Designation: E 821 – 96

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

This standard is issued under the fixed designation E 821; 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 simulate or provide understanding of, or both, the mechanical behavior of materials during exposure to neutron irradiation. 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 E 521. 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 *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:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry[3]

E 521 Practice for Neutron Radiation Damage Simulation by Charged-Particle Irradiation[3]

3. Terminology

3.1 *Definitions:*

3.1.1 Descriptions of relevant terms are found in Terminology E 170.

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 two techniques: (*1*) where the test specimen properties exactly equal bulk material properties; (*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

[1] This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.08 on Procedures for Neutron Radiation Damage Simulation.

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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 E10.08.

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

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 preirradiation 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 preirradiation 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 (see section 3.4 for more detail). 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, 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 E 521 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 E 521 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 E 521 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 analogue 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 analogue 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—Non-uniform 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 non-uniform 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 strainmeasuring 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 should be performed on the load measuring device. The load-measuring resolution, linearity, and reproducibility should be examined on a regular basis using calibrated standards.

6.2.2 The effect of secondary radiation, electric, or magnetic fields on the load transducer should be considered, along with lead length and shielding. Variations in ground potentials shall be considered.

6.2.3 Possible loading errors associated with misalignment shall be evaluated and reported. Frictional forces shall be measured where applicable, since friction anywhere in a mechanical load train may affect the strain measurement. The hysteresis in load application and measurement should be reported in relation to the strain measurement.

6.3 *Temperature Monitoring and Control*—For these experiments temperature monitoring and control capabilities may be the dominant factors that limit the overall accuracy and resolution of the primary strain measurement. For uniaxial tension the temperature dependence of the strain error arises from thermal expansion ($d\epsilon/dT \sim 10^{-5}$/K) and, to a lesser degree, from the temperature dependence of the modulus

($d\epsilon/dT \sim 10^{-7}$/K). For torsion experiments, the temperature dependent strain error is that of the modulus only. Primary emphasis will be given to uniaxial tension experiments, since strain resolution requirements are likely to limit the allowable temperature variations to less than 1 K over the desired temperature range (from room temperature to about 1000 K). The overall reproducibility from experiment to experiment may not be so stringent, however. If this reproducibility is 5 K or less, reasonably good agreement between thermal creep rates will be obtained, and very good agreement should be obtained on radiation-induced mechanical property changes.

6.3.1 *Temperature Monitoring*—This section is devoted to the description of techniques for detecting the absolute temperature of the specimen (accuracy) and for detecting changes in specimen temperature from some set point (resolution). Each of the following techniques may be important for either the accuracy or resolution of the temperature measurement, or both. In a given experiment, one technique may be utilized for estimating the overall specimen temperature to within several K; whereas, in another experiment the same technique may be used to resolve small (~0.1 K) temperature fluctuations (presumably in conjunction with the temperature control function). However, it should be kept in mind that beam heating at high beam currents can adversely affect the temperature resolution. It is recommended that direct monitoring of the specimen temperature be performed. If, however, an indirect monitoring technique is used (for example, a dummy specimen or an ambient heat sink temperature measurement is used) then it should be demonstrated that the factors controlling the heat transfer from the specimen to the point where temperature is monitored remain constant. For example, if heat transfer must occur through an oxide film on the specimen or, perhaps, on an adjacent heat sink, the stability of this film during an experiment should be evaluated. Three temperature-monitoring techniques have been applied to these experiments, (*1*) thermocouples, (*2*) infrared pyrometry, and (*3*) resistance thermometry. The method for absolute calibration of any of these techniques used shall be reported.

6.3.1.1 *Thermocouples*—There are several problems associated with the use of thermocouples applied directly to specimens for these experiments. The thin specimens normally used are subject to local perturbations in temperature through heat conduction from specimen to thermocouple wire. Thermal analysis shall be performed to determine the magnitude of temperature error associated with this thermal shunting. Another difficulty with applying the thermocouple directly to the specimen is the effect on the specimen material at the point where the thermocouple is welded. The "heat-affected'' zone shall be minimized, and the percent of the total cross section that is affected by the welding of two thermocouple wires shall be reported. Radiation can affect the performance of thermocouples. Radiation damage and, to a lesser degree, transmutation will affect thermocouple calibration. Therefore, it is recommended that thermocouples that are irradiated during an experiment should be calibrated before and after each experiment. Radiation can also affect the temperature of the thermocouple junction. Radiation heating shall be included in the thermal analysis mentioned above. Another possibly important effect of radiation is ionization events which can occur in the thermocouple wire, in its insulation, or in the medium surrounding a bare thermocouple wire. In all these cases spurious voltages or currents can give rise to errors (only, of course, when the two thermocouple wires are ionized to dissimilar degrees). Furthermore, thermocouples that are not directly in the beam, but that receive significant gamma radiation, will undergo ionization. These effects should be considered. Special problems can arise when split thermocouples are employed. For example, when an electrical heating current passes through the specimen, the output voltage of the thermocouple will reflect the IR drop between the two points of contact of the thermocouple wires. Caution shall be exercised in the use of split thermocouples for the following reasons: (*1*) the specimen, with its radiation-sensitive Seebeck coefficient, may undergo the equivalent of thermocouple decalibration, (*2*) split thermocouples can mask temperature spikes or hot spots between the wire contact points, and (*3*) the averaging of temperature gives rise to error, except under the ideal condition of linear variation in temperature between the contact points.

6.3.1.2 *Infrared Pyrometers*—The accuracy of infrared pyrometers is dependent upon several factors. First, the surface emissivity must remain constant. This shall be demonstrated in pre- and post-experimental evaluation. If specimen preoxidation is necessary for keeping the emissivity constant, it shall be demonstrated that thermal creep properties in the temperature and stress range of interest are not affected by the oxidation treatment. Second, since instruments will receive a significant level of gamma radiation during some experiments, these infrared pyrometers shall be regularly calibrated.

6.3.1.3 *Resistance Thermometry*—Resistance thermometry is potentially a very high accuracy approach to temperature measurement. A comprehensive reference on this subject is Ref **(1)**.[4] A major source of error is associated with a change in the intrinsic or low-temperature specimen resistance. This change results from microstructural changes caused by annealing or irradiation, or both. It must be demonstrated that the appropriate T versus R curve is known during the period measurements are taken. Also, compensation must be provided for resistance changes associated with specimen deformation. Specimen voltage leads present a problem area similar to that for thermocouples. Thermal shunting and specimen structural changes at the points of lead welding shall be minimized. Since resistance thermometry is an averaging technique, the temperature shall be monitored at several discreet points over the entire specimen to detect thermal spikes.

6.3.2 *Temperature Control*:

6.3.2.1 The types of temperature control may be grouped as direct or indirect methods. These terms refer to the specimen heating or cooling technique applied. An indirect technique is one in which the electrical control signal heats or cools (or changes the flow of) some intermediary substance which in turn changes the specimen temperature. It is recommended that an indirect control method not be used in conjunction with an indirect monitoring technique; see 6.3.1. If this is done, it shall

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