Designation: E821 – 16 (Reapproved 2023)

Standard Practice for Measurement of Mechanical Properties During Charged-Particle Irradiation[1]

This standard is issued under the fixed designation E821; 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.

PART I—EXPERIMENTAL PROCEDURE

1. Scope

1.1 This practice covers the performance of mechanical tests on materials being irradiated with charged particles. These tests are designed to provide an understanding of the effects of neutron irradiation on the mechanical behavior of materials. Practices are described that govern the test material, the particle beam, the experimental technique, and the damage calculations. Reference should be made to other ASTM standards, especially Practice E521. Procedures are described that are applicable to creep and creep rupture tests made in tension and torsion test modes.[2]

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E521 Practice for Investigating the Effects of Neutron Radiation Damage Using Charged-Particle Irradiation

3. Terminology

3.1 *Definitions:*

3.1.1 Descriptions of relevant terms are found in Terminology E170.

4. Specimen Characterization

4.1 *Source Material Characterization:*

4.1.1 The source of the material shall be identified. The chemical composition of the source material, as supplied by the vendor or of independent determination, or both, shall be stated. The analysis shall state the quantity of trace impurities. The material, heat, lot, or batch, etc., number shall be stated for commercial material. The analytical technique and compositional uncertainties should be stated.

4.1.2 The material form and history supplied by the vendor shall be stated. The history shall include the deformation process (rolling, swaging, etc.), rate, temperature, and total extent of deformation (given as strain components or geometrical shape changes). The use of intermediate anneals during processing shall be described, including temperature, time, environment, and cooling rate.

4.2 *Specimen Preparation and Evaluation:*

4.2.1 The properties of the test specimen shall represent the properties of bulk material. Since thin specimens usually will be experimentally desirable, a specimen thickness that yields bulk properties or information relatable to bulk properties should be selected. This can be approached through either of

[1] This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.02 on Behavior and Use of Nuclear Structural Materials.

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[2] These practices can be expanded to include mechanical tests other than those specified as such experiments are proposed to Subcommittee E10.02.

[3] 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.

two techniques: (*1*) where the test specimen properties exactly equal bulk material properties; and (*2*) where the test specimen properties are directly relatable to bulk properties in terms of deformation mechanisms, but a size effect (surface, texture, etc.) is present. For the latter case, the experimental justification shall be reported.

4.2.2 The specimen shape and nominal dimensions shall be stated and illustrated by a drawing. Deviations from ASTM standards shall be stated. The dimensional measurement techniques and the experimental uncertainty of each shall be stated. The method of specimen preparation, such as milling, grinding, etc., shall be stated. The degree of straightness, flatness, surface condition, edges, fillets, etc., shall be described. The method of gripping the specimen during the test shall be stated and, preferably, illustrated by a drawing.

4.2.3 The heat treatment conditions such as time, temperature, atmosphere, cooling rate, etc., shall be stated. Because of the small specimen dimensions, it is essential to anneal in a non-contaminating environment. Reanalysis for O, N, C, and other elements that are likely to change in concentration during heat treatment is recommended.

4.2.4 Special care shall be exercised during specimen preparation to minimize surface contamination and irregularities because of the possible effect the surface can have on the flow properties of small specimens. Visible surface contamination during heat treatment shall be reported as a discoloration or, preferably, characterized using surface analysis technique. It is recommended that surface roughness be characterized.

4.2.5 The pre-irradiation microstructure shall be thoroughly evaluated and reported, including grain size, grain shape, crystallographic texture, dislocation density and morphology, precipitate size, density, type, and any other microstructural features considered significant. When reporting TEM results, the foil normal and diffracting conditions shall be stated. The specimen preparation steps for optical and transmission electron microscopy shall be stated.

4.2.6 The pre-irradiation mechanical properties shall be measured and reported to determine deviations from bulk behavior and to determine baseline properties for irradiation measurements. It is recommended that creep rates be measured for each specimen before and after irradiation. The thermal creep rate shall be obtained under conditions as close as possible to those existing during irradiation. The temperature, strain rate, atmosphere, etc., shall be stated.

4.2.7 It is recommended that other material properties including microhardness, resistivity ratio, and density be measured and reported to improve interlaboratory comparison.

4.3 *Irradiation Preconditioning:*

4.3.1 Frequently the experimental step preceding charged-particle irradiation will involve neutron irradiation or helium implantation. This section contains procedures that characterize the environment and the effects of this irradiation preconditioning. For reactor irradiations the reactor, location in reactor, neutron flux (fluence rate), flux history and spectrum, temperature, environment, and stress shall be reported. The methods of determining these quantities shall also be reported. The displacement rate (dpa/*s*) and total displacement (dpa) shall be calculated; see Practice E521 for directions. For ex-reactor neutron irradiation the accelerator, neutron flux and spectrum, temperature, environment, and stress shall be stated, including descriptions of the measurement techniques. The dpa/*s* and dpa should be calculated (see Sections 7 – 10). For helium implantation using an accelerator, the accelerator, beam energy and current density, beam uniformity, degrader system, temperature, environment, stress, helium content, and helium measurement technique and any post-implantation annealing shall be stated. The helium distribution shall be calculated as shall the resulting dpa (or shown to be negligible); see Sections 7 and 8 and Practice E521 for assistance. If another helium implantation technique is used, a description shall be given of the technique. It is recommended that chemical analysis follow any of the above preconditioning procedures.

4.3.2 The microstructure of irradiation preconditioned material shall be characterized with respect to dislocation loop size and density, total dislocation density, voids, and any microstructural changes from the unirradiated condition. Specimen density changes or dimensional changes shall be reported. It is recommended that changes in hardness or tensile strength, or both, be reported. Furthermore, any change in surface condition, including coloration, shall be reported.

4.4 *Analysis After Charged-Particle Irradiation:*

4.4.1 The physical, mechanical, and chemical properties of the specimen should be characterized prior to irradiation and any irradiation-induced changes reported. Practice E521 provides information on post-irradiation specimen preparation and examination.

4.4.2 After charged-particle irradiation, the specimen dimensions and density shall be measured. The microstructure and surface conditions shall be reexamined, with changes being reported. Chemical analysis for those elements likely to change during the mechanical test (O, C, N, H) shall be performed on the test specimen or on a dummy specimen held under conditions closely approximating those during irradiation. It is recommended that changes in hardness, tensile strength, or creep strength, or both, be measured and reported.

5. Particle Beam Characterization

5.1 *Beam Composition and Energy:*

5.1.1 Most accelerator installations include a calibrated magnetic analysis system which ensures beam purity and provides measurement and control of the energy and energy spread, both of which should be reported. A possible exception will occur if analog beams are accelerated. For example, a cyclotron can produce simultaneous beams of $^{16}O^{4+}(Z/A = ¼)$ and $^{12}C^{3+}(Z/A = ¼)$ at different energies ($E + E_oZ^2/A$) which cannot easily be separated magnetically or electrostatically. This situation, normally only significant for heavy ion beams, can be avoided by judicious choice of charge state and energy. For Van de Graaff accelerators analog beams of light ions, such as D^+ and He^{++}, can be generated, and under certain circumstances involving two-stage acceleration and further ionization (for example, $He^+ \rightarrow 5$ MeV $He^+ \rightarrow 5$ MeV He^{++}), beams of impurity ions can be produced that may not be easily separated from the primary beam (for example, 5 MeV H^+).

5.1.2 For most cases, ion sources are sufficiently pure to remove any concern of significant beam impurity, but this

problem should be considered. Beam energy attenuation and changes in the divergence of the beam passing through windows and any gaseous medium shall be estimated and reported.

5.2 *Spatial Variation in Beam Intensity:*

5.2.1 The quantity of interest is beam intensity/unit area at the specimen. It is usually desirable to produce a uniform beam density over the specimen area so that this quantity can be inferred from a measurement of the total beam intensity and area.

5.2.2 Total beam intensity should be measured using a Faraday cup whenever possible; however, this may not be possible on a continuous basis during irradiation. The Faraday cup shall be evacuated to $P < 10^{-5}$ and shall be electron-suppressed; otherwise, spurious results may be generated. Various secondary beam monitors may then be used, such as ionization chambers, secondary emission monitors, transformers or other induction devices (for pulsed beams), beam scanners, or particles scattered from a foil. All such devices shall be calibrated through Faraday cup measurements or through activation analysis. These calibrations shall be reported.

5.2.3 Displacement rate gradients occur in charged-particle irradiation specimens in the *Z* (beam) direction because of changes in ion energy and, therefore, displacement cross section with penetration (see 10.5.1), and in the *X* and *Y* (lateral and longitudinal specimen axes, respectively) directions because of spatial variations in beam intensity.

NOTE 1—Nonuniform specimen cross section may give rise to displacement rate variations in the *x*- and *y*-directions, even under a spatially uniform beam.

5.2.3.1 Displacement rate ratios of 1.2 to 2.5 (ratio of displacement rate at exit surface to rate at entrance surface of specimen in the *Z*-direction) are common, but it is recommended that this ratio be minimized. In the case of foil specimens it is also recommended that the variations in beam intensity in the *X*-direction be minimized, since a gradient in this direction will affect both the temperature and the creep compliance so as to maximize the stress gradient from specimen center to edge.

5.2.4 The beam may be rastered over the specimen to improve uniformity. The frequency of rastering shall be reported. The beam profile shall be measured regularly during the irradiation experiments, if possible. If this is not possible, some secondary measurement, such as temperature gradient, should be made. Analysis of the variation in specimen activity along the gauge section can provide an integrated average of the spatial variation in beam intensity; this is recommended.

5.3 *Time Variation in Intensity:*

5.3.1 Accelerator beams often have a built-in time structure which must be accepted; this should be reported. The history of beam interruptions due to occasional electrical breakdown shall be reported. The long-term stability of beam focusing and directing equipment shall be considered. If the beam spot is rastered to produce a uniform intensity profile, a further time dependence will be introduced, depending on the frequency and amplitude of the scan, and the size of the raw beam spot; this should be reported. When scanning a pulsed beam at a subharmonic of its natural frequency, it should be noted that the beam spot will strike the specimen at discrete locations, rather than be distributed continuously across the specimen. The raw beam spot must therefore be considerably larger than the distance between these locations or a very nonuniform intensity distribution will result. It is most desirable to use a continuous rather than rastered beam. If a rastered beam is used, the degree of defect annealing between pulses shall be considered.

6. Mechanical Testing Apparatus

6.1 *Strain Measurement:*

6.1.1 The strains measured during light ion irradiation tests, for measurement periods ~1 day and for conditions where the irradiation has a significant effect on the elongation rate, are very small (typically $\sim 10^{-3}$ to 10^{-5}). Therefore, the strain resolution normally required for continuous measurements is 1 to 10×10^{-6}. The strain resolution as well as displacement resolution shall be reported.

6.1.2 Normally for these experiments the limiting factor in strain measurement is not the resolution of the actual displacement measuring device (for example, LVDT, LVDC, strain gage, laser extensometer, etc.); it is the ability of the apparatus to transmit the displacement with fidelity. To minimize these displacement measurement errors, it is recommended that the temperature be monitored or controlled, or both, on each critical part of the apparatus and that thermal sensitivity experiments be performed; that is, a local temperature fluctuation should be imposed on individual elements of the strain-measuring system while the strain signal is monitored. It is recommended that the strain sensitivity to ambient temperature fluctuations be recorded. It is recommended that the strain sensitivity to vibrations and coolant flow rates be monitored and reported. The strain-measuring resolution, linearity, and reproducibility should be examined at several test temperatures on a regular basis using calibrated standards developed for such a purpose.

6.1.3 The sensitivity of the strain measurement shall be considered with respect to large magnetic or electrostatic fields, both of which may be present in these experiments. The effect of stray ion currents caused by secondary radiation should also be considered. The effect of lead length and shielding between the strain transducer(s) and the indicating device should be considered. Grounding may give rise to problems, especially with long lead lengths and associated ground potential differences.

6.1.4 The means of defining the deforming gage length of the specimen should be reported along with the accuracy of its measurement. Possible errors arising from deformation occurring outside the gauge section should be reported. It is also recommended that strain measurement errors caused by specimen bending be evaluated and reported.

6.2 *Load Application and Measurement:*

6.2.1 The requirements for load measurement in these experiments are much less stringent than those for strain measurements; accuracies of ≤1 % are recommended. Temperature, pressure, and vibration sensitivity measurements