



Designation: E821 – 16 (Reapproved 2023)

Standard Practice for Measurement of Mechanical Properties During Charged-Particle Irradiation¹

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 Recom-*

mendations 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 $^{16}\text{O}^{4+}$ ($Z/A = 1/4$) and $^{12}\text{C}^{3+}$ ($Z/A = 1/4$) at different energies ($E + E_0 Z^2/A$) 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, $\text{He}^+ \rightarrow 5 \text{ MeV He}^+ \rightarrow 5 \text{ MeV He}^{++}$), 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