Designation: E 531 – 76 (Reapproved 1996)$^{\epsilon 1}$

Standard Practice for Surveillance Testing of High-Temperature Nuclear Component Materials[1]

This standard is issued under the fixed designation E 531; 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.

ε[1] Note—Keywords were added editorially in August 1996.

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).

2. Referenced Documents

2.1 *ASTM Standards:*

A 370 Test Methods and Definitions for Mechanical Testing of Steel Products[2]
E 3 Methods of Preparation of Metallographic Specimens[3]
E 8 Test Methods for Tension Testing of Metallic Materials[3]
E 21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials[3]
E 23 Test Methods for Notched Bar Impact Testing of Metallic Materials[3]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]
E 45 Test Methods for Determining the Inclusion Content of Steel[3]
E 112 Test Methods for Determining the Average Grain Size[3]
E 139 Practice for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials[3]
E 184 Practice for Effects of High-Energy Neutron Radiation on the Mechanical Properties of Metallic Materials, E706 (IB)[5]
E 185 Practice for Conducting Surveillance Tests for Light Water-Cooled Nuclear Power Reactor Vessels, E706 (IF)[5]
E 206 Definitions of Terms Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data[6]
E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques[5]
E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials[3]
E 453 Practice for Examination of Fuel Element Cladding Including the Determination of the Mechanical Properties[5]
E 482 Guide for Application of Neutron Transport Methods for Reactor Vessel Surveillance, E706 (IID)[5]
E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E706 (IIC)[5]

3. Significance and Use

3.1 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.

3.2 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:

[1] This recommended practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.02 on Behavior and Use of Metallic Materials in Nuclear Systems.

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[2] *Annual Book of ASTM Standards*, Vol 01.03.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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

[6] Discontinued—see *1986 Annual Book of ASTM Standards*, Vol 03.01.

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 E 45 and E 112),

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 A 370 and Test Methods E 8, 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 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 E 8, E 21 and E 23 and Practice E 139. The use of the word *specimen* or words *test specimen* as used in this practice is described in Section 4.

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 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 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 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. 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 E 8 and E 21. Either threaded or button-head ends will be acceptable. For plate or sheet specimens, pin ends as described in Test Methods E 8 are recommended. The location and orientation of test specimens shall be as defined in Test Methods E 8 or Test Methods and Definitions A 370, or in Practice E 185. Both base metal and weld metal specimens will be taken. A set of tension specimens shall consist of three 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 E 453 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.

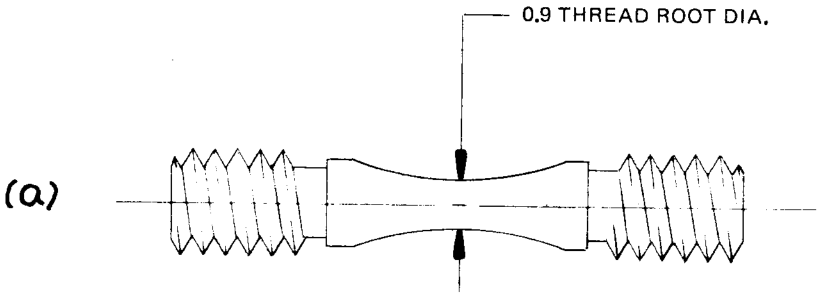
5.6 *Low-Cycle Fatigue Specimens*—For base metal the type and size of specimen to be used may be the "hourglass" type with threaded or button-head ends as shown in Fig. 1. For weld metal specimens the uniform gage type may be used. Machining and polishing of the test specimens shall be performed with 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 amounts of 0.2 mm until 0.1 mm remains. After exposure the final 0.075 mm shall be removed by cylindrical grinding at no more than 0.005 mm per pass. The final 0.025 mm shall be buffed and finished with an 0.2 µm R_a (8 µin. AA) surface roughness. After polishing, all remaining grinding and polishing marks shall be longitudinal and any circumferential grinding marks must 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 E 3. 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.

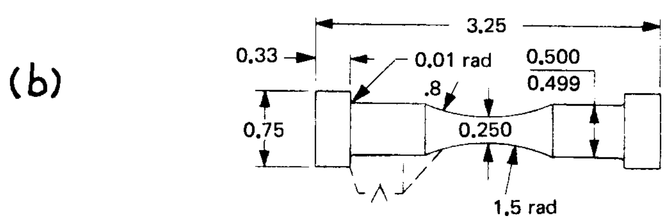
5.7 *Swelling Specimens*—The swelling specimens shall be right circular cylinders 5.0 mm diameter and 10.0 mm long. The sharp-cornered specimens shall be finished by turning or grinding and have a surface finish of 0.2 µm R_a (8 µin. AA). The specimens shall be degreased in suitable solvent and stored 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 E 23. The notch shall not be formed prior to exposure as a surveillance specimen. The location and orientation of test specimens shall be as defined in Practice E 185 and Test Methods and Definitions A 370. A set of specimens shall be made up of twelve each 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





NOTE 1—A = 0.2 µm R_a (8 min AA).

Metric Conversion

mm	in.
0.3	0.01
6.35	0.250
8.4	0.33
12.68	0.499
12.70	0.500
19.1	0.75
38.1	1.5
82.6	3.25

FIG. 1 Standard Hour-Glass Low Cycle Fatigue Specimen (Threaded Ends (*a*) and Button Head (*b*))

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.

6.3 *Flux and Spectrum*:

6.3.1 The size of certain reactor components may introduce irradiation conditions that cover a wide range of neutron fluxes and spectra. Accordingly, surveillance irradiations carried out at locations other than exactly in contact with the component of interest will require consideration of the change in neutron flux and spectrum between component and surveillance location. Frequently, surveillance specimens are placed such that the neutron flux is higher than at the component of interest; due consideration must then be given to changes in neutron energy spectrum when making such compromises.

6.3.2 Also of considerable importance during elevated temperature exposure are the effects of time-at-temperature. It should be recognized that the placement of surveillance specimens in a region of higher flux may change the total outcome for a given neutron fluence just because of the different time-temperature effect; that is, the higher flux level produces a shorter irradiation time, and precludes potential aging-type effects. In the case where surveillance specimens are placed in certain high neutron-flux regions so as to attain a specific fluence level more quickly than the component itself, an appropriate damage-equivalence factor or analysis scheme should be used in evaluating the test results from the samples for relating to the expected results from the component.

6.4 *Coolant Environment*:

6.4.1 The choice of environment for a surveillance specimen depends on the other reactor environments surrounding the specimen itself. If temperatures higher than provided by the surveillance environment are needed, these can be attained by using an inert gas blanket between the specimen and the capsule enclosure.

6.4.2 It is intended that specimens be evaluated solely for irradiation, time and temperature effects; therefore, other environmental influences shall be minimized or precluded if at all possible. However, if alternative reactor coolants or other environmental factors must be used, their effects upon the surveillance specimens should be carefully evaluated with respect to the test results and the conclusions drawn therefrom.

6.5 *Specimen Withdrawal Schedule*—The specimen withdrawal schedule shall be as specified in Practice E 185, Case B, based on the percentage of component life as specified in Table 1.

7. Measurement of Neutron Exposure

7.1 The neutron flux and neutron energy spectrum at the surveillance location shall be given as well as the method used for determination. All assumptions should be clearly stated, and all physical constants, such as cross sections, half lives, fission yields, etc., should be listed. The neutron flux level should be determined using more than one single flux detector material. It is recommended that a series of detectors be used for the fluence determination itself, and also for subsequent use in either verifying the results of reactor physics spectrum code calculations or in unfolding a neutron spectrum from the activation data itself.

7.2 Guidance for use of multiple-flux detectors in spectrum evaluation procedures is given in the Related Material section[7] under the title, "Discussion of Computer Codes for Determining Neutron Flux Spectra by Multiple Foil Measurements." A specific practice for neutron dosimetry in high-temperature reactor irradiations has not been written, but guidance is available from Guide E 482, which was written by Subcommittee E10.05 for light water power reactor irradiations. The selection of specific flux detector materials can be aided by reference to Guide E 844. Measurement techniques and general guidance are given in Practice E 261.

8. Swelling

8.1 *Introduction*—The purpose of the swelling surveillance would apply only when the design consideration requires that swelling be within a reasonable limit and design estimates require swelling surveillance for the component. The swelling could be evaluated from destructive examination of periodically removed components or in the case of more permanent components, the evaluation of a 5-g portion of a tension specimen or Charpy specimen related to the component. The sensitivity of irradiation induced swelling to irradiation temperature, flux, and spectrum would require very close attention to the irradiation conditions of the swelling specimen so that they approximate as closely as possible that of the component. If possible, the swelling specimen should be attached to the component; however, where the component is in a nonaccessible position, other positions could be considered.

8.2 *Swelling Evaluation Based on Dimensional Changes*—Dimensional changes including considerations for calibration, surface preparation, temperature, and lengths shall be in accordance with Section 7 of Practice E 453. Specified limiting values shall be in accordance with Practice E 29.

8.3 *Density Measurements to Evaluate Swelling*—Density measurements shall be conducted in accordance with Section 12 of Practice E 453.

9. Creep and Stress-Rupture Tests

9.1 *Introduction*—The intent of creep and stress-rupture tests is to provide supplemental data when significant materials properties changes are first indicated from short term mechanical properties tests on specimens removed from the reactor. The creep strain measurements are made to verify that the material meets the *design* criteria (if dimensional tolerances are critical).

[7] See *1974 Annual Book of ASTM Standards*, Part 45.

10. Fatigue Tests

10.1 *Introduction*—The intent of fatigue tests is to provide data from which the service life and safe operation of those critical components as defined by the designers can be monitored. The fatigue tests should be applied to specimens representative of the components. The specimens may be samples inserted in the reactor at startup or taken from components which have been in service and have been removed to ascertain the integrity of other remaining components for further service. (A standard is in preparation by ASTM Committee E–9.)

10.2 *Type of Test*:

10.2.1 *Axial or Bending Tests*—Axial strain controlled cyclic testing at temperatures corresponding to component operating temperatures is recommended for the low and axial push-pull or completely reversed bending are recommended in the intermediate and high life range (10^5 to 10^7 cycles).

10.2.2 *Fracture Mechanics Type Tests*—The use of fracture mechanics techniques in design and safety analyses provides a quantitative means for assessing the service life of components containing flaws (either real or hypothetical). Therefore, inclusion of fracture-mechanics-type fatigue specimens in surveillance programs is recommended.

10.2.2.1 Several acceptable specimen designs exist, including center-notched, single-edge-notched, bend, and compact specimens (see Test Method E 399 for the latter two types). The specimen design chosen should have a well documented solution for the stress intensity factor (K). The maximum fatigue loadings should be such that plasticity should be confined to a small (relative to the planar dimensions of the specimen) region in the vicinity of the crack tip.

10.3 *Conduct of the Test*—The major test parameters, temperature, cyclic frequency, stress ratio A or R (see Definitions E 206), loading waveform, and environment, should be selected to correspond as closely as practical to the expected component operating conditions for all types of tests.

10.3.1 For strain controlled push-pull tests the stress should be monitored to determine the extent of cyclic hardening or softening.

10.3.2 For sub-critical crack growth tests the crack length and the number of fatigue cycles corresponding to that crack length will be determined periodically throughout each test. Crack lengths may be determined periodically throughout each test. Crack lengths may be determined with any device (for example, cathetometer, eddy current, ultrasonic, electrical resistance, etc.) possessing suitable accuracy and resolution for the specimen employed.

11. Report

11.1 The results of these tests will cover long periods of time and it is essential that each report be complete. Pre-exposure condition of the specimen, details of exposure and environment, testing equipment, and technique shall be included in the report of results. If certain details have been adequately reported elsewhere, a reference to that report is sufficient.

11.1.1 *Tension Testing*—The following requirements for reporting are minimum requirements.

11.1.1.1 Specimen dimensions and type,

11.1.1.2 Temperature,

11.1.1.3 Test atmosphere,

11.1.1.4 Strain rate,

11.1.1.5 Extensometer and testing machine model number, accuracy, and range used,

11.1.1.6 Temperature-measuring system and accuracy, and

11.1.1.7 Yield point (if applicable), tensile strength, uniform and total elongation, and reduction of area. An autographic load-elongation record is desirable.

11.1.2 *Creep and Stress Rupture Testing*—The reporting procedure in Practice E 139 shall be followed.

11.1.3 *Fatigue Testing*:

11.1.3.1 *Fracture Mechanics Type Tests*—The reporting procedure in Test Method E 399 shall be followed. The method of calculating crack growth rates (for example, graphical differentiation, differentiation of a mathematically-fitted function, or point-by-slopes) should be stated. Customarily, the logarithm of fatigue-crack growth rate is plotted as a function of the logarithm of the stress intensity factory range. However, several relationships are available that correlate temperature, frequency, and stress ratio, and these may be used if appropriate.

11.1.3.2 *Axial or Bending Tests*—(Standard in preparation.)

11.1.4 *Charpy Impact Testing*—The reporting procedure in Test Methods E 23 shall be followed.

12. Keywords

12.1 irradiation; nuclear reactor vessels (high temperature); radiation exposure; surveillance (of nuclear reactor vessels)