Designation: E 521 – 96 (Reapproved 2003)

Standard Practice for
Neutron Radiation Damage Simulation by Charged-Particle Irradiation[1]

This standard is issued under the fixed designation E 521; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This practice is intended to provide the nuclear research community with recommended procedures for the simulation of neutron radiation damage by charged-particle 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. It is generally confined to studies of microstructural and microchemical changes carried out with 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 The word simulation is used here in a broad sense to imply an approximation of the relevant neutron irradiation environment. 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:

	Section
Apparatus	4
Specimen Preparation	5–10
Irradiation Techniques (including Helium Injection)	11–12
Damage Calculations	13
Postirradiation Examination	14-16
Reporting of Results	17
Correlation and Interpretation	18-22

1.4 *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:*
C 859 Terminology Relating to Nuclear Materials[2]
E 798 Practice for Conducting Irradiations at Accelerator-Based Neutron Sources[3]
E 821 Practice for Measurement of Mechanical Properties During Charged-Particle Irradiation[2]
E 910 Test Method for Application and Analysis of Helium Accumulation Fluence Monitors for Reactor Vessel Surveillance, E706 (IIIC)[2]
E 942 Guide for Simulation of Helium Effects in Irradiated Metals[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*
3.1.1 Descriptions of relevant terms are found in Terminology C 859 and Terminology E 170.
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

---

[1] This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.08 on Procedures for Neutron Radiation Damage Simulation.
Current edition approved Jan. 10, 1996. Published March 1996. Originally published as E 521 – 76. Last previous edition E 521 – 89.

[2] *Annual Book of ASTM Standards*, Vol 12.01.
[3] *Annual Book of ASTM Standards*, Vol 12.02.

transfer in a single encounter must exceed the energy required to displace an atom from its lattice cite.

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:Symbols:*

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.

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

σ_{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 in not-yet-existing environments, 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 equipment.

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 E 821.

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, 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 commercially 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 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 form 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 E 942.

12.1.2 The influence of implantation temperature on helium distribution (that is, dispersed atomistically, 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 E 910 and Guide E 942 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^{18} 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 $\pm$ 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 as small as practical relative to nonbeam heating to minimize temperature fluctuations of the specimen due to fluctuations in beam flux 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*:

13.6.1 The particle flux 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·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. 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 (**1-5**).[4]

14.3.1.2 Although the work by Janni (**4**) appears to be the most comprehensive one for protons, experimental range data (**6**) 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 (**2**) with these data, it was recommended (**7**) 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 (**5**). 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 (**1**), and a more recent tabulation of range distributions and stopping powers is contained in a series of books edited by Ziegler and co-workers (**5**). Note that the ranges in Ref (**1**) (actually path lengths) have been corrected for nuclear stopping, whereas their tabulated stopping powers are for electronic stopping only.

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 (**8**).

14.3.2.4 Ranges can be computed with a code developed by Johnson and Gibbons (**9**). It is included as a sub-routine in the E-DEP-1 Code (see 13.5.3). It permits evaluations of projected ranges and range straggling as well.

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:

$$dE/dx\,|_{ion} \, \alpha \, NZ \tag{3}$$

$$dE/dx\,|_{rad} \, \alpha \, NZ^{2}\, E \tag{4}$$

for $E > 1$ MeV. Hence, given values for some reference material, energy dissipation for any other material can be

---

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

estimated. A convenient reference material is lead, in which both mechanisms contribute approximately equally at 10 MeV:

$$dE/dx \,|_{ion} \cong dE/dx \,|_{rad}$$
$$\cong 16 \text{ MeV/cm}$$
$$\cdot(\text{or } 1.6 \text{ keV/}\mu\text{m}) \ 10 \text{ MeV in Pb} \tag{5}$$

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

$$dE/dx \,|_{ion} \cong 0.357 \ p_0 Z_2/A_2 \text{keV/}\mu\text{m} \tag{6}$$

or:

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

$$dE/dx \,|_{rad} \cong 0.000435 \ E(\text{MeV}) \ p_0 Z_2{}^2/A_2 \text{keV/}\mu\text{m}$$

or:

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

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

$$dE/dx \,|_{ion} \cong 13 \text{ MeV/cm}$$

and:

$$dE/dx \,|_{rad} \cong 4 \text{ MeV/cm}$$

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 (7,10,11) is due to Lindhard et al (12); the expression for the damage energy T_{dam} lost by a knock-on of initial kinetic energy T is:

$$T_{dam} = T[1 + kg(\epsilon)]^{-1}$$
$$k = 0.1337 \ Z_1{}^{2/3}/A_1{}^{1/2}$$
$$= T/(0.08693 \ Z_1{}^{7/3}) \tag{8}$$

Following Robinson and coworkers (13, 14):

$$(9) \quad g(\epsilon) = \epsilon + 0.40244\epsilon^{[3/4]}P$$
$$+ \ 3.4008\epsilon^{1/6}$$

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

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

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 (13). 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 $25 \cdot Z^{4/3} \cdot A_1$ (in keV) (13).

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 (15), 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 (16-18); the result is a cross section $d\sigma(T,E)$ 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:

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

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:

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

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, T_d, is an effective displacement energy. So long as E exceeds several keV, T_d can be taken as 0 and σ_{de} is independent of T_d.

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

$$\text{depa/s} = \int_0^\infty \phi(E)\sigma_{de}(E)dE \tag{14}$$

This can be converted to damage energy per cubic centimetre per second by multiplying by N, the atom density. The cumulative damage energy density is obtained by integrating over the irradiation time. There is some error incurred in using Eq 12 and Eq 10 with the lower limit of the integral set at the displacement threshold due to the neglect of inelastic energy losses. Robinson and Oen have discussed this in detail and provide an expression for a simple correction factor (19).

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 simply used computer code, E-DEP-1 (20), has been developed and is recommended for calculating damage energy versus depth distributions for heavy ions. It makes the simplifying assumption of approximating energy straggling by using the range straggling theory of Lindhard et al (21). 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 (22) has performed computer experiments and Winterbon (23) 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. A Monte Carlo code such as TRIM (24,25) can also be used to perform these calculations. The use of TRIM permits more sophisticated analyses to be performed than does EDEP-1. TRIM 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 % (7). Nearer the specimen surface where the gradient and damage energy is less, the uncertainty is perhaps 20 %. 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 (21), or reading in some other value. k is the proportionality factor between the electronic stopping power and the ion velocity. Lindhard et al gives the approximate expression:

$$k = 0.0793 \, Z_1^{1/6} (Z_1 Z_2/Z)^{1/2} A_2/A_0^{\frac{3}{2}} \qquad (15)$$

in which:

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

It is suggested that better k values may be determined directly from the tabulated stopping powers of Northcliffe and Schilling (1).

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 number of knock-ons in the interval dT at knock-on energy T that is produced by an ion of energy E_x:

$$d\sigma_R(T,E_x) = (B\gamma^2/E_x)(dT/T^2) \qquad (17)$$

where:

B = $4\pi a_0^2 E_R^2(A_1/A_2)Z_1^2 Z_2^2$,
$\gamma_1 Z_1$ = 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 (26) 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, $y = (4.63/Z_1^{2/3}) [E_x(\text{MeV})/A_1]^{1/2}$. The damage energy cross section is given by integrating over the product of the number of events producing a knock-on of energy T [$d\sigma_R(T,E_x)$] and the damage energy associated with the knock-on, T_{dam}:

$$\sigma_{de}(E_x) = (B\gamma^2/E_x) \int_{Tm}^{Td} T_{\text{dam}} \, (dT/T^2) \qquad (18)$$

The lower limit of the integral is the mean energy required to displace an atom and the upper limit is the maximum possible energy transferred to an atom given by:

$$T_m = 4A_1 A_2/(A_1 + A_2)^2 E_x. \qquad (19)$$

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 $\phi t N \sigma_{de}$.*

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 (27) 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 (28) as $T_{\text{dam}} \cong 2 \, T_d \sigma_d$, 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 has been adopted by both the IAEA (10) and the USERDA (7) (for iron, nickel, and their alloys):

$$N_d = 0 \qquad\qquad\qquad T < T_d \qquad\qquad (20)$$
$$N_d = 1 \qquad\qquad\qquad T_d \leq T < 2T_d/\beta$$
$$N_d = \beta T_{dam}/2T_d/\beta \qquad T \geq 2T_d/\beta$$

The previously recommended values for iron, steel, and nickel-base alloys are $\beta = 0.8$ and $T_d = 40$ eV, or $N_d = 10\ T_{dam}$, 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 16 is recommended.

14.5.1.2 The actual displacement energy depends on the direction of ejection of the atom (29) (see Appendix X1). The value of T_d used in Eq 16 should represent an average overall ejection direction. Sufficient data to permit calculation of T_d exist for only a few metals. Furthermore, it is not clear that a simple unweighted average is appropriate because of the dominant role played by focused collision sequences. 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 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 recent molecular dynamics simulations that have investigated the directional dependence of the displacement threshold in a number of materials (29). 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.

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.

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 12. 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 $N_d(T_{dam})$ 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 12 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 12 and Eq 16 are consistent with current practice in Europe for calculating displacement 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 12 and Eq 16 are equivalent, are not identical to calculations using the data in Ref (30). 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 (30).

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

$$\bar{\sigma}_d = \int_0^\infty \sigma_d(E)\, \phi(E)\, dE / \int_0^\infty (E)\phi dE \qquad (22)$$

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 Code (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.

14.5.4 *Light Ions*—The calculation of the damage energy cross section in Eq 14 of 14.4.4 is easily modified to give a displacement cross section by substituting N_d from Eq 16 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 some-

where in the range from 700 to 2000 eV. Using beam currents of approximately 1 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.

15.2.3 *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