



Designation: **E531–76 (Reapproved 2007) E531 – 13**

Standard Practice for Surveillance Testing of High-Temperature Nuclear Component Materials¹

This standard is issued under the fixed designation E531; 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.

1. Scope

1.1 This practice covers procedures for specimen testing to establish changes occurring in the mechanical properties due to irradiation and thermal effects of nuclear component metallic materials where these materials are used for high temperature applications above 370°C (700°F).

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*²

[A370 Test Methods and Definitions for Mechanical Testing of Steel Products](#)

[E3 Guide for Preparation of Metallographic Specimens](#)

[E8E8/E8M Test Methods for Tension Testing of Metallic Materials](#)

[E21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials](#)

[E23 Test Methods for Notched Bar Impact Testing of Metallic Materials](#)

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

[E45 Test Methods for Determining the Inclusion Content of Steel](#)

[E112 Test Methods for Determining Average Grain Size](#)

[E139 Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials](#)

[E185 Practice for Design of Surveillance Programs for Light-Water Moderated Nuclear Power Reactor Vessels](#)

[E206 Definitions of Terms Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data; Replaced by E 1150 \(Withdrawn 1988\)](#)³

[E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques](#)

[E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness \$K_{Ic}\$ of Metallic Materials](#)

[E453 Practice for Examination of Fuel Element Cladding Including the Determination of the Mechanical Properties](#)

[E468 Practice for Presentation of Constant Amplitude Fatigue Test Results for Metallic Materials](#)

[E482 Guide for Application of Neutron Transport Methods for Reactor Vessel Surveillance, E706 \(IID\)](#)

[E606 Practice for Strain-Controlled Fatigue Testing](#)

[E647 Test Method for Measurement of Fatigue Crack Growth Rates](#)

[E844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E 706 \(IIC\)](#)

[E1823 Terminology Relating to Fatigue and Fracture Testing](#)

[E2006 Guide for Benchmark Testing of Light Water Reactor Calculations](#)

[E2215 Practice for Evaluation of Surveillance Capsules from Light-Water Moderated Nuclear Power Reactor Vessels](#)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *test specimen*—a coupon or a piece of metal cut from a larger metal piece which is then machined to final size for testing to determine physical or mechanical properties.

¹ This recommended 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.

Current edition approved June 1, 2007; Jan. 1, 2013. Published July 2007/January 2013. Originally approved in 1975. Last previous edition approved in 2004/2007 as E531–76(2001); E531–76(2007). DOI: 10.1520/E0531-76R07-10.1520/E0531-13.

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.

4. Significance and Use

4.1 The requirements contained herein can be used as a basis for establishing conditions for safe operation of critical components. The requirements provide for general plant assessment and verification that materials meet design criteria. The test specimens and procedures presented in this practice are for guidance when establishing a surveillance program.

4.2 This practice for high-temperature materials surveillance programs is used when nuclear reactor component materials are monitored by specimen testing. Periodic testing is performed through the service life of the components to assess changes in selected material properties that are caused by neutron irradiation and thermal effects. The properties are those used as design criteria for the respective nuclear components. The extent of material property change caused by neutron irradiation depends on the composition and structure of the initial material, its conditioning in component fabrication, as well as the nature of the irradiation exposure. The need for surveillance arises from a concern of specific material behavior under all irradiation conditions including spectrum and rate effects on material properties.

4. Description of Term

~~4.1 *test specimen*—a coupon or a piece of metal cut from a larger metal piece which is then formed to final size for testing to determine physical or mechanical properties.~~

5. Test Specimens

5.1 *Pre-Exposure Material Characterization*—It is important that test specimen materials be characterized prior to exposure and that the following should be considered as a minimum:

5.1.1 Process history, material designation, manufacturer, heat number, weld and fabrication procedures used, and heat treatment,

5.1.2 Original location and orientation in the parent material,

5.1.3 Specimen weight and dimensions,

5.1.4 Metallographic characteristics (grain size, microstructure, and inclusion content established in accordance with Test Methods [E45](#) and [E112](#)),

5.1.5 Chemical analysis results,

5.1.6 All specimens shall be taken from the specified location and orientation specified in Test Methods and Definitions [A370](#) and Test Methods [E8E8/E8M](#), and

5.1.7 Mechanical properties including yield strength, tensile strength, stress rupture life, creep strength, fatigue strength, and impact strength as a function of test temperature.

5.1.8 The information described in [5.1.1-5.1.7](#) should be reported in a single document.

5.2 *Post Exposure Material Characterization*—After exposure, the following should be reported:

5.2.1 Observations from visual examination,

5.2.2 Changes in specimen weight and dimensions,

5.2.3 Metallographic characteristics (grain size, microstructure, and inclusion content),

5.2.4 Results of chemical analysis,

5.2.5 Appropriate mechanical properties being ~~surveyed~~ assessed including considerations of changes in tensile strength, stress-to-rupture strength, creep strength, fatigue strength, impact strength (control tests are recommended to be performed simultaneously with the tests of exposed specimens to ensure that deviations in test results can be attributed to the exposed specimen's environment and not to variations in testing methods), and

5.2.6 Optional quantitative examination of surface chemistry and subsequent changes.

5.2.7 Exposed test specimens should be cleaned in accordance with accepted cleaning procedures. (Refer to Subcommittee G01.08 for practices for preparing, cleaning, and evaluating test specimens.)

5.3 *Specimen Preparation*—Test specimens shall be standard recommended specimens where possible as described in Test Methods [E8E8/E8M](#), [E21](#) and [E23](#) and Practice [E139](#). The use of the word *specimen* or words *test specimen* as used in this practice is described in Section [43](#).

5.3.1 The test area of a specimen (for example, Charpy notch, reduced section of a fatigue specimen) may be left unfinished if further environmental exposure prior to testing is anticipated.

5.3.2 *Size*—In general, due to the limited space available in surveillance capsules the smaller sizes of test specimens are recommended. Where it is not possible to use specimens of the recommended size, the least deviation possible from recommended sizes should be adhered to. Non-standard specimens shall be evaluated prior to use as surveillance specimens to ensure that test results from the use of non-standard specimens can be correlated with test results from specimens of recommended size. In the event that non-standard specimens are used for surveillance specimens, the archive, base line, and thermal control specimens shall be identical ~~with~~ to the surveillance specimens.

5.3.3 *Surface Condition*—Test specimens where surface condition is critical to the test results should not be finish machined in such critical areas (Charpy notch, fatigue specimen test area, surface of density change sample) until just prior to test. Specimens should be oversized to allow for removal of at least 0.1 mm (0.004 in.) of surface prior to test. Where possible, test specimens with

the exception of weight change specimens shall be encapsulated in an inert environment so as to determine only the effect of neutron irradiation and temperature on mechanical or physical properties. It is recognized the integrity of the encapsulation may be breached in some cases during long exposure and an allowance for final machining even of the encapsulated specimens should be considered. This will ensure a meaningful comparison between baseline and exposed specimens.

5.3.4 Number of Specimens—The number of specimens employed for mechanical property testing should be selected so as to include each critical component that varies significantly in composition, processing, or in exposure conditions from similar components. Specific recommendations as to the number of specimens will be found in the respective specimen sections. At least four sets of specimens shall be included in each surveillance program.

5.3.5 Material—Test specimens shall be taken from the material used in component fabrication. The material shall be processed at the same time as the component or processed in a fashion identical to the component ~~surveyed~~investigated. Weld and heat-affected zone test specimens shall be taken from equivalent material welded at the same time as the particular component or equivalent material welded with the same welding parameters. It is not necessary to include each heat or minor variation, but only to select those receiving the highest exposure or those previously found to be most sensitive to neutron irradiation and temperature or those that can restrict the operation of the reactor. Test specimens may be taken from components periodically removed from the reactor for other reasons. These specimens can be used to provide supplemental surveillance information. For this information to be meaningful a full characterization of the pre-exposure condition must be available along with measured exposure conditions.

5.4 Tension Test Specimens—The type and size of specimen to be used shall conform to the smaller sizes as recommended in Test Methods ~~E8E8/E8M~~ and ~~E21~~. Either threaded or button-head ends will be acceptable. For plate or sheet specimens, pin ends as described in Test Methods ~~E8E8/E8M~~ are recommended. The location and orientation of test specimens shall be as defined in Test Methods ~~E8E8/E8M~~ or Test Methods and Definitions ~~A370~~, or in Practice ~~E185~~. Both base metal and weld metal specimens will be taken. A set of tension specimens shall consist of three of each base metal and weld metal.

5.5 Creep and Stress-Rupture in Specimens—The type and size of specimen to be used shall be the same as those used for tension specimens except that button-head or pinned-end specimens are recommended for high-temperature testing. Practice ~~E453~~ describes the attention that must be paid to specimen alignment and dimensional tolerances. One set of tests shall be conducted at the operating temperature of the component of interest. A set shall comprise a minimum of six stress rupture tests at six different stress levels. The stress levels should be selected so that the time-to-rupture ranges from not less than 100 h to at least 3000 h. Creep strain measurements may be made if desired.

5.6 Low-Cycle Fatigue Specimens—For base metal the type and size of specimen to be used may be the “hourglass” type with threaded or button-head ends as shown in ~~Fig. 1~~. For weld metal specimens the uniform gage type may be used. Machining and polishing of the test specimens shall be performed with care so as to minimize the effects of specimen preparation on fatigue life. In the final stages of machining, material shall be removed in the radial thickness by amounts of 0.2 mm (0.008 in.) until 0.1 mm (0.004 in.) remains. After exposure the final 0.075 mm (0.003 in.) shall be removed by cylindrical grinding at no more than 0.005 mm (0.002 in.) per pass. The final 0.025 mm (0.001 in.) shall be buffed and finished with an 0.2 μ m R_a (8 μ m. AA) surface roughness. After polishing, all remaining grinding and polishing marks shall be longitudinal and any circumferential grinding marks ~~must~~shall be removed. The finished specimens shall be degreased in suitable solvent. Specimens to be exposed to liquid sodium shall not be degreased in halogenated solvents. If surface observations are to be made, the test specimen may be electropolished in accordance with Methods ~~E3~~. Test specimens that are susceptible to corrosion in room-temperature air shall be stored as soon as practicable after preparation in an inert dry gas or vacuum. A set of specimens shall consist of ten each of base metal and weld metal.

5.7 Swelling Specimens—The swelling specimens shall be right circular cylinders 5.0 mm (0.2 in.) diameter and 10.0 mm (0.4 in.) long. The sharp-cornered specimens shall be finished by turning or grinding and have a surface finish of 0.2 μ m R_a (8 μ m. AA). The specimens shall be degreased in suitable solvent and stored in an inert gas or vacuum.

5.8 Charpy Impact Specimens—The specimens to be used shall conform to the Charpy V-notch specimens recommended in Test Methods ~~E23~~. The notch shall not be ~~formed~~machined prior to exposure as a surveillance specimen. The location and orientation of test specimens shall be as defined in Practice ~~E185~~ and Test Methods and Definitions ~~A370~~. A set of specimens shall be made up of twelve each of base metal, heat-affected zone, and weld metal.

6. Irradiation Conditions

6.1 Introduction—The intent of the section on irradiation conditions is to provide guidance on how to place surveillance samples to obtain the desired irradiation conditions in terms of temperature, neutron flux, and neutron spectrum to ensure a realistic evaluation of the component that the surveillance specimen is representing.

6.2 Irradiation Temperature—It is very important that adequate consideration be given to test specimens to ensure that they experience the correct temperature during irradiation. Temperature must be controlled for the surveillance specimens to match as nearly as possible the temperature of the component being surveyed. When temperature variations arise because of separation of the surveillance specimens from the component, additional specimens should be included in other positions which will cover a temperature range including the component operating temperature. The irradiation temperature shall be given and the method for determination shall be documented.