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

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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

[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 $-\mathrm{d}E/\mathrm{d}x$.

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 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 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.