculations of this contribution have not yet been made, although the approximate method used by Logan et al is probably adequate. It appears that nonelastic scattering will become dominant with increasing energy and will generally more than offset the decrease in the elastic contribution relative to coulomb scattering. That is, Eq 2 may significantly underestimate the damage energy cross section for light ions above ~10 MeV.

14.4.5 *Electrons*—The concept of damage-energy density is not particularly helpful in electron irradiations except for very high electron energies because mean knock-on energies generally do not greatly exceed displacement thresholds. However, the damage energy can be estimated from Oen's tables (**31**) as $T_{\mathrm{dam}} \cong 2\,T_d\,\sigma_d$, where σ_d is Oen's displacement cross section. Note that Oen used the energy partition model of Kinchin and Pease rather than that of Lindhard et al.

14.5 *Conversion of Damage Energy to DPA:*

14.5.1 *Model:*

14.5.1.1 A secondary displacement model describes the number of displacements N_d produced in a cascade initiated by a PKA of kinetic energy T. The simplified model recommended here is based on Ref (**16**) and has been adopted by both the IAEA (**13**) and researchers in the U.S. (**10, 14**) (for iron, nickel, and their alloys):

$$\begin{aligned} &N_d = 0 && T < T_d \\ &N_d = 1 && T_d \le T < 2T_d/\beta \\ &N_d = \beta T_{dam}/2T_d && T \ge 2T_d/\beta \qquad (20) \end{aligned}$$

The previously recommended values for iron, steel, and nickel-base alloys are $\beta = 0.8$ and $T_d = 40$ eV, or $N_d = 10\,T_{\mathrm{dam}}$, if T_{dam} is expressed in keV. While the value assigned to the effective displacement energy, T_d, is somewhat arbitrary, it is most important that a specific secondary displacement model be used for the purpose of standardization; hence the model presented in Eq 20 is recommended. There is some error incurred in using Eq 20 due to the neglect of inelastic energy losses at very low energies. Robinson and Oen have discussed this in detail and provide an expression for a simple correction factor (**32**).

14.5.1.2 The actual displacement energy depends on the direction of ejection of the atom (**33**) (see Appendix X1). The value of T_d used in Eq 20 should represent an average overall ejection direction. Sufficient data to permit calculation of T_d exist for only a few metals. In any event, the value of 40 eV recommended for steels is based largely on computer simulation of low-energy cascades, rather than directly on displacement threshold measurements. The point here is that there is no basis for assigning overly precise T_d values for various metals. In order to foster uniformity in displacement calculations, a list of recommended T_d values is given in Table 1, along with some measured threshold values. The T_d values are rounded to emphasize their approximate nature. The recommended values are generally consistent with molecular dynamics simulations that have investigated the directional dependence of the displacement threshold in a number of materials (**33**). The values obtained for iron using molecular dynamics simulations are in generally good agreement with an extensive investigation using *ab initio* calculations to determine the angular dependence of the displacement threshold (**34**). For those metals for which Lucasson (see Table 1) gives average values, the agreement is with 10 % except for Cr, Ni, and Nb. The value for Cr was set equal to that recommended for Fe and Ni (Lucasson gives 60 eV for Cr and 33 eV for Ni), since it is generally of concern only as a component of stainless steel. The value for Nb (Lucasson gives 78 eV) was set equal to that for Mo, consistent with some existing displacement calculations; there is little evidence for using different values.

14.5.2 *Neutrons:*

14.5.2.1 The calculation of a damage energy cross section, σ_{de} (see 14.4), is simply converted to the calculation of a displacement cross section, σ_d, by replacing T_{dam} with N_d in Eq 13. σ_d, usually expressed in barns, represents the number of displacements per atom (dpa) per unit neutron fluence. For practical purposes, the difference in the form of $N_d(T_{\mathrm{dam}})$ between T_d and $2T_d/\beta$ can be ignored and one can write:

$$\sigma_d = (\beta/2T_d)\sigma_{de} \qquad (21)$$

Furthermore, as pointed out in 14.4, for any neutron spectrum not dominated by neutrons of energy less than several keV, the lower limit of the integral of Eq 14 can be taken as zero and σ_{de} becomes independent of T_d, while σ_d becomes inversely proportional to T_d.

NOTE 1—The above recommendations embodied in Eq 14 and Eq 17 are consistent with current practice in Europe for calculating displacement

TABLE 1 Recommended Values of the Effective Displacement Energy for Use in Displacement Calculations

Metal	$T^{\min}$ (eV)[A]	T_d (eV)
Al	16	25
Ti	19	30
V	—	40
Cr	28[B]	40
Mn	—	40
Fe	20[B]	40
Co	22	40
Ni	23	40
Cu	19	30
Zr	21	40
Nb	36[B]	60
Mo	33	60
Ta	34	90
W	40	90
Pb	14	25

[A] See review by P. Lucasson in *Proceedings of International Conference on Fundamental Aspects of Radiation Damage in Metals,* Gatlinburg, Tenn., October 1975.

[B] An effective threshold measured in a polycrystalline specimen.

rates in iron and nickel alloys. However, this does not ensure the equivalence of all displacement calculations because different sets of neutron-scattering cross sections and different treatments of those cross sections may be used. For example, displacement calculations made in the U. K. for steel based on the so-called NRT standard, to which Eq 14 and Eq 17 are equivalent, are not identical to calculations using the data in Ref (**35**). This is because an elastic-isotropic scattering approximation is used in the former, whereas inelastic scattering and anisotropy are included in the latter.

14.5.2.2 Tabulations of $\sigma_d\,(E)$ (easily converted to σ_{de}) calculated in accordance with the above recommendations are available (**35**).

14.5.2.3 It is often convenient to employ spectrum-averaged values of $\sigma_d\,(E)$, denoted here by $\bar{\sigma}_d$ (or $\sigma_{d\varepsilon}$), in order to characterize the particular irradiation facility having a neutron spectrum $\varphi(E)$. These are defined by:

$$\bar{\sigma}_d = \int_0^\infty \sigma_d(E)\,\varphi(E)\,\mathrm{d}E / \int_0^\infty (E)\varphi\mathrm{d}E \tag{22}$$

The displacement rate (dpa/s) in such a facility is then simply the product of the total flux, φ, and $\bar{\sigma}_d$. Again, for practical purposes, $\bar{\sigma}_d$ is proportional to T_d^{-1}.

14.5.3 *Heavy Ions*—The damage energy density, as calculated for example using the E-DEP-1 or SRIM Codes (see 14.4), can be converted to a displacement density by multiplying by $\beta/2\ T_d$. As in the neutron case, the change in form for N_d between T_d and $2T_d\,/\beta$ is ignored. Recommendations for the use of SRIM for computing dpa are given in Ref (**36**).

14.5.4 *Light Ions*—The calculation of the damage energy cross section in Eq 15 of 14.4.4 is easily modified to give a displacement cross section by substituting N_d from Eq 17 for T_{dam}.

14.5.5 *Electrons:*

14.5.5.1 As indicated in 14.4, the concept of damage energy is not particularly useful in low-energy electron bombardments. The proper calculation of dpa requires a knowledge of the direction-dependent displacement energy for the crystal under study, which is unknown for most metals (see Appendix X2). If an effective displacement energy is used instead, that is, a step function displacement probability rising from 0 to 1 at T_d, the table of Oen can be consulted to determine the displacement cross section for any metal. This approach gains validity as the electron energy is increased. However, if Oen's tables are used for energies so great that secondary displacements are important, then his values, calculated with a Kinchin-Pease model, are inconsistent with the present recommendations. (The secondary displacement contribution would have to be greater than perhaps 50 % for the inconsistency to exceed 10 %.) The effective displacement energy is a parameter in Oen's tables. Using the values for T_d in Table 1 (or similarly derived values) probably leads to unrealistically low displacement cross sections under some conditions. An alternative procedure is to use an estimated displacement energy function (for example, a ramp starting from zero at the threshold displacement energy, T_d^0, rising to unity at 2 to 4 times T_d^0) rather than a step function. Applying it also to the light ion (particularly proton) case will increase the consistency of electron and light ion displacement calculations.

14.5.5.2 It should be recognized that the displacement cross section can be a sensitive function of the orientation of the electron beam relative to the crystal axes. This becomes an additional variable to be controlled in HVEM irradiation of oriented specimens and may produce grain-to-grain differences in irradiations of polycrystalline specimens.

15. Extraction of Foils for Transmission Electron Microscopy

15.1 *Scope*—This section covers several recommended methods for extracting a foil for transmission electron microscopy from within an irradiated specimen. These methods involve controlled removal of material from the irradiated front surface and from the unirradiated back surface so that the distance of the foil from the irradiated front surface is accurately known. These methods are not necessary in the case of electron irradiations where the electrons pass through the specimens producing the same radiation damage throughout.

15.2 *Removal of Material from Irradiated Surface*—Several techniques are available for the careful removal of material from the irradiated surface, prior to back-thinning, so that damage structures may be examined at selected positions along the particle range.

15.2.1 *Electropolishing:*

15.2.1.1 Part of the irradiated surface is protected by lacquer to provide a reference plane and the rest of the surface is carefully electropolished either continuously or in short pulses. It should be noted that polishing rates of irradiated surfaces may differ considerably from rates determined on non-irradiated surfaces. It is important that the electrolyte and current density chosen should produce a good polished surface. A badly etched or pitted surface makes subsequent microscopy rather difficult, as well as introducing a further uncertainty in the measurement of the position of the foil below the irradiated surface.

15.2.1.2 Material removal is rapid, typically of the order 0.1 to 0.5 µm/s. The major disadvantage is nonuniformity. Polishing generally tends to be more rapid at the edges of the specimen and at the edge of the protective lacquer. In complex alloys, electropolishing rates may change rapidly in the vicinity of large second phase particles.

15.2.2 *Ion Milling:*

15.2.2.1 In this technique, specimens are bombarded with rare gas ions, usually argon or xenon, accelerated to somewhere in the range from 700 to 2000 eV. Using beam currents of approximately 1 $\mathrm{mA/cm^2}$, milling rates with metallic specimens are typically of the order 10^{-3} µm/s. Uniform removal of layers as small as 20 nm thick is readily achievable. The rate of material removal is orientation-dependent, the sensitivity to orientation varying greatly with alloy composition and metallurgical condition. This is not usually a problem if the amount of material being removed is approximately 1 µm. However, when it is required to mill to greater depths, differences in material removal from grain to grain may become unacceptably large.

15.2.2.2 In order to measure the amount of material removed, some part of the specimen surface is masked off from the beam. This may be done in several ways: (*1*) by electroplating several very small patches of copper on to the specimen surface. After milling, the copper is removed in nitric acid. This would not apply, of course, to specimens susceptible to attack by nitric acid; (*2*) by placing several dots of lacquer on the specimen surface and dissolving in a suitable organic solvent after milling. In some instances, lacquers may be rendered insoluble during ion milling by radiation-induced polymerization; (*3*) placing a suitable metallic mask (for example, a stainless steel ring) in contact with the specimen surface.

15.2.2.3 The major advantages of ion milling are that the surfaces produced are very clean and that the material removal rate is easily controlled. The disadvantages are that blackspot irradiation damage is produced to a depth of 20 to 40 nm below the surface.

15.2.2.4 A more recent variant of ion milling is known as focused ion beam (FIB) milling (**37**, **38**). The use of this approach permits local thinning and extraction of very small specimens for electron microscopy and small-scale mechanical testing.

15.2.3 *Vibratory Polishing:*

15.2.3.1 In this technique, specimens are mounted flat and placed with the irradiated face downwards in a suspension of fine abrasive powder (for example, 50-nm particle diameter alumina) on a vibrating polishing cloth pad. Polishing rates are of the order 0.5 to 1.0 µm/h. The amount of material removed may be determined by careful periodic weight loss measurements. In this way it is possible to measure the removal of layers 100 nm thick. Since it is often found that the polishing rate is not uniform across the specimen surface, an alternative method is to measure the change in dimensions of conical surface microhardness indentations using interferometry. The major disadvantage of this method of sectioning is that even under the best conditions, a damaged layer is produced that extends 100 to 200 nm below the specimen surface. This layer must be removed by a short electropolish or ion mill with an accompanying measurement.

15.2.3.2 Vibratory polishing finds its most useful application in cases where the region of interest is greater than 1.5 to 2.0 µm below the bombarded surface.

15.3 *Determination of Distance from Irradiated Surface:*

15.3.1 *Surface Profilometry*—A stylus with a spherical diamond tip having a diameter of about 25 µm or less bears upon the specimen surface with a load of about 0.3 mN. The specimen is translated and the stylus movement across the original and the new lower surface is sensed by a differential transformer. With this technique it is possible to detect differences in surface heights of about 3 nm. However, in most instances, sensitivity is limited by the specimen surface roughness, which is rarely better than ±25 nm. Some caution should be exercised in the measurement of step heights on nonplanar surfaces. The major advantage of this technique is its rapidity and the wide range of surface heights that may be measured reproducibly. Another important advantage is that the measurement is not confined to the vicinity of the surface step. Information on the surface shape across the entire specimen is presented in a readily interpretable form. Some plastic deformation may occur under the action of the stylus and so profilometer measurements should be made well away from areas that are to be examined in the electron microscope.

15.3.2 *Interferometry:*

15.3.2.1 Both two-beam and multiple-beam interferometry provide a means of measuring step heights in the range from 0.01 to 10.0 µm. The sensitivity of the two-beam technique is about ±25 nm, while the multiple-beam technique is capable of detecting displacements as small as 5 nm. On the other hand, it is sometimes difficult to measure steps that produce more than 2 to 3 fringe displacements using the multiple-beam technique, particularly when the step is sharp. Multi-fringe displacements are easier to follow in the two-beam case since it is possible to use white light to produce chromatic fringes.

15.3.2.2 In practice, accuracy of measurement is limited by the surface roughness and the steepness of the step height being measured. It becomes difficult to make measurements when the surface roughness begins to exceed 50 to 75 nm, or if the boundary between the original and the new lower surface is an irregular slope rather than a sharp step.

15.3.2.3 Care must be taken to avoid errors due to effects associated with the interface between the new and original surfaces of the specimen. For example, electropolishing is usually more rapid in the region adjacent to the masking lacquer. If a metallic mask is used during ion milling, it is possible for sputtered material to be redeposited between the mask and the specimen surface.

15.3.2.4 Interferometric techniques have the advantage of not introducing any surface damage. The multiple-beam technique requires a highly reflective surface and it is usually necessary to evaporate a thin layer of aluminum on the area where the measurement is made.

15.3.3 *Radiation Attenuation:*

15.3.3.1 As material is removed from the irradiated surface of a sample for the purpose of reaching a preselected position, the sample thickness can be monitored periodically by measurement of radiation attenuation. The sample thickness is determined by comparison of attenuation for that sample with a standard plot of attenuation versus thickness. Attenuation is measured as I/I_0, where I is the intensity of radiation passing through a sample and I_0 is the absolute source intensity measured with no sample. The thicknesses used in obtaining the standard plot are from foils whose thicknesses have been measured by an independent means. For example, an interferometer that has an accuracy within 25 nm can be used. A standard plot of I/I_0 versus thickness must be determined for each pure metal or alloy that is to be examined.

15.3.3.2 The standard plot of radiation attenuation should be checked frequently by use of one or more standard foils. A precise foil-positioning system must be employed to ensure that the radiation beam passes through the region in which the original thickness measurement was made by interferometric or other means. This eliminates errors that may occur because of variations in standard foil thickness.

15.3.3.3 Both β and X rays have been used for thickness measurements. For the former, a β-emitter such as ^{147}Pm is an excellent source because of beam stability. In the use of β and x rays, beam collimation is important. The beam should be collimated to as small a diameter as possible without sacrificing detection accuracy. With a small beam, the sample can be scanned to determine variations in thickness that may be present in the original foil or may develop during the thinning process. Scanning is facilitated by the use of an accurate positioning device that allows the sample to be moved about under the beam. It should be noted that the attenuation method measures mass thickness, which differs from the linear thickness when voids are present.

15.4 *Preparation of Foils by Removal of Material from Unirradiated Surface (Back-Thinning)*—The preparation of electron-transparent foils is accomplished by applying a protective lacquer to the irradiated surface, or to a new surface prepared below the original surface (to be referred to as the front surface), and then electropolishing the other surface of the specimen until perforation occurs. This process, named back-thinning, may be accomplished by one of several techniques:

15.4.1 *Jet Electropolishing:*

15.4.1.1 With this technique, it is convenient to have the specimen in the form of a 3-mm diameter disk, 100 to 500 µm thick. The front surface is protected by a lacquer and the unirradiated or back surface is electropolished with a jet of electrolyte to perforation. Perforation may be detected by directing a light source at one surface and observing the other surface visually or by using an electronic light detection system that automatically cuts off the polishing voltage. The lacquer must maintain its integrity and be transparent to light.

15.4.1.2 It is particularly important that protection of the front surface is maintained when the foil perforates so that electrochemical attack of this surface does not occur in the short time before the current is switched off. The front surface should be examined carefully by optical microscopy after perforation to check for signs of attack in the vicinity of the perforation. Due to the action of the jet, there is a tendency for the front surface to be attacked on perforation if the lacquer is forced off, and there is also a tendency for the thinner regions of the foil to be buckled.

15.4.2 *Electropolishing*—The back surface of a 3-mm disk specimen is first jet-electropolished to within 25 to 40 µm of the bombarded surface. This stage is unnecessary if the irradiated specimen is of a similar thickness. The perforation stage is carried out in a static or slowly stirred electrolyte, with visual or automatic detection of perforation. This technique is more time-consuming than jet electropolishing but protection of the front surface during perforation is better and foils are somewhat flatter.

15.4.3 *Ion Milling*—In some instances, ion milling may be used to increase the total electron transparent area in a back-thinned foil. The back surface is protected with lacquer and the front surface is electroplated with a thin metallic coating for support. The lacquer is removed and the back surface of the specimen is ion-milled. The metallic coating is then dissolved away from the front bombarded surface.

15.5 *Determination of Error in Distance from Irradiated Surface:*

15.5.1 Errors in determining the position of the final foil surface arise from two sources. First, there is a basic uncertainty in the measuring technique, which includes the accuracy and precision of the instrument and the roughness of the surface. This uncertainty may be determined by making a number of measurements of the same step and calculating the standard deviation. A second source of uncertainty arises from nonuniformity in the rate of material removal over the area of the specimen. This is particularly important when the masked regions are at the periphery of the specimen. For example, it is frequently observed that electropolishing is more rapid at the edge of the lacquer mask and then decreases in rate towards the center of the specimen. It is therefore essential to investigate the uniformity of material removal by profilometry of dummy specimens and to determine the range of uncertainty involved. This source of error may be minimized by placing the mask adjacent to the region where perforation is expected to occur and making measurements, although there is a risk of damaging the area to be examined.

15.5.2 If the ion milling or electropolishing rate is sensitive to grain orientation, the amount of material removed will vary from grain to grain. It is therefore recommended that the grain-to-grain height differences be measured before and after sectioning.

15.6 *Uncertainties in Assigning dpa Values to Foils*—In addition to the uncertainties in the calculation of energy deposition and dpa, further errors in assigning dpa values arise from experimental sources.

15.6.1 Since energy deposition varies with depth, uncertainties in the experimentally determined depth position of the foil give rise to corresponding uncertainties in the displacement dose.

15.6.2 There is a variation in energy deposition within the thickness of an electron-transparent foil. The magnitude of this variation depends upon the depth below the irradiated surface at which the foil is prepared.

15.6.3 In cases where specimens undergo limited dimensional changes, <10 % swelling, it is reasonable to assume that the energy deposition curve calculated for a fully dense homogeneous alloy is still applicable. However, at some higher value of swelling, some correction must be applied to account for the effects of voids on particle range and straggling. Methods of calculating the magnitude of these effects have been described **(11).**

15.6.3.1 The application of these corrections requires a knowledge of the swelling profile, which may be determined in one of four ways: (*1*) by taking a number of specimens irradiated to the same peak dpa and preparing sections at various depths, (*2*) using a single specimen to prepare a cross section parallel to the beam and examining the entire ion range **(39),** (*3*) by using a single back-thinned specimen and making observations at several successive depths following repeated ion milling of the bombarded surface, and (*4*) in cases where the ion range is ≤2 µm, 1 MeV-electron microscopy may be used to examine a foil back-thinned to the irradiated surface, and the swelling profile may be determined by stereo microscopy.

15.6.3.2 Having determined the swelling-depth profile for a given ion fluence, a corrected energy deposition curve may be calculated and used to determine the correct linear depth for subsequent sectioning operations, or to correct dpa values assigned to previously prepared sections.

15.6.3.3 It should be noted that this correction is not required when an attenuation method is used to determine distances and the energy deposition curve is described in terms of areal density (kg/m^2).

16. Determination of Swelling and Void Density by Transmission Electron Microscopy

16.1 *Scope*—This section covers methods for calculating swelling by making measurements on electron photomicrographs to determine void volumes and void densities (number of voids per unit volume). It does not deal with methods of imaging voids in the electron microscope nor with methods for determining the thickness of the foil specimen in the electron microscope.

16.2 *Definition*

16.2.1 *swelling*—defined as:

$$\% \, \Delta V/V_0 = 100[\Delta V/(V_f - \Delta V)] \tag{23}$$

where the void volume, ΔV, is the summation of the volume contributions of each void measured in a total volume, V_f. The original metal volume, V_0, is equal to the final metal volume, $V_f - \Delta V$.

16.3 *Calculation of Void Volumes by Two-Dimensional Measurements:*

16.3.1 Voids appear as projections in two-dimensional electron photomicrographs. The volume of a spherical void may be calculated by measuring the diameter of the void with a reticle. For nonspherical voids, the precise geometry must be determined. The dimension appropriate to the shape is then used to calculate the void volume, for example, the edge length for cubic voids. An approximate volume may be calculated for voids where the shape is unknown but close to spherical.

16.3.2 Void diameters may also be measured with a particle-size analyzer in which a variable diameter circle of light is matched to the void image and the void size is automatically recorded into one of a number of size classes. The dimension, D_i, recorded here, is an average diameter of the voids in size class i.

16.4 *Calculation of Void Densities:*

16.4.1 Void density is defined as the number of voids per unit volume expressed as voids/m^3. It is calculated by counting all the voids in a fixed area and computing the volume in which they are embedded. The third dimension necessary for this computation is the thickness of the foil, which may be measured by several methods **(40, 41)**.

16.4.2 Height-, pressure-, or position-sensitive analog techniques are also available for void characterization and are easily and directly coupled to computers.

16.4.3 *Correction of Void Density:*

16.4.3.1 Voids that intersect the foil surface during electropolishing quickly erode to larger sizes and more irregular shapes, and may lose observable contrast. Void number data are frequently corrected to bulk-representative densities by assuming that the void size distributions are randomly distributed and homogeneous in each size class. Voids whose centers lie within 0.5 (D_i) of each foil surface may still be detectable but should not be counted.

16.4.3.2 Bulk-representative void densities are then given by:

$$\rho_v = \frac{1}{A}\sum^{i}[\eta_i/(t_f - D_i)] = (1/At_f)\sum^{i} N_i \tag{24}$$

where:

η_i = observed number of voids in size class *i*,
N_i = number of voids corrected for surface intersections

= $\eta_i t_f/(t_f - D_i)$
A = area in which measurements are made,
t_f = foil thickness, and
D_i = mean diameter of size class *i*.

This equation assumes that both foil surfaces slice through bulk-produced void distributions.

16.4.3.3 In some cases, however, a region of voids is developed between or behind surface zones, free of voids called denuded zones, and the appropriate thickness is not t_f but $t_v = t_f - KL_{DZ}$, where L_{DZ} is the denuded zone thickness. Depending on both the simulation and data retrieval techniques, K can be either 0, 1, or 2. Surface corrections for electron-microscope irradiation experiments sometimes involve void loss due to shrinkage when void outer surfaces grow through the denuded boundaries. In such cases, bulk representative void numbers are calculated using the following correction:

$$p_v = \sum^{i}{}_{i} = (1/\mathrm{At}_v)\sum^{i}[(t_v + D_i/t_v)\,]\eta_i \, . \tag{25}$$

16.5 *Calculation of Swelling*—The void volume fraction, corrected for surface intersections on two sides, is:

$$\Delta V/V = (1/A)\sum^{i}[\eta_i \Delta V(D_i)/(t_f - D_i)] \tag{26}$$

and the swelling, % $\Delta V/V_0$, should be calculated using Eq 23.

16.6 *Description of Void Size:*

16.6.1 It is convenient to report the mean value of the void diameter as:

$$\bar{D} = \sum N_i D_i / \sum N_i \tag{27}$$

or the volume-averaged diameter as:

$$\bar{D}_m = \left[\sum N_i D_i{}^3 / \sum N_i\right]^{1/3} \tag{28}$$

16.6.2 If a size frequency distribution is not presented, the standard deviation for the mean diameter is often reported as:

$$S_D = \left[\frac{\sum (D_i - D)^2 N_i}{\sum N_i}\right]^{1/2} \tag{29}$$

Void densities calculated with Eq 24 are usually reported also.

16.7 *Statistical Considerations in Sampling*—The transmission electron microscope samples a very small volume of approximately 10^{-16}m^{-3}. Many materials exhibit local variations in void formation over distances larger than those contained within fields of view of the electron microscope. This requires random sampling of many volumes to achieve a value of swelling that is representative of bulk swelling. Several measurements of void populations, using separate fields of view, in different grains, should be made to achieve an acceptably low standard deviation of the results if the void formation is homogeneous. In the case of inhomogeneous void formation, many more volumes must be sampled.

17. Determination of Swelling by Surface Profilometry

17.1 *Scope*—Surface profilometry provides a method for rapidly evaluating large swellings produced by ions whose range does not exceed several micrometres. Because the specimen thickness is large compared to the ion range, the thin damaged layer is constrained by the underlying material and the growth due to swelling occurs only in the direction perpendicular to the surface. If the surface is partially masked during ion irradiation, the irradiated region will become elevated with respect to the adjacent shielded surface as swelling occurs, and a step will form at the boundary between the two regions. The step height is equal to the total swelling integrated along the ion path, subject to corrections.

17.2 *Measurement of Step Height*—The height of the step at the boundary between the bombarded and protected region can be conveniently measured with a stylus-type profilometer (**42**, **43**). There is no practical limitation on the maximum step height that can be measured, and the limitation on small step heights arises, not with the instrument, but with the roughness of the specimen surface. A step height as small as 10 nm can be measured on a surface that was electropolished prior to irradiation, provided the specimen has not been plastically deformed by clamping during irradiation, nor become otherwise roughened.

17.3 *Sources of Error*—Ion irradiation can cause removal of material from the surface (sputtering) and this may diminish the observed step height. Irradiation with metal ions increases the volume of the specimen and may also increase the surface roughness. These effects should be calculated and the results applied to correct the observed step height.

17.4 *Interpretation of Results*—A step height indicates the total swelling integrated along the range of the bombarding particles. The depth distribution of swelling is determined by the depth distribution of displacement damage. The information provided by step height measurements at several ion doses may be used in conjunction with the displacement damage curve to construct an entire curve of swelling versus dpa (**43**). Although a step height indicates the swelling over a range of dpa values, an empirical correlation of step heights and values of peak swelling obtained by transmission electron microscopy may provide a factor for converting a single step height measurement into the swelling at peak dpa (**44**). Step height measurements are particularly useful for determining relative swellings of various specimens.

18. Reporting of Results

18.1 *General Description of Microstructure*—The general appearance of the microstructure of the irradiated specimens should be reported in terms of phases, precipitates, dislocations, and voids. These features should be qualitatively compared with the preirradiation microstructure. The homogeneity of the spatial distribution of voids and dislocations should be noted. In particular, the association of voids with precipitates, void agglomeration, and the nature of the microstructure in the vicinity of grain boundaries should be reported.

18.2 *Quantitative Description of Microstructure:*

18.2.1 *Dislocations and Precipitates*—The dislocation density (m^{-2}), dislocation loop density (m^{-3}), and precipitate density (m^{-3}) should be determined. Several methods are described in the literature (**45**, **46**).

18.2.2 *Voids*—The following information should be reported for each value of the damage along with estimated uncertainties:

18.2.2.1 Void density,

18.2.2.2 Distribution of voids by diameter,

18.2.2.3 Average void diameter,

18.2.2.4 Swelling as % $\Delta V/V_0$,

18.2.2.5 Number and size of volumes sampled,

18.2.2.6 Number of voids counted in each volume, and

18.2.2.7 Total number of voids counted.

18.3 *Damage and Damage Rate*—The damage should be reported as dpa, displacements per atom, and the damage rate should be reported as dpa/s and computed by simply dividing the damage by the duration of the particle irradiation. The method and parameters used to obtain dpa from ion fluence should be reported (**36**).

19. Correlation

19.1 The correlation of the results of a charged particle irradiation with the results of a neutron irradiation can range from primarily empirical to primarily mechanistic, that is, based on quantitative descriptions of the effects of the differences in the irradiation environments on the microstructural/microchemical evolution of the irradiated material. The latter type of correlation permits meaningful predictions of neutron irradiation effects from charged particle data. The irradiation environment is generally considered to be defined by the particle type, energy or spectrum of energies, flux (fluence rate), fluence, applied stress, and irradiation temperature. This definition must be expanded, however, to include another class of variables, namely, those connected with differences in the irradiation geometry and the dimensions of the irradiated volume. This practice is intended to point the way toward mechanistically based correlations. In the interest of brevity, the preponderance of the discussion is left to the references (particularly useful collections are Refs (**47-51**).

20. Evolution of Radiation-Induced Microstructure

20.1 *Role of Pre-existing Microstructure*—The microstructure of unirradiated metals contains a variety of components that can act as sinks for point defects, defect aggregates or solute atoms. These are grain boundaries, dislocations, stacking faults, twin boundaries, and second-phase precipitates. The preirradiation densities and character of these components are often changed extensively by irradiation.

20.2 *Radiation-Induced Microstructure:*

20.2.1 The production of point defects in large supersaturations not only leads to enhanced diffusion but also provides additional modes of material transport not available in thermal environments. This leads to changes in the stability and mobility of existing microstructure and allows the formation of new components such as stacking fault tetrahedra, cavities (both void and gas-filled), and interstitial loops. The loops provide the source of new dislocation line length. These radiation-produced microstructural components arise as a consequence of the different point defect capture efficiencies exhibited by all microstructural sinks (**52**, **53**).

20.2.2 In general, all radiation-produced microstructural components undergo two evolutionary stages, nucleation and growth. Each component is sensitive to a different balance of a large number of variables in each evolutionary stage, and each stage responds differently to changes in the displacement rate. Eventually a steady-state microstructure emerges during irradiation. The point defect fluxes appear to be the major determinant of this state (**53**). In metals of any complexity, however, the possibility exists that substantial alteration of the matrix and second phases will occur. In some cases the various phases are transformed, and in other cases the phases undergo alteration of their elemental composition. Although such changes may arise from the alteration of phase stability by the supersaturation of point defects, most transformations are the result of changes in elemental composition, caused by selective migration and precipitation of some elements at various microstructural sinks. The path by which the elemental segregation proceeds is quite dependent on displacement rate, preirradiation microstructure, and temperature history (**54**). Some of the irradiation produced phases cannot exist in the absence of irradiation and require a minimum displacement rate for stability.

20.3 *Role of Solute Interactions With Point Defects*—Both solute and solvent atoms of complex alloys have been found to interact with point defects in a manner very sensitive to temperature, defect flux and gradients, and currently existing microstructure. This leads to an evolution of matrix composition that interacts with the concurrent microstructural evolution. In some alloys both evolutions appear to proceed to a steady state (**54**, **55**).

21. Comparison of Charged Particle and Neutron Irradiations

21.1 *Displacement Production:*

21.1.1 A neutron or charged particle interacting with an atom (the primary recoil atom) of a material may displace the atom from its lattice site, thus initiating a localized damage state. This may vary from a single Frenkel pair of defects (vacant site plus interstitial atom) to a displacement cascade of thousands of defects pairs (**56**). Procedures for calculation of total atomic displacements are given in Section 13.

21.1.2 The spatial configuration of the defects introduced into the lattice has a significant effect on the net production of residual defects, especially at low irradiation temperatures. The spatial configuration depends on the energy of the primary displaced atoms, which depends in turn on the type and energy of the incident radiation, and on the type of matrix atoms into which the primary is introduced. There is no charged particle that produces a primary recoil spectrum that is identical to that produced by reactor neutrons although the correspondence can be close in certain cases. Light ions such as protons produce an excessively high proportion of low energy recoils, while self-ions of sufficient range produce recoil spectra with excessively high components of both low and high energy. Electron irradiations, as commonly performed at energies of a few megaelectron volts or less, are characterized by very low energy recoils.

21.2 *Composition Changes*—Neutron irradiations produce transmutations; charged-particle irradiations, for energies within the scope of this recommended practice, do not. Ion irradiations of alloys, on the other hand, can influence the microstructural evolution through many processes. These include vacancy and interstitial trapping with resulting increases in point defect recombination; segregation to sinks such as voids and dislocations with resulting changes in sink capture efficiencies for point defects; creation of a locally strained matrix resulting in possible changes in cascade morphology and the fraction of surviving residual defects; gettering or trapping of other impurities, such as gases, with the resulting mitigation of their effects; and alterations of the relative stability of phases in multiphase systems. The effects of alloying elements on swelling have received the most study (**57-62**). The effects on other properties, especially phase stability, are receiving increased attention.

21.2.1 *Non-Gaseous Transmutation Products*—Neutron irradiations generally produce some non-gaseous transmutation products. Investigation of their potential influence is required for each alloy and experiment.

21.2.2 *Gaseous Transmutation Products and Impurities:*

21.2.2.1 Helium and hydrogen produced in neutron environments are known to play a role in the development of radiation-induced microstructure and various radiation-induced property changes, even at concentrations in the parts per million range. Hence, the appropriate gas concentrations must be introduced into a simulation experiment. This can best be done by implanting gas ions simultaneously with the damaging particle, although pre-injection or intermittent injection are often employed. The role of these gases in microstructural development has been found to depend sensitively on the temperature, injection rate, and schedule of introduction (**63-65**). Therefore, the impact of nonsimultaneous injection on the correlation should be investigated. This issue is discussed further in Guide E942. The effects of helium have been incorporated in models of swelling (**53**, **66-69**).

21.2.2.2 Other sources of gaseous impurities exist. They can be inadvertently introduced during material fabrication, specimen preparation (for example, electropolishing), and ion bombardment. The temperature sensitivity of gas solubility may also be an issue since charged particle irradiations are generally performed at higher temperatures than neutron irradiations.

21.2.2.3 Gaseous impurities must be considered a significant variable. Therefore, impurity content should be controlled and measured.

21.2.3 *Implanted Ions:*

21.2.3.1 Materials irradiated with ions will generally experience a composition change where the ions come to rest. For example, carbon ion irradiations have been used to study swelling in steels (**70**, **71**). If the implanted element is sufficiently mobile, compositional changes will occur outside the implanted region as well. The effect of dissimilar ions on void swelling through changes in defect trapping has been incorporated in models (**72**).

21.2.3.2 When a specimen is bombarded with self-ions, the ion is injected as a self-interstitial that is indistinguishable from a displacement-produced interstitial. The latter, however, has a vacancy counterpart while the injected interstitial does not. Theoretical methods have been developed for evaluating the reduction in cavity volume caused by injected interstitials (**73-76**). The effect can be significant (**76**) and is largest at low irradiation temperatures and for intrinsically low-swelling materials where recombination is the dominant mode of loss of radiation-produced point defects.

21.2.3.3 The effect of injected interstitials can be minimized by extracting data at depths well removed from the projected range of the bombarding ions. When the effect of injected interstitals is large, the use of step-height measurements as a measure of swelling is precluded (**77**).

21.2.4 *Elemental Redistribution*—Irradiation generally leads to a redistribution of alloying elements and impurities caused by differences in the interaction of each species with the vacancy and interstitial fluxes (**48**, **78**, **79**). In addition to these effects, which occur even when material is uniformly irradiated, additional redistribution may be introduced during ion irradiation by the variation in damage rate along the particle path or by defect concentration gradients produced by free surfaces. Gradients in damage rate can be minimized if necessary by rocking the specimen during irradiation or by variably degrading the energy of the ion beam. Free surface effects are avoided by using sufficiently high ion energies. (See 21.5.)

21.3 *Irradiation Time History*—The neutron and charged-particle environments can each be characterized by the instantaneous displacement rate and its time dependence.

21.3.1 *Displacement Rate*—Although some simulation experiments have displacement rates similar to that of the neutron environment, the majority of them have displacement rates that are one to four orders of magnitude greater. In general, the temperature regime in which complex phenomena such as void swelling, irradiation creep, and solute segregation exist shifts upward with increasing displacement rate (**79**, **80**). Methods of estimating some temperature shifts have been developed, for example, the temperature shift of void swelling due to intrinsic point defects (**81-83**).

21.3.2 *Irradiation Schedule*—The neutron environment to be simulated may involve continuous or pulsed irradiation. The use in a simulation experiment of an irradiation schedule that differs from that of the neutron environment requires careful analysis, since significant differences in microstructural evolution have been observed in comparative pulsed and continuous ion irradiations (**84**, **85**). When simulating actual pulsed or cyclic irradiations, the waveform may have to be altered depending on the nature of the microstructural process under study and the displacement rate.

21.4 *External Stress*—In neutron irradiations, the evolution of microstructure has been shown to be sensitive to the application of external stresses (**86**, **87**). Corresponding stress states in charged-particle irradiations may be impossible to achieve.

21.5 *Spatial Variations*—The interpretation of experiments utilizing charged particles of low penetration is complicated by the presence of displacement gradients which are not typical of neutron environments. The damage region is also bounded generally by two free surfaces, or one free surface and a transition region between damaged and undamaged regions. Several studies (**88**, **89**) have shown that the presence of surface and gradient-related processes distort the depth-dependent profiles of various radiation-induced phenomena from those expected on the basis of displacement damage only. The influence of these factors can be minimized by judicious choice of the depths from which data are extracted.

21.5.1 *Free Surfaces:*

21.5.1.1 Free surfaces, acting as sinks for point defects, alter the point defect concentration profiles, the densities and configurations of various microstructural sinks as well as their rate of growth, and the kinetics of various phase transformations (**74**, **89**, **90**). (Grain boundaries may exhibit similar effects.) Surfaces also serve as sites for solute segregation and may allow entry of various elemental contaminants. The most serious influence of the surface is the depression of point defect concentrations. Since this effect increases as the temperature is increased, it may preclude an unperturbed measurement of the full temperature response of a given phenomenon using charged-particle irradiation.

21.5.1.2 The presence of surfaces must also be taken into account in neutron experiments where the phenomenon under study is sensitive to the local chemical environment (for example, sodium, fission gas, helium, or hydrogen charging). Since most charged particle studies are conducted in a vacuum, this aspect of the neutron experiment may be impossible to simulate.

21.5.2 *Displacement Gradients:*

21.5.2.1 Gradients arise not only from the presence of free surfaces but also from the strongly depth-dependent energy deposition of charged particles. Since a basic irradiation parameter is the displacement rate, care must be taken to extract data from portions of the charged particle range wherein the displacement rate does not vary strongly.

21.5.2.2 The presence of point defect gradients may introduce diffusion processes not present in relatively homogeneous neutron irradiations. For example, when the defect production rate exhibits a maximum with depth, diffusion of defects broadens and lowers the point defect concentrations compared to a bulk irradiation at the peak damage rate. The effects are most pronounced for high temperatures, low sink densities, and steep damage gradients (**74**). They are particularly significant in metals having elemental components of differing diffusivity and degree of interaction with point defects.

21.5.3 *Internal Stresses*—The presence of both free and constrained boundaries and displacement gradients in charged-particle irradiations lead to the buildup of internal stresses that are atypical of those found in neutron irradiations. These stresses, invariable compressive and anisotropic in the irradiated volume, can reach large values which are maintained throughout the irradiation (**91**). They tend to influence the rate of microstructural development, particularly those components such as Frank loops which dominate the incubation period. The balance of various microstructural components, such as Frank and diamond loops, can also be influenced by the stress state.

22. Experimental Correlation Programs

22.1 Several experimental programs have been conducted to assess the applicability of charged-particle simulation techniques and to seek simple correlation factors. These programs are reviewed in Refs (**77**, **80**, and **92-96**). The success of each program appears to be inversely proportional to the complexity of the microstructure involved, the degree of difference in the two irradiation environments, and the number of elemental components in the alloy studied. In relatively complex simulations, the ability to establish a correlation was influenced by the microstructural criteria employed, since all facets of the microstructure could not be simulated simultaneously.

22.2 *Correlations Based on Equivalent Damage Only*—It is sometimes possible to establish a correlation between the microstructural changes produced by two different bombarding particles in terms of equivalent displacement damage only. For such an approach to succeed there should not be large differences in displacement rates or primary recoil atom spectra and the choice of exposure parameter should be consistent with the measured property. For example, it has been found that the damage structures produced in copper by 16 MeV protons and 14 MeV neutrons, at a given particle fluence, are nearly identical when examined by electron microscopy (**97**). The corresponding displacement dose for the protons is twice that for the neutrons; therefore the damage microstructures appear to correlate with total fluence but not with total displacements. This may be illusory, however. Logan has shown that, above about 10 keV, the primary recoil spectra from the two particles are very similar (**98**). It is a low-energy component (below 10 keV) of the recoil spectrum of the 16-MeV protons that doubles the displacement dose relative to that of the neutrons. Yet this component may contribute little to the visible microstructure. Therefore, the more appropriate correlation parameter for the visible microstructure may be the displacements created by primary recoils of energy greater than 10 keV.

22.3 *Correlations Involving a Temperature Shift:*

22.3.1 *Pure Metals*—At temperatures where void swelling occur, several experiments have been conducted that involve a substantial difference in displacement rate (**70**, **73**, **99**). These experiments have shown that void swelling cannot be correlated well on the basis of a simple temperature shift due to the different sensitivity of nucleation and growth processes to changes in displacement rate. When care is taken to compensate for differences in sink strengths, the correlation of void swelling improves substantially. The influence of the surface in perturbing the correlation is still rather large, however (**89**, **93**).

22.3.2 *Simple and Complex Alloys*—Where complex evolutionary sequences are involved in alloys, it becomes increasingly difficult to define a set of criteria on which to base a meaningful correlation. The combined influence of different activation energies for various competing processes, and different elemental interactions with point defects, surface, and gradient-induced phenomena, frequently acts to preclude the definition of a set of criteria that is applicable to any other set of irradiation conditions. Several approaches have been employed to attempt to circumvent the difficulties involved; two are described below.

22.3.2.1 *Steady-State Swelling Criterion*—Since the incubation period of swelling has been found to be most sensitive to variables in the simulation process, some correlations have been based on steady-state swelling rates. For a given alloy, some success has been obtained in defining correlations that include contributions from spatial and rate-related variables and that also compensate for uncertainties in total displacements arising from the spatial configurations of defects (**93**). Unfortunately, these correlations do not appear to be applicable to other alloys, because each alloy responds differently to the set of irradiation variables. For example, phases produced in-reactor may not be reproduced in charged particle irradiations (**92**).

22.3.2.2 *Neutron Preconditioning*—In complex alloys, it was thought that a low exposure irradiation or "preconditioning" of specimens in the neutron environment to be simulated might allow a better correlation of the high exposure behavior. The idea was to nucleate the appropriate microstructural and microchemical evolutionary paths, then find the correct temperature shift to maintain the evolution at the higher displacement rate of the charged particle irradiation. This approach met with only very limited success. In many cases the neutron-produced phases were not stable at the higher displacement rates at any relevant temperature (**92**).

22.4 *Experimentally Determined Correlation Factors:*

22.4.1 The various intercorrelation experiments based on steady-state swelling rates per calculated dpa demonstrated that self-ions in the range from 2 to 50 MeV produce rates roughly comparable to or slightly lower than those produced by fast neutrons (correlation factor ≤ 1). Protons (0.2 to 1 MeV) yield correlation factors of 3 to 20 and 1.0-MeV electrons give 2 to 6. However, a recent investigation of this issue found contrary evidence (**100**); swelling in copper was highest with neutron irradiation, lower with protons, and lower still with electrons when the damage rate was the same for all three types of irradiation (~10^{-8} dpa/s).The factors for electrons are typically based on a displacement cross section of 40 barns per electron (for iron), although some experiments indicate that the appropriate cross section is 20 barns (**101**).

22.4.2 The temperature shift associated with these correlations is roughly 35°C per decade difference in effective displacement rate (defined as the calculated displacement rate times the correlation factor) (**93**).

22.4.3 The effect of rate-related temperature shifts for various microstructural components along an ion path has also been demonstrated experimentally (**102**).

22.5 *The Use of Simulation for Screening Experiments*—The use of charged-particle irradiations to screen alloys for relative behavior has met with limited success. While neutron and heavy ion irradiations of simple ternary alloys yield similar swelling results with composition changes (**57**), the relative swelling resistance of complex alloys varies widely in comparative neutron, heavy ion and electron irradiations (**103**).

22.6 *Summary of Intercorrelation Experiments*—Intercorrelation experiments have demonstrated that the variables in charged-particle irradiations that are not typical of the neutron environment perturb correlation efforts to varying degrees. The stress state typical of charged-particle experiments affects primarily the incubation period of void swelling and not the steady-state regime. Surface effects are most pronounced in pure metals in the incubation regime, but become increasingly important in all alloys at higher temperatures, lower displacement rates, and shorter charged particle ranges. The injected interstitial effect (see 21.2.3) is found to completely dominate the simulation when minor solute additions exert a strong influence on the alloy's response to radiation. Elemental segregation in response to surface proximity and displacement gradients is particularly severe in alloys in which solutes exert effects on microstructural evolution that are disproportionate to their concentrations.

23. Guidelines for Correlation of Two Irradiation Environments

23.1 Charged-particle irradiations are best suited to the study of specific mechanisms involved in microstructural evolution. Depending on the complexity and sensitivity of the phenomena under study, it may or may not be possible to relate the results quantitatively to a neutron irradiation environment. If such correlations can be defined they may not be applicable to other environments, alloys, or property changes.

23.2 An exposure parameter based on total displacements per atom (dpa) should be employed. This should be calculated in a consistent fashion for the different environments (**36**) (see Section 14).

23.3 The effect of irradiation on the property of interest should be modeled, at least phenomenologically, to try to identify significant variables.

23.4 The dominant damage production portion of the primary recoil spectrum should be identified. For example, low-energy recoils are more efficient at producing point defect fluxes, whereas high energy recoils are more efficient at producing defect clusters (**56**). If the primary recoil spectra associated with two irradiation environments differ widely in the significant energy regime, the effective damage generation and accumulation per dpa can be expected to be different.

23.5 If the effective damage rates are significantly different, an anticipated temperature shift should be estimated based on the thermally activated process, or processes, associated with the property of interest (**73**).

23.6 Differences in specimen material and preparation should be minimized, as should gradients, free-surface effects, and differences in stress states.

23.7 The effects of differences in transmutation production should be accounted for.

23.8 The effects of differences in irradiation schedule should be accounted for.

24. Keywords

24.1 accelerators; beam heating; charged particle irradiation; damage calculations; dosimetry; transmission electron microscopy; ion irradiation; metallography; microstructure; radiation damage correlation; radiation damage simulation; void swelling

APPENDIXES

(Nonmandatory Information)

X1. ENERGY DISSIPATION BY NEUTRONS AND CHARGED PARTICLES

X1.1 *Neutrons*—Neutrons lose energy through both elastic and nonelastic interactions with nuclei; the cross sections for these reactions are often very energy-dependent. The kinetic energy imparted to the nucleus is shared by the atom as a whole (for the range of energies of interest) and is usually sufficient to displace the atom from its lattice site. An in-reactor specimen is generally assumed to be irradiated isotropically by neutrons that have an energy spectrum characteristic of the particular location in the reactor. The directional nature of accelerator-produced neutrons, on the other hand, may be significant.

X1.2 *Ions:*

X1.2.1 Ions traversing solids lose energy primarily through the interaction of their electric fields with the fields of the charged particles (electrons and nuclei) of the solid. Interactions with electrons raise them to excited states or cause them to be freed from their atoms (ionization). The major energy-dissipating interactions with nuclei are elastic collisions. The division into electronic and nuclear "stopping powers" is somewhat artificial but useful.

X1.2.2 An ion moving through a solid is continually undergoing capture and loss of electrons. The steady-state charge depends on the velocity and atomic number of the ion. For light ions the departure of the effective charge from Z, the atomic number, is simply a correction to be applied at energies less than a few tenths MeV. For heavy ions in the energy range considered here, the charge state is more critical but it will be unnecessary to consider it explicitly.

X1.2.3 The apparent charge of the atoms of the medium, and hence the type of interaction, depends also on the energy of the ion. A very energetic ion can penetrate the electron cloud (giving up energy to the electrons as it does so) and be coulomb-scattered by the full nuclear charge. At lower energies, penetration is incomplete and the charge of the nucleus is partially screened by the atomic electrons. At sufficiently low energies, screening will be essentially complete, and the collisions are between neutral atoms.

X1.2.4 Electronic stopping dominates except near the end of the particle range. For light ions this dominance is maintained down to energies of several tenths of keV. The relatively infrequent nuclear interactions are with the unscreened nuclear charge, simply described by Rutherford, the scattering cross section, and the nuclear potential. Other interactions are possible but are not significant energy loss mechanisms (see 14.4.4).

X1.2.5 Heavy ions at the energies of interest, particularly the low MeV range, are partially ionized and interact with heavily-screened nuclei. Lindhard et al (**104**) have derived a cross section to describe ion-ion elastic scattering in this range, as well as an expression for the electronic stopping power (**24**). Furthermore, they have presented a very convenient statistical theory of the partition between electronic and nuclear interactions of the total energy dissipated by an ion in slowing down in a solid (**15**). A basic assumption is that only the nuclear component, that is, the total kinetic energy minus that given up to electrons, can cause lattice damage.

X1.3 *Electrons*—Electrons lose energy principally through ionizing collisions with atomic electrons and through radiation emitted as a result of collisions with nuclei (bremsstrahlung). The former mechanism completely dominates at energies below a few tenths MeV; however, at higher energies the losses from the two mechanisms are comparable (see 13.4.2).

X2. DISPLACEMENT CALCULATIONS FOR ELECTRONS

X2.1 A general expression for the displacement cross section for an electron of energy, *E*, can be written in terms of an unprimed coordinate system taken with respect to the direction of the electron beam, *k*, and a primed coordinate system fixed in the crystal:

$$\sigma_d\left(E,\bar{k}\right) = \int_0^{O_{max}} \int_0^{2\pi} Q(\theta,\varphi,\theta',\varphi') \qquad \text{(X2.1)}$$

$$\cdot(\mathrm{d}\sigma/\mathrm{d}\theta)[\theta(T),\, E]N_d(T)\mathrm{d}\varphi\mathrm{d}\theta$$

where:

Q = 1 $T \geq T_d(\theta', \varphi')$,
= 0 $T < T_d(\theta', \varphi')$,
θ = $\cos^{-1} (T/T_{\max})^{1/2}$,
$\theta_{\max}$ = $\cos^{-1} (T_d/T_{\max})^{1/2}$,
$T_{\max}$ = 2148. $E(E + 1.022)/$(atomic weight), and
$N_d(T)$ is given by Eq 20.

X2.2 The most widely used scattering cross section, due to McKinley and Feshbach (**105**), is an approximation to the rigorous but unwieldy theory due to Mott (Note X2.1) and is most accurate for $Z <\sim 27$. The differential form of the McKinley-Feshbach cross section is:

$$\mathrm{d}\sigma/\mathrm{d}\theta = K[1 - \beta^2 \cos^2 \theta \qquad \text{(X2.2)}$$

$$+\pi\alpha\beta\cos\theta(1 - \cos\theta)(\sin\theta/\cos^3\theta)$$

where:

K = $(0.25\, Z^2/\pi)[(1 - \beta^2)/\beta^4]$,
α = $Z/137$, and
β = velocity of electron/velocity of light or
β^2 = $[E(E + 1.022)/(E + 0.511)^2]$ when E is in MeV.

The dependence of the displacement threshold energy on the direction of ejection is represented by $T_d(\theta',\varphi')$ and is incorporated in the displacement cross section through the function Q.

NOTE X2.1—The differences between McKinley-Feshbach and Mott cross sections can be found in the report by Oen (**31**).

X2.3 For irradiation of a polycrystalline specimen, the displacement probability is given by:

$$P(T) = (1/4\pi) \int_0^{\pi} \int_0^{2\pi} Q\,(\theta',\varphi')\cdot \sin\theta'\, \mathrm{d}\varphi'\, \mathrm{d}\theta' \qquad \text{(X2.3)}$$

Eq X2.1 can then be expressed in terms of $T_r = \mathrm{T}/\mathrm{T}_{\max}$:

$$\sigma_d\,(E) = \pi\, K\int_{T_{d,\min}}^{T_{\max}} N_d^{(E)}\,(T)\cdot P(T)\cdot[1 - \beta^2\, T_r \qquad \text{(X2.4)}$$

$$+ \alpha\,\beta\,\pi\,\sqrt{T_r}\;\left(1 - \sqrt{Tr}\right)](\mathrm{d}T_r/T_r^{\,2})$$

Comprehensive measurements of $T_d(\theta, \varphi)$ have been made only for Cu and Pt (**106**) and Ta (**107**), and then measurements were made at liquid helium temperatures so they are not directly applicable to irradiation at elevated temperatures. The results for Cu and Ta are not inconsistent with the effective T_d values suggested in Table 1 (**108, 109**).

X2.4 Computer experiments can, in principle, be used to determine $T_d(\theta, \varphi)$ (**32, 110-113**). Beeler's (**113**) results for γ-iron differ somewhat from the measurements of copper (**105**); they appear to be reasonably consistent, however, with the value of 40 eV suggested for T_d of iron and nickel alloys.

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