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Standard Practice for Examination of Fuel Element Cladding Including the Determination of the Mechanical Properties[1]

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1. Scope

1.1 This practice covers the procedures for postirradiation examination of cladding, determination of breached nuclear fuel elements, selection of material and tests for radiation studies, determination of radiation conditions, conduction of tests for mechanical properties of cladding, and the reporting of data.

1.2 The purpose of this practice is to provide detailed guidelines for the postirradiation examination of fuel element cladding and to achieve better correlation and interpretation of the data in the field of radiation effects.

1.3 This practice may be applied to metallic cladding from all types of fuel elements. The tests described in this practice for determining mechanical properties of the fuel element cladding practice should be included in the preirradiation characterization of the cladding and for planning irradiation effects tests for evaluation of materials for cladding and other components in nuclear reactors.

NOTE 1—The values stated in SI units as described in Standard E 380 are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

B 353 Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service (Except Nuclear Fuel Cladding)[2]

E 3 Methods of Preparation of Metallographic Specimens[3]

E 8 Test Methods for Tension Testing of Metallic Materials[3]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]

E 21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials[3]

E 45 Test Methods for Determining the Inclusion Content of Steel[3]

E 92 Test Method for Vickers Hardness of Metallic Materials[3]

E 94 Guide for Radiographic Testing[5]

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 146 Methods of Chemical Analysis of Zirconium and Zirconium Alloys (Silicon, Hydrogen, and Copper)[6]

E 165 Test Method for Liquid Penetrant Examination[5]

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques[7]

E 263 Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Iron[7]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[8]

E 466 Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials[3]

E 606 Practice for Strain-Controlled Fatigue Testing[3]

F 77 Test Method for Apparent Density of Ceramics for Electron Device and Semiconductor Application[9]

3. Significance

3.1 The physical and mechanical properties of cladding materials can be altered by exposure to a high-energy neutron flux. The magnitude and relationship of these changes are functions of the material and its metallurgical condition; the amount, rate, and energy spectrum of the radiation; the exposure temperature; the test temperature; the purity, velocity, volume, and purification system of the coolant; and effects of contained fuel.

4. Determination of Radiation Conditions

4.1 Report all available data on the nuclear environment including reactor operating history, reactor geometry, and operating temperature. Provide neutron dosimetry for each experiment (refer to Practice E 261 for applicable practices). Suggestions as a minimum guide in measuring the radiation

[1] This practice is under the jurisdiction of ASTM CommitteeE10 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] *Annual Book of ASTM Standards*, Vol 02.04.

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

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 03.03.

[6] Discontinued—see *1989 Annual Book of ASTM Standards*, Vol 03.05.

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

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

[9] *Annual Book of ASTM Standards*, Vol 15.02.

environment are described in 4.1.1-4.1.5.

4.1.1 *Neutron Spectrum*—For the particular facility utilized, report the most complete neutron spectrum description available from reactor physics computations, from preexperiment surveys, and from monitoring of the experiment. Calculate the neutron spectrum from suitable transport or diffusion theory computer codes to best simulate the actual reactor conditions (that is, one-dimensional, cylindrical geometry, etc.). Report information on the neutron flux distribution versus neutron energy.

4.1.2 *Reactor Conditions*—Report information on the local reactor operating time history and reactor geometry including dimensions, materials, and locations. Obtain or calculate the number of equivalent full-power days (EFPD).

4.1.3 *Neutron Dosimetry*—Determine the neutron fluence as described in Practice E 261 and Test Method E 263. Normally, include the fast fluence above a given energy such as $E > 0.1$ MeV or $E > 1.0$ MeV, the displacements per atom (dpa), and reference the dpa technique used. For example, determine the fast fluence from the $^{54}Fe(n,p)^{54}$ Mn reaction in Zircaloy®[10] or stainless steel fuel cladding. In this case, accurate values of the iron content would be required, preferably preirradiation values. Then, calculate the neutron fluence from a knowledge of induced activities, spectrum-averaged cross sections, and reactor operating time histories.

4.1.4 *Temperature Knowledge*—For the evaluation of irradiation effects on physical and mechanical properties, knowledge of the absolute irradiation temperature and its uncertainty is extremely important. Where possible, use sufficient sensors to provide a complete knowledge of the temperature pattern throughout the assembly. When the use of sensors is not possible, state the method of determining the irradiation temperature.

4.1.5 *Irradiation Atmosphere or Medium*—Report the composition and impurities of the medium surrounding the specimens during irradiation in as much detail as practical.

4.1.6 Document the method of supporting/spacing, location of fuel rod, and pitch-to-diameter ratio.

5. Handling, Shipping, Storage, and Surface Preservation

5.1 It is essential that the conditions of the cladding caused by irradiation and exposure to the operating environment be preserved between the time the fuel element is discharged from the reactor and the nondestructive examinations are completed. Overheating or even aging at the irradiation temperature during handling and storage can result in partial annealing of the radiation effects, thereby influencing the mechanical and chemical properties. If the temperature of the fuel element becomes excessive because of decay heat, then provide adequate cooling. Ordinarily, this will be water or sodium for light-water reactor (LWR) and liquid metal fast-breeder reactor (LMFBR) fuel rods, respectively. The coolant should not have any detrimental effect on the cladding.

5.1.1 *Handling*:

5.1.1.1 Exercise utmost care in handling and packaging the fuel element or fuel rod. Clean each item in such a manner as to preserve its irradiated condition. Take special precautions to ensure that the cleaning procedure does not entail high temperature or high stress that might alter the as-irradiated condition or state.

5.1.1.2 All tools selected for handling of irradiated fuel elements should be of such a design and construction to minimize the risk of accidental damage to the element. Do not use elastomers and soft metals such as aluminum, copper, and lead, in contact with the fuel element. During handling do not subject the fuel elements to any stresses for which they are not designed.

5.1.2 *Shipping*:

5.1.2.1 The shipping containers should be equipped with suitable inserts, baskets, spacers, and supports so that the surface of the element is not damaged during transit. Take special care to avoid excessive vibration that can lead to fretting wear.

5.1.2.2 It may be necessary to cool the fuel element during shipment. If a coolant is not used, then calculations must show that the decay heat will not create an unacceptable increase in the temperature of the fuel element.

5.1.3 *Storage*:

5.1.3.1 Observe the same precautions and procedures at the receiving site as those observed at the shipping site.

5.1.3.2 Storage facilities commensurate with the size and mass of the fuel assemblies must be provided. The storage facility must have an environment that provides adequate cooling and protection to the element. For LWR fuel elements, demineralized water or air is adequate. Store LMFBR fuel elements in an inert gas atmosphere or high-purity sodium. Moisture and oxygen can react with residual sodium and form corrosion products on the LMFBR cladding. Avoid these reactions, as they can lead to attack of the cladding, thereby destroying the as-irradiated surface condition.

5.1.4 *Surface Preservation*:

5.1.4.1 In many postirradiation examinations, the surface of the fuel element, including the corrosion film and crud deposits, is of interest. Preserve these deposits as much as practicable until the visual examinations are complete or until an adequate sample has been obtained.

5.1.4.2 If the examination of loosely adherent surface deposits is of no interest, the deposits may be removed with a soft-bristled brush. For LWR fuel elements, water or organic solvents such as ethyl alcohol or acetone may be used as an aid in removing loosely adhering deposits. For LMFBR fuel elements, avoid the use of water. Alcohol is a suitable solvent for cleaning the LMFBR elements. Avoid vigorous cleaning agents, such as acids or strong bases, unless these agents are the only means of effectively removing the surface deposits.

5.1.4.3 If the composition of the crud or loosely adhering surface deposits is to be determined, then remove the samples by scraping the element with a suitable instrument. Take special care to avoid contamination of the sample with metallic particles scraped from the element or the instrument.

6. Visual Examination

6.1 Place the element (for example, fuel rod, plate, etc.) to be examined in a suitable holder. The holder should provide means of manipulating the element so that all areas of interest

[10] Zircaloy is a registered trademark of Westinghouse Electric Corp.

can be examined conveniently with a minimum of handling.

6.1.1 The holder should be sufficiently rigid to minimize the possibility of movement during photographing.

6.1.2 An appropriate scale should be incorporated into either the holder or the viewing instrument to aid the observer in establishing relative scale and magnification and to provide a convenient method of locating specific areas with reference to a fixed point, such as the end of a fuel rod. Use a color standard if color photography is utilized.

6.2 Perform visual examinations either in a hot-cell facility or in an underwater facility, such as a spent-fuel pit. Use viewing instruments, such as periscopes, telescopes, remote stereomicroscopes, or closed-circuit television systems.

6.2.1 The instrument should be equipped with interchangeable eyepieces, interchangeable or variable focal length (zoom) objectives, or some other convenient method for obtaining a range of magnifications.

6.2.2 Means should be provided for documenting the conditions and appearance of the cladding with photographs. This is best accomplished with a camera attached to the viewing instrument or by the use of video tape for television examination. Give a written description of the visual examination features to augment the photographs or video tape.

7. Dimensional Measurements

7.1 *Calibration*:

7.1.1 Calibrate all measuring apparatus by checking its indicated readout against standards.

7.1.2 Certify any standards, gage blocks, etc., used, and it is best if these standards can be compared directly to National Bureau of Standards masters. If this is not possible, compare the standards to primary standards or masters.

7.1.3 Include the dimensions of the standard, the applicable limits of precision and accuracy, and the temperature at which the measurements were taken in the certification report.

7.2 *Surface Preparation*—It may be necessary or desirable to remove any loosely adhering deposits on the cladding surface, so that the dimensional measurements are as accurate as possible. Tightly adherent oxide films can also affect the dimensional measurements. Therefore, use a metallurgical examination to determine exactly how these films affect the dimensional measurements.

7.2.1 Perform the removal of these loose deposits by mechanical means, such as light brushing in demineralized water.

7.2.2 Do not attempt to use vigorous chemical reagents to remove any of the tightly adhering oxide film which is frequently present. To do so would risk removal of the corrosion films and base metal, thereby decreasing the value of dimensional measurements and the corrosion data obtained from the film thickness measurements.

7.3 *Temperature Determination*—In making any dimensional measurements, also measure the temperature of the cladding. Due to gamma heating, the cladding surface temperature may be significantly higher than the ambient temperature of the environment.

7.4 *Length Measurements*:

7.4.1 It is important that the ends of the element to be measured be clean and free of any deformation.

7.4.2 Adequately support the element to be measured. If bowing is not reflected in the length measurement, indicate so in the data.

7.5 *Diameter Measurements*:

7.5.1 It is important that the surface of the element being measured be clean. Document the cleanliness of the surface of the element. In a manner least destructive to the element cladding, wipe away loose extraneous material (for example, particulates) adhering to the surface.

7.5.2 Take the measurements performed with a micrometer or similar device at two or more positions at each measurement location. Take these measurements at equally spaced positions, if possible, around the circumference of the rod. Use minimum or known force devices when measuring readily deformed shapes such as thin-wall fuel elements. Centering is an important consideration.

7.5.3 Select the axial location of diameter measurements on a fuel rod to characterize adequately the dimensional stability or performance of the rod. If possible, use the same location as the preirradiation measurements.

7.5.4 It is preferable that continuous diameter measurements be taken. This can be done by a profilometer, employing one or more dial indicators, LVDTs, or other suitable sensing devices, that bear on the fuel rod. Means can be provided to advance the sensing head axially as the rod is rotated and to record the output of the sensing head, thereby obtaining a continuous trace of the fuel rod surface. This will reveal not only gross changes in diameter, but other anomalies such as ridging, blisters, ovality, etc.

7.6 *Thickness Measurements*:

7.6.1 It is important that the surfaces of the element being measured be clean.

7.6.2 Determine the thickness of noncylindrical fuel elements (flat plates, convolutions, etc.) by using a micrometer or other suitable measuring device.

8. Determination of Breach and Defect Location

8.1 *Leak Tests*—Experience has shown that defects in fuel cladding cannot always be detected through a visual inspection or dimensional measurements, or both. To detect defects resulting in a breach of the cladding, some type of leak test is frequently performed as follows:

8.1.1 In the liquid nitrogen-methanol leak test, immerse the item to be tested in liquid nitrogen for approximately 15 min. Then remove and quickly plunge the item into methanol. A leak is indicated by the presence of a steady stream of bubbles. Do not use this test if there is any reason to believe that the cladding or fuel may be adversely affected by the thermal shock involved or that it might undergo a phase transformation.

8.1.1.1 In principle, the liquid nitrogen penetrates the cladding through a defect, such as a crack, pinhole, etc. When the rod is plunged into the warm methanol, the nitrogen immediately vaporizes and escapes through the defect.

8.1.1.2 In the case of large leaks, all of the gas may escape from the rod in a short burst of bubbles. However, this type of leak or defect is ordinarily detected during the visual examination.

8.1.1.3 In the event of a very small leak, the bubbles may be so small or infrequent as to escape detection. Also, the defect

may be actually covered or plugged with deposits of foreign matter.

8.1.2 A helium leak test is generally more sensitive than the liquid nitrogen-methanol. In addition, it has the advantage of being performed at room temperature, and thus does not subject the cladding to thermal shock. This method can be used to determine whether a leak is present but is not satisfactory for actually locating the defect.

8.1.2.1 In this test, expose the rod to an external helium pressure, remove from the pressurization chamber, then insert into the leak test chamber. Then evacuate the chamber and measure the leak rate with a helium leak detector.

8.1.2.2 Variations of this test make it possible to perform the test after the rod is punctured or sectioned. This test provides satisfactory results only when there are interconnecting voids throughout the length of the item to be tested.

8.2 *Dye Penetrant Inspection*—Various types of dye penetrant inspection may be used to detect surface flaws or cracks. Use Test Method E 165 as a guide.

8.2.1 In general, clean the cladding surface to obtain conclusive results.

8.2.2 The presence of a tightly adhering oxide film can render the results of a dye penetrant inspection inconclusive.

8.3 *Ultrasonic Inspection*—Ultrasonic inspection provides a means to detect defects on the hidden, as well as exposed, surface of fuel cladding. It may also be applied to the measurement of cladding wall thickness and the nondestructive examination of welds, brazed joints, etc.

8.3.1 Inasmuch as either water or a low-viscosity oil is required to couple the transducer with the target, perform this inspection in an underwater facility or in a tank of water in a hot cell.

8.3.2 The basic mechanical equipment generally required for ultrasonic inspection includes suitable holding fixtures for both the target and the transducer.

8.3.2.1 The target-holding fixture must be capable of moving the target with respect to the transducer or the transducer with respect to the target, or moving both, so that the entire area of interest may be inspected (for example, if the target is a fuel rod, a device for rotating the rod is required).

8.3.2.2 The transducer-holding fixture must maintain the desired orientation of the transducer and the distance away from the target.

8.3.3 Basic electronic equipment should include an oscillator to produce the signal for the transducer, a pulsing circuit, a receiver to detect the pulse echo, and a display or readout device, such as a strip-chart recorder or an oscilloscope.

8.3.4 A calibrated standard is necessary to adjust the equipment so that the size of a defect may be estimated.

8.4 *Weight Measurement*:

8.4.1 Employ weight measurement, particularly in sodium-bonded elements, to detect the presence of a breached fuel element by the indication of a greater-than-expected weight change due to egress of sodium bond or ingress of primary coolant.

8.4.2 The balance must possess sufficient sensitivity to detect the expected weight change.

8.4.2.1 Prior to any series of weight measurement, calibrate the balance with traceable standards. Between measurements, return the balance to a zero or known value. As appropriate, check the balance against working standard weights during the measurement process.

8.4.2.2 Periodically, replace or calibrate the working standard weights against certified reference weight standards.

8.4.3 Shield the fuel element being weighed from air currents that could adversely affect the accuracy of the data obtained.

8.4.4 Report the calibration data and measurement results with the system accuracy stated. Suspect defective elements can be identified as those in which the difference between the preirradiation and postirradiation weights is significantly greater than the system accuracy for both measurement systems.

8.5 *Eddy-Current Cladding Integrity Testing*—Eddy-current nondestructive testing techniques provide a means of testing the integrity of the cladding material of nuclear reactor fuel elements without contacting the cladding. By these techniques, it is possible to detect (*1*) the location of a breach in elements that have been identified as “leakers” by other test methods, and (*2*) defect areas in cladding after irradiation, but prior to breach.

8.5.1 Eddy-current testing does not require a liquid coupling medium. Therefore, the cladding is maintained free of contact. Employ either an encircling coil that tests the entire element circumference in one pass or a probe coil that tests only a small (about 15 deg) segment of the clad circumference at a time. The latter is somewhat more sensitive and provides easier interpretation of defect location with respect to circumferential orientation and inner or outer surface of the cladding.

8.5.2 The basic mechanical system requires a means of holding the test item and the sensing coil and providing the appropriate relative motion between the two. Position the test item accurately with respect to the test probe or encircling coil to maintain repeatable coupling.

8.5.3 Basic electronic equipment includes the necessary controls for operation of the mechanical equipment, eddy-current signal generator and receiver, and display and recording devices such as an oscilloscope and strip-chart recorder.

8.5.4 Interpretation of results depends on the use of calibrated and well characterized standards. Standards should contain defects that approximate typical defects. They should possess several defect sizes located both on the inside surface and the outside surface. Standards should also contain typical internal components, such as fuel pellets, spacers and springs, cladding variations, or components that would simulate these items.

9. Gamma Scanning

9.1 Employ gamma scanning to determine the axial distribution of cladding activation and fission-product activity, to determine the position and dimensions of element components, and to help determine suspect breached elements by loss of fission-product activity.

9.1.1 Use the distribution of the activation of the cladding or specific fission-product activity to determine the relative fast and thermal fluence received by the various portions of the element.

9.1.2 Determine the position and dimensions of element internal components (fuel pellets, bond-sodium level, etc.) by counting the appropriate specific activities.

9.1.3 Deficiency of fission-product activity within the gas plenum or bond-sodium indicates a breach of cladding. A loss of bond-sodium and dissolved fission products within the bond-sodium indicates breach below the sodium level.

9.2 The elements to be gamma scanned are moved past a thin slit through which the gamma and beta rays are allowed to pass. The gamma and beta rays are then collimated by a shielded slit over a suitable distance. The collimated beam is directed at a high-resolution detector attached to a monitor and readout system. The design of the system will depend on the size, shape, and activity of the item being scanned. A multichannel analyzer can be employed to identify and measure the activity produced by individual nuclides.

10. Neutron Radiography

10.1 Neutron radiography is a nondestructive technique for determining the internal condition of fuel elements or experimental capsules. A film image is produced, with the film density being a function of the neutron attenuation characteristics of the material being radiographed.

10.2 Neutron radiography provides information such as: internal component location, fuel condition (pellet separations, voids, fissile density variation, cracking, etc.), dimension of components, water ingress, bond-sodium level, hydride formation in cladding, etc.

10.3 Place the fuel elements including length and contrast standards in a collimated thermal neutron beam emanating from the neutron source (which may be a reactor, accelerator, radioisotope, or other source of neutrons). Place a neutron detector, usually a dysprosium or indium foil, behind the fuel elements and expose to the neutron beam. Indium resonance energy neutrons (1.4 eV) provide more penetration of nuclear fuels and are therefore useful in revealing internal characteristics in the fuel. With this technique, place a thermal neutron attenuater (gadolinium, cadmium) in front of the indium neutron detector. Subsequently place the detector in contact with X-ray films and allow to decay for three or more half-lives to produce the image. Since the radiation emanating from the activated neutron detector foils is low-energy, it is very important to ensure that the foil and films are in intimate contact and that no foreign material is allowed between the foil and film. Failure to do so will result in poor-quality radiographs. The film thus exposed can then be processed by using normal X-ray film processing techniques (see Guide E 94 as a guide). Dysprosium foils provide more detail in non-fuel components of a fuel element and are used in generally the same way as for indium foils.

11. Metallography

11.1 *Significance*—The cladding should be examined metallographically to determine the extent of any microstructural changes and the corrosion behavior resulting from the exposure in the reactor. The optical microscope can provide a significant amount of data regarding the in-reactor behavior. The electron microscope may enhance the accuracy of the analyses and interpretation.

11.2 *Specimen Preparation*:

11.2.1 Obtain a section of the fuel rod or plate with the fuel intact in a manner that prevents excessive heat generation or cold working during sectioning. Mount this section in a cold-setting compound that cures at temperature less than 150°C or lower and is resistant to gamma-irradiation and to deterioration or swelling at temperatures as high as 150°C. Maintain the record of specimen location and orientation, particularly if the orientation of the rod with respect to the rest of the assembly is known.

11.2.2 Rounding of the edges of the cladding during grinding or polishing is undesirable, especially when evaluating the corrosion behavior. If routine polishing techniques are not available to prevent this rounding of the surface, then prevent the rounding by mounting the specimen in a potting compound containing a fine abrasive material such as aluminum oxide or plating the specimen with a metal such as nickel or chromium. The latter method is undesirable in cases where examination of the fuel within the cladding is to be performed.

11.2.3 Prepare the metallographic specimen in accordance with Methods E 3. In some instances, it may be desirable to avoid grinding and polishing using an aqueous media. Certain organic liquids can be effectively used as a coolant in these instances. Take special care, however, to prevent heating of the specimen during grinding. The final polishing steps will vary with the material being processed and should be established on a trial specimen prior to conducting the examination.

11.2.4 After the specimen is polished satisfactorily, examine the specimen in the as-polished condition. If microprobe analysis is to be performed, then complete it before etching. Following this examination, etch the cladding or fuel to establish the desired microstructural characteristics. It is preferable to examine the fuel first since the etchants used for the cladding may attack the fuel thereby destroying areas of interest. In many instances, the corrosion rates are enhanced by irradiation. Therefore, take special care to avoid overetching. The etchants used for cladding are generally somewhat different than those for fuels. In the examination of both materials it is usually necessary to repolish and reetch the specimens after one examination has been completed.

11.3 *Fuel Cladding Interaction*:

11.3.1 Fuel-cladding contact, especially at high temperatures and high burnups, can lead to both chemical and mechanical interaction. The extent of both the chemical and mechanical interaction is often quite important in establishing the useful life of the fuel element. The mechanical interaction is the result of differential thermal expansion between the fuel and cladding. The chemical reaction can either be a reaction between the cladding and the fuel or a reaction between the fission products and the cladding.

11.3.2 Determine the depth of attack and the cause of the attack. Knowing the depth of the attack is very important, especially when sections of the cladding are to be used for determining the mechanical properties. Make special efforts to preserve this interaction. This may be difficult as these reaction products are often destroyed during normal handling and mounting operations. Impregnation of the specimens with

cold-setting epoxy compounds prior to any major metallographic preparations is useful for preserving the as-irradiated conditions. The epoxy resin, however, has a tendency to destroy the chemical nature of some of the fission products. The mode of attack (for example, stress-corrosion cracking, intergranular attack or cracking and general uniform corrosion) should also be established.

11.3.3 Measure the width of the fuel-cladding interaction zone from a specified plane of polish with a metallograph containing a calibrated filar eyepiece or from a photograph of calibrated magnification.

11.3.4 From the as-polished surface, obtain the composition and composition gradients by scanning the sample surface with ion or electron microprobes and performing electron or mass spectroscopy.

11.4 *Corrosion Behavior*:

11.4.1 Certain metals form an adherent protective film during corrosion, whereas other metals corrode by the loss of material. In the former case, it is possible to measure the oxide film thickness and to convert the oxide film thickness to a mass gain and to determine whether corrosion produces any compositional changes through the thickness of the cladding. In the latter case, it is not possible to establish the corrosion rate unless the mass of the specimen is known prior to exposure to the corrosive environment. However, it is possible to determine whether corrosion occurs by pitting, intergranular attack, etc., or produces compositional variation within the metal.

11.4.2 *Oxide Film Thickness*—The oxide film on many alloys, in particular zirconium alloys, is very seldom completely uniform in thickness. Consequently, a large number of measurements must be made to obtain a reliable value for the film thickness. Before making the measurements, examine the entire periphery of the as-polished cladding to determine whether there are any areas of abnormally thick or thin corrosion films. If such areas are present, treat these areas in an individual manner. If no abnormally thick or thin areas are observed, then take at least eight film thickness measurements at equally spaced locations around the periphery. Take these measurements with a filar eyepiece with an accuracy of ± 0.1 µm. The resulting film thickness, T_f, can be converted to a mass gain, M, using the following equation:

$$M = T_f \times K \quad (1)$$

K will vary from material to material, and this value must be determined experimentally. For Zircaloy-2 and Zircaloy-4,® K is equal to 15.6 mg/dm^2·µm.

11.4.3 *Hydrogen Content*—Evaluate the orientation of the hydride platelets within Zircaloy® fuel cladding using metallographic techniques. Determine the hydrogen content using the vacuum extraction techniques in accordance with Methods E 146. Because cooling rates and thermal gradients can affect the size and concentration of hydride platelets, quantitative metallographic techniques for determining hydrogen content are not recommended.

11.5 *Hardness*:

11.5.1 Irradiation of the fuel elements can produce gross or localized changes in the hardness of the cladding. The hardness of the irradiated cladding can provide some indication of the strength of the cladding and possibly some insight into the effects of operation on the microstructural changes in the cladding.

11.5.2 The cladding on many fuel elements is often less than 0.38 mm in thickness. Furthermore, the hardness may vary through the cladding thickness. Many hardness tests are therefore not suitable for this material. The Knoop and Vickers test have generally been found suitable for determining the hardness of the cladding. Therefore, follow the procedure in accordance with Test Method E 92. Take special care to ensure that valid results are obtained.

11.5.3 Obtain at least five individual readings at each location. Obtain hardness at a minimum of three locations: inside surface, outside surface, and mid-radius. If possible, determine the hardness at areas of abnormal appearance.

12. Density Measurement

12.1 *Scope*—This method covers the determination of the density of cladding using immersion techniques. Accuracies of 0.05 % can be achieved using this method. However, the obtained accuracy will vary with the mass of the specimen. Methods and definitions of terms for density measurement of solids by immersion techniques are documented in Test Method F 77 and Terminology E 12. The calculation of the density of solids to make determinations to better than 0.05 % requires careful attention to technique. Many factors, such as air buoyancy, temperature differences, surface tension of the liquid, chemical changes, and calibration of the density of the displacement liquid, contribute significantly to the results.

12.2 *Definition:*

12.2.1 *density*—the mass per unit volume, expressed in grams per cubic centimetre.

12.3 *Apparatus*:

12.3.1 *Chemical Balance*, capable of measuring to 0.1 mg.

12.3.2 *Thermometer or Thermal Sensor*, capable of being read to 0.2°C within the range from 15 to 25°C.

12.3.3 *Hook and Wire*—A hook suspended by a wire about 0.13 mm in diameter or less if possible, both materials being fabricated from corrosion-resistant material. This arrangement can be used either to support the specimen or a wire mesh basket that contains the specimen.

12.3.4 *Wire Mesh Basket*, constructed from a corrosion-resistant material to suspend the specimen during immersion when the hook and wire combination is not a feasible method of suspension.

12.3.5 *Tank*, with a sufficiently large opening to allow unhindered introduction and removal of the specimen and specimen holder to and from the immersing liquid. For small specimens, water currents and motion caused by introducing the specimen into the bath must be eliminated before weighing proceeds. If possible, a constant temperature liquid container should be used.

12.4 *Materials*:

12.4.1 *Test Liquid or Immersing Fluid*—Some acceptable common fluids used for density measurements are as follows:

12.4.1.1 Distilled water containing a wetting agent such as a detergent,

12.4.1.2 Carbon tetrachloride, CCl_4,

12.4.1.3 Bromobenzene, C_6H_5Br, and

12.4.1.4 Perfluorotributylamine, $(C_4F_9)_3N$.

12.5 *Procedure*:

12.5.1 Weigh the test specimen in air.

12.5.2 Immerse the specimen and holder into fluid. Allow sufficient time for the temperature of the specimen and the temperature of the bath to equilibrate. Ten minutes is generally sufficient for samples of 10 g or less. Measure and record the temperature of the fluid to 0.2°C.

12.5.3 Determine the density of the immersing fluid. This determination can be made either by weighing a known volume of fluid or by determining its buoyancy on a precision-machined standard specimen such as Invar or gold.

12.6 *Calculation*:

12.6.1 *Density of Liquid*—Obtain the density of the liquid as specified in 12.5.3. Since the density of most liquids varies with temperature, it will be necessary to make a correction if the specimen and liquid densities were measured at different temperatures.

12.6.2 *Density of Specimen*—Calculate the density of the specimen as follows:

$$\Delta = \{(W_A \times d) - (W_L \times D)\}/(W_A - W_L) \quad (2)$$

where:

Δ = density of specimen at temperature T, g/cm^3,
W_A = mass of specimen in air, g,
W_L = mass of specimen in liquid, g,
d = density of liquid at temperature T, g/cm^3, and
D = density of air, g/cm^3.

12.6.3 *Temperature Corrections*—Temperature can cause perturbations in the equation in 12.6.2. The equation can be modified to include variations in temperature as follows:

$$\Delta = \{(W_A \times d \times X) - (W_L \times D \times Y)\}/(W_A - W_L) \quad (3)$$

$$X = 1 - K(T - T_L)$$

$$Y = 1 - K(T - T_A)$$

where:

K = volumetric coefficient of thermal expansion, and
T, T_L, T_A = temperature of interest, of the immersion liquid and of the air, respectively.
When T_L = T_A, X = Y and the equation reduces to a correction involving K and the temperature of interest.

12.7 *Report*—The report shall include the following:

12.7.1 Chemical composition of specimen,

12.7.2 Previous mechanical and thermal treatment,

12.7.3 Irradiation data, including reactor, irradiation temperature, total fluence, neutron dosimetry and monitor, and instantaneous flux,

12.7.4 Mass of specimen,

12.7.5 Density of specimen to three significant figures,

12.7.6 Temperature at which density measurements are made, and

12.7.7 Immersion liquid.

13. Analytical Methods

13.1 *Routine*—Routine analytical methods are covered in the *Annual Book of ASTM Standards*, Section 3, where over 100 standard methods are described.

13.2 *Special*:

13.2.1 *Hot Vacuum-Extraction*—Hot vacuum-extraction techniques are methods used to remove absorbed and dissolved gases from solids. The usual manner by which this is accomplished is to heat the solid in a vacuum at a sufficiently high temperature to allow removal of the gases from the solid by diffusion and collect the released gas. The gas can then be analyzed by accepted standard methods for gas analysis. The techniques and methods used for this determination on irradiated samples are identical with those used for unirradiated samples with the exception of the special handling required to protect personnel from the residual radioactivity of the specimen. For hydrogen analysis by this technique, see Methods E 146.

13.2.2 *Vacuum Fusion*—Vacuum fusion analysis is a technique by which gas, adsorbed, absorbed, and combined in a solid, is released by melting or fusion, and the gas is collected for subsequent analysis. The methods recommended for irradiated materials are similar in most respects to those for unirradiated materials, with the exception of the special techniques required to protect personnel from the residual radioactivity and contamination of equipment with radioactive materials. The techniques vary slightly with each subject material and recommended procedures are not currently available.

13.2.3 *Microprobe:*

13.2.3.1 *Scope*—The term “microprobe analysis” covers a number of operations ranging from quantitative chemical analysis to qualitative photographic displays. There are several ways in which the electron microprobe can be used depending on the application of the data to be obtained. These include:

(1) X-Ray Spectrum Scan—A method to determine the elements present in an unknown sample.

(2) Line Scan—A semiquantitative method of determining the relative concentration of an element across a portion of the sample.

(3) Step Scan—A line scan in which the specimen is moved through the electron beam in finite increments, and X-ray counts are recorded during these incremental steps.

(4) Area Scan—The X-ray spectrometric method set to receive the characteristic X-ray wavelength from a specific element that can be used to scan a given area.

13.2.3.2 *Procedure*—Prepare the sample in the same manner as a metallographic specimen. Obtain sufficiently smooth surface for examination by a final polish with 0.25-µm diamond paste. A light chemical or electrolytic etch may be required after polishing to bring out the metallurgical structure of the surface. Examine test samples in the as-polished condition if there is danger of removing elements of interest by etching. The procedures for examination are highly dependent upon the type of examination required. The examinations generally can be classified into two types, qualitative analysis and quantitative analysis as follows:

(1) Qualitative Analysis—When only some indication of the elements in the subject material is known and positive identification is desired, use the following techniques: (*a*) spectrum analysis for the unknown elements; (*b*) area scan to show the distribution of the elements present; and (*c*) line scan

to show the change in compositional distribution of the elements present.

(2) Quantitative Analysis—Use the following techniques when the compositional elements of the sample are known but their relative amounts are unknown: (*a*) single spot analysis and (*b*) trace analysis over a small zone.

13.2.3.3 *Report*—Standardization of report forms is not applicable for reporting microprobe analysis because of the lengthy remarks that often are needed to accompany the data.

14. Mechanical Property Test

14.1 *Selection of Materials*—Obtain the chemical composition, properties, and metallurgical structure of the material before irradiation for all materials used to determine the mechanical properties. This information should include the following:

14.1.1 Processing history, including such information as metal designation, manufacturer, heat number, fabricating procedures, and heat treatment. (When possible, archive samples shall be maintained and stored for future reference.),

14.1.2 Chemical composition,

14.1.3 Original location and orientation of all test specimens in the parent material,

14.1.4 Appropriate mechanical properties prior to radiation, determined on specimens taken from a location and orientation equivalent to that of the irradiated specimens,

14.1.5 Optical micrographs showing the structure of the material at the test location and, if possible, electron micrographs,

14.1.6 Average grain size of these materials, estimated in accordance with Test Methods E 112, and for steels, the inclusion content shall be determined in accordance with Test Methods E 45,

14.1.7 Other information such as hardness, the variation in hardness, and other mechanical properties with locations and orientations through the thickness of the material, etc.,

14.1.8 Crystallographic orientation of the as-fabricated material, and

14.1.9 Density of the material being irradiated.

14.2 *Removal of Fuel*:

14.2.1 For many of the mechanical property tests, it will be necessary to remove the fuel from the cladding. Where the mechanical and chemical interaction between the fuel and cladding has been minimal, removal of the fuel should not be a problem. Where the fuel-clad interaction has been large, then special techniques must be developed to remove the fuel as there are no standard techniques available.

14.2.2 Both chemical (dissolution of the fuel) and mechanical (grinding or drilling of the fuel) techniques have been used to remove the fuel. Regardless of the technique used, take special care to prevent damage of the cladding. Evaluate any procedure that is developed on some dummy specimens before being used. Then evaluate the dummy specimens to ensure that the mechanical or chemical attack has not affected the properties of the cladding.

14.3 *Design of Specimens*:

14.3.1 *Tension Specimens*—In general, four types of specimens can be obtained from the various types of fuel cladding: (*1*) full-size tubular sections, (*2*) specimens from sheet or strip, (*3*) longitudinal specimens obtained from a segment of tubing, and (*4*) short rings obtained from the tubing. Where possible, the type of specimen employed shall conform to the requirements of Test Methods E 8. In some instances, it may not be possible to select a specimen that conforms to Test Methods E 8 because of the configuration of the cladding or because of the limited irradiation space. Where it is not possible to use standard test specimens, it is recommended that the length to width ratio of the gage length be about 4.0. The thickness of the specimen should be equal to that of the cladding, and nonirradiated specimens shall be of the same size and shape. Make every effort to establish the correlation between the type of specimen used and a standard specimen. Attach the grips, extender tab, or tubing fittings in such a way to avoid bending, cutting, or changing the material properties near the attachment. Perform the tension test at temperatures and strain rates most closely simulating service condition or expected conditions.

14.3.2 *Burst Specimens*—Two types of specimen designs will be generally utilized: (*1*) a piece of tubing cut from a reactor cladding section that contained a fuel material, and (*2*) a specimen designed and irradiated as a study material. Each of these specimens will vary slightly because of its history. The cladding will generally be open at each end and can be closed by welding or by mechanically attaching plugs in one end. The second specimen may be open or have an end plug welded in place prior to irradiation. In all cases, the basic specimen will consist of a section of tubing closed at one end by a blind plug and open at the other end to allow pressurization. Irradiated and nonirradiated control specimens shall be of the same size and type. The ratio of gage length to diameter (L/D) should be >10; however, where shorter specimens are used to conserve material or reactor space, run adequate testing to ensure the absence of end effects that alter the test information.

14.3.3 *Creep Specimens*:

14.3.3.1 Test specimens of the type, size, and shape described in Test Methods E 8 are generally suitable for tests at elevated temperatures. Where it is not possible to use standard test specimens, irradiated and nonirradiated specimens shall be of the same size and shape, and every effort shall be made to establish the correlation between the type of specimen used and a standard specimen. When the dimensions of the material permit, except for sheet and strip, the specimens should have circular cross section over the gage length. The ratio of gage length to diameter should be 4. If different ratios are used, direct specific attention to this fact in reporting the test results. The reduced section of sheet specimens do not need to be longer than 57 mm or wider than 25 mm. Take sheet specimens from the sheet stock in the direction of rolling, unless otherwise specified.

14.3.3.2 Specimens of circular cross section may have either threaded or shouldered ends for gripping.

14.3.3.3 For sheet or strip specimens, some modification of the standard specimens described in Test Methods E 8 is usually necessary to permit application of the load to the specimen in a furnace. If the material available is sufficient, the use of elongated shoulder ends to permit gripping outside the furnace is the easiest method. When the length of the specimen

is restricted necessarily, several methods of gripping are used, as follows:

(1) High-temperature sheet grips similar to those illustrated in Test Methods E 8 and described as self-adjusting grips. These have been proved satisfactory for testing sheet materials that cannot be tested satisfactorily in the usual type of wedge grips,

(2) By means of extension tabs welded or brazed to the specimen shoulders and extending to grips outside of the furnace, or

(3) Specially designed grips that conform to and apply the load against the fillets at the end of the gage length.

14.3.3.4 Test full sections of tubing using modified tubing fittings as grips or as closures when using the tubing as a biaxial test specimen in creep.

14.3.4 *Tubular Creep Specimens*:

14.3.4.1 A variety of creep tests using tubular specimens are possible depending on the methods for applying the load and the results desired. These tests include uniaxial creep, biaxial creep using an internal pressure, and biaxial creep using internal pressure and axial tensile or compressive loads. The type of information required will determine which test to use. Tests to compare different materials can generally use the uniaxial test or biaxial creep test using internal pressurization. Creep tests to obtain precision data for code development will generally require the use of biaxial creep specimens using a combination of internal pressure and axial loads.

14.3.4.2 The internal pressurization tests can use a short section of tubing, L/D ratio of >10, especially if the test is run similar to standard burst test.

14.3.5 *Fatigue Specimens*—Test specimen design depends on the type of testing machine used and the type of fatigue to be simulated. Configurations for specimen gage lengths suitable for testing in stress-controlled high-cycle fatigue (greater than 50 000 cycles) are provided in Practice E 466. Configurations for specimens suitable for testing in strain-controlled, low-cycle fatigue are provided in Practice E 606. Both irradiated and nonirradiated controls shall be of the same size and shape.

14.3.6 *Bend Specimens*—Specimen design for bend tests is relatively fixed. The recommended specimen is 130 mm long with a width-to-thickness ratio of 20:1 for thicknesses of 1.3 to 2.5 mm. Specimens thinner than 1.3 mm shall be a minimum of 25.4 mm wide. The maximum recommended thickness is 2.5 mm. When it is not possible to use the standard 130-mm long specimen, irradiated and nonirradiated specimens shall be of the same size, and every effort shall be made to establish the correlation between the subsized specimen and a standard specimen. The same specimen design is applicable for both room temperature and elevated temperature testing.

14.3.7 *Transient Specimens*—Two types of specimen designs will be generally utilized: (*1*) a piece of tubing cut from a reactor cladding section which contained a fuel material and (*2*) a specimen designed and irradiated as a study material. Each of the above specimens will vary slightly because of its history. The cladding will generally be open at each end and can be closed by welding or by mechanically attaching plugs in one end. The second specimen may be open or have an end plug welded in place prior to irradiation. In all cases, the basic specimen will consist of a section of tubing closed at one end by a blind plug and open at the other end to allow pressurization. Irradiated and nonirradiated control specimens shall be of the same size and type. The ratio of gage length to diameter should be >10; however, where shorter specimens are used to conserve material or reactor space, run adequate testing to ensure the absence of end effects that alter the test information. Insert a quartz or fused silica rod into the specimen before attaching the end caps. The quartz rod should allow for gas flow from one end of the specimen to the other. The quartz rod minimizes the gas volume (that is, stored energy), thereby producing a burst event typical of a fuel pin.

14.4 *Preparation of Specimens*:

14.4.1 *Tension Specimens*:

14.4.1.1 Test specimens should be of uniform thickness throughout the gage length.

14.4.1.2 Obtain the specimens from the cladding using any suitable machining technique that minimizes the amount of cold work introduced into the specimen. Cool the specimens with a suitable medium where heat may be generated during machining.

14.4.1.3 All surfaces, both machined and unmachined, of the finished specimen shall be smooth and reasonably free of undercuts and scratches. Do not use chemical methods for polishing or deburring, because the chemical could selectively attack the specimen and result in poor specimens.

14.4.2 *Burst Specimens*:

14.4.2.1 Test specimens should be of uniform wall thickness throughout the gage length.

14.4.2.2 Keep the type of end closure constant. Several types of closures have been found to be successful. Utilize a type that will produce an effective closure and not contribute to or detract from the properties of the tube. Common closures include end plugs welded in place, mandrels held in place by pressure fittings, and blind-end pressure fittings. Attach a pressure fitting for introduction of the pressurization fluid or gas to the open end. Take care to ensure that the attachment of pressurization fittings does not affect the properties of the tubes. Where welded end plugs are used, constrain the cladding adjacent to the weldment so that failure does not occur in the heat-affected zone. Where applicable, use the procedures recommended in Specification B 353 as a guide.

14.4.3 *Creep Specimens*:

14.4.3.1 Test specimens should be of uniform cross section throughout the gage length and should be within the following limits:

Nominal Diameter or Width, mm	Tolerance, Percent of Diameter of Width
2.5	±0.5
6.5	±0.4
8.0	±0.3
13.0	±0.2

The diameter or width at the ends of the reduced section should not be less than the diameter or width at the center of the reduced section. It may be desirable to have the diameter or width of the reduced section of the specimen slightly smaller at the center than at the ends. This difference should not exceed the tolerance limits specified above.

14.4.3.2 All surfaces, both machined and unmachined, of the finished specimens shall be smooth and reasonably free of undercuts and scratches. Exercise special care to minimize the disturbance of surface layers by cold work. Exercise great care to minimize eccentricity in the specimen to provide for axial application of the load. When extension tabs are used for sheet or strip samples, perform the brazing or welding in a jig or fixture to ensure accurate alignment, and take precautions to avoid overheating the specimens. Where loading pins are used for sheet or strip specimens, accurately center the pin holes on extensions of the center line of the gage sections.

14.4.3.3 Overall specimen length, length of shoulders, threaded ends, etc., should not be less than prescribed in Test Methods E 8, or by customary sheet practice for specimens for tension tests. Increasing the length of the shoulder facilitates obtaining the proper temperature distribution and axiality of loading.

14.4.4 *Tubular Creep Specimens*:

14.4.4.1 Select sections of cladding that have minimum variations in wall thickness, ovality, and eccentricity. Decrud the irradiated fuel cladding and remove the fuel. Make every effort to avoid damage of the cladding during removal of the fuel. Specimens requiring application of an axial load should be long enough to minimize the end effects of the grips. Specimens having a gage length of 70 to 100 mm should be at least 400 mm long. This will depend on the type of grips and the length of the furnace.

14.4.4.2 The specimen for axial creep tests requires no special preparation as a section of the tubing will be adequate. Take special care in the design of the grips to ensure that slippage does not occur and that they do not fracture the cladding embrittled by irradiation.

14.4.4.3 The biaxial creep tests will require special end fittings, one end being capable of introducing the gas pressure. The optimum design is a special end cap welded to the tubular specimens. The end cap can be designed such that grips may be used to apply the axial load. Swagelock®[11] devices can also be modified to be acceptable as end caps. These devices, however, are not as satisfactory as the welded end plugs for high-temperature and long-duration tests.

14.4.5 *Fatigue Specimens*:

14.4.5.1 Test specimens should be of uniform cross section throughout the nominal gage length and within the following limits:

Nominal Diameter or Width, mm	Tolerance, Percent of Diameter or Width
2.54	±0.5
6.35	±0.4
8.13	±0.3
12.83	±0.2

The diameter or width at the ends of the reduced section should not be less than the diameter or width at the center of the reduced section. It may be desirable to have the diameter or width of the reduced section of the specimen slightly smaller at the center than at the ends. This difference should not exceed the tolerance limits specified above.

14.4.5.2 All surfaces, both machined and unmachined, of the finished specimens must be as smooth as possible and free of undercuts and scratches. Undercuts and scratches will reduce the endurance limit of most materials. Exercise special care to minimize disturbance of the surface layers by cold work. Regardless of the machining, grinding, or polishing method used, remove the final metal in a direction approximately parallel to the long axis of the specimen.

14.4.6 *Bend Specimens*:

14.4.6.1 Test specimens should be of uniform cross section throughout their length, width, and thickness. The maximum allowable limits are ± 0.01 mm in the gage-section area of the sample.

14.4.6.2 All surfaces, both machined and unmachined, of the finished specimens should be smooth and reasonably free of undercuts and scratches. Exercise special care to minimize disturbance of surface layers by cold work.

14.4.7 *Transient Test*—See 14.4.2.1 and 14.4.2.2.

14.5 *Procedure*:

14.5.1 *Tension Test*:

14.5.1.1 Conduct the tension test in accordance with Test Methods E 8 or E 21, except for the following modifications:

(1) Measure and control the strain rate, when possible, by using a suitable extensometer attached to the gage length or the shoulders of the specimen. Rate of crosshead separation is a permissible method of controlling the speed of testing; when speed of testing is controlled in this manner, determine the accuracy of this method.

(2) If the test is interrupted for removal of the extensometer, make every effort to ensure that the load does not decrease significantly during the test.

(3) Record the complete stress-strain or load-deflection curves.

(4) Determine the uniform elongation, which is the increase in length of the gage length prior to the onset of necking, expressed as a percentage of the original gage length, from the elongation recorded by the extensometer at the maximum load. The uniform elongation can often be determined from the change in cross-sectional area of the unnecked portion of the broken tension test specimen.

14.5.2 *Burst Test*:

14.5.2.1 Conduct the temperature control in accordance with Practice E 139 and using Specification B 353 as a guide.

14.5.2.2 Apply the pressure to the tube at a controlled, constant, or known rate. Apply this pressure with oil, water, gas, etc., dependent upon the test temperature and the type of pressurization equipment. Avoid the use of equipment that causes a pulsating pressure. The pressurization fluid should not cool the test specimen. Preheating of the fluid may be needed. Consider instrumentating the test and pressurizing in a controlled volume (strain rate) mode for better comparison and possibly correlation with conventional tension tests.

14.5.2.3 Do not apply the load until the specimen is uniformly heated and the thermal equilibrium is sufficiently established to ensure that the temperature can be maintained within the specified limits. Record a pressure versus strain curve where possible.

[11] Swagelock is a registered trademark of Crawford Fitting Co.

14.5.2.4 Obtain stress-strain data using the thick-walled-tube relationship for an unrestrained, uniformly, internally pressurized cylinder:

$$\sigma = P\,(R_I^{\,2} + R_O^{\,2})/(R_O^{\,2} - R_I^{\,2}) \quad (4)$$

where:

σ = hoop stress, Pa,
P = internal pressure, Pa,
R_O = outside radius of the tube, mm, and
R_I = inside radius of the tube, mm.

This equation is applicable even when $R_O \geq 1.05\ R_I$.

14.5.3 *Creep Test*:

14.5.3.1 Conduct temperature control and measurement in accordance with Practice E 139 except for the following modifications:

(1) Temperature variation of the specimen from the indicated nominal test temperature shall not exceed ± 3°C for the duration of the test.

(2) Measure indicated temperatures prior to adjustments at least twice every working day.

(3) Continuously record the indicated test temperature by a suitable device during the test.

(4) Indicated temperature variations along the gage length of the specimen should not exceed the following limits:

Up to 540°C, incl	±2°C
540 to 1090°C, incl	±3°C
Over 1090°C	±4°C

(5) Overheating prior to attaining the limits specified shall not exceed 1 % above the indicated nominal test temperature; the duration of such heating not to exceed 10 min.

(6) The control of bending moment during the testing of irradiated material is an important consideration. It is recognized there is difficulty in obtaining the required alignment standard specified in Practice E 139; however, make a concerted effort to keep the bending moment as low as possible for postirradiation testing. When possible, obtain and report the alignment characteristics of the creep unit being used.

14.5.3.2 The desirability of attaching the strain-measuring equipment to the specimen is clearly recognized; however, this is not always possible because of the difficulties sometimes encountered due to the use of manipulators. Under carefully controlled conditions, make strain measurements by measuring the movement in the load train. When this method is used, take the following precautions: (*1*) make strain measurements on control specimens by placing a suitable strain device on the gage section of the specimen and on the load train, (*2*) then make a correlation between the actual strain and the apparent strain, and (*3*) test a sufficient control specimen to establish this correlation at each test temperature and for each method.

14.5.3.3 Place the specimen in the test equipment and bring to uniform temperature, taking care not to overheat the specimen beyond the nominal temperature.

14.5.3.4 Do not apply the load until the specimen is uniformly heated and thermal equilibrium is sufficiently established to ensure that the temperature can be maintained within the specified limits. Preheat for not less than 1 h after obtaining equilibrium temperature. The most important feature governing the preheat time before applying the load to the specimen is the attainment of adequate control of the temperature. Experience indicates that specimens can be brought to a temperature and the temperature stabilized adequately if sufficient attention is given to temperature adjustments prior to and subsequent to applying the load. Short preheat times require special attention to temperature variables prior to and after loading the specimen. It has long been recognized that a practice of heating the specimens to within 28°C of the temperature in 2 to 4 h with a final adjustment period of 20 h results in good temperature stabilization. This practice is preferred when such prolonged heating periods do not introduce structural changes that affect the properties of the material being tested or constitute poor utilization of equipment time. Note the practice that is used in the report.

14.5.3.5 Apply the load in such a manner as to avoid shock loads or overloading due to inertia. Apply the load in increments with strain readings between increments to provide stress-strain data for the application of the load. Apply the load as quickly as possible within these limitations. Strain measurements for creep tests should measure all of the creep after the load is applied and, preferably, should measure the strain from applying the load.

14.5.3.6 For rupture tests, provide a suitable means for measuring the elapsed time between the complete application of the load and the time at which the fracture of the specimen occurs to within 1 % of the elapsed time.

14.5.3.7 If any temperature disturbances cause the temperature of the specimen to rise above or below the limits specified, reject the test and retest. Exception may be made to this when the length of time below the nominal temperature is so short that it does not significantly influence the creep rate or rupture time. Allowing the temperature to fall below the nominal temperature reduces creep rate and prolongs rupture time, both characteristics being very sensitive to test temperatures. Low temperatures usually do not damage the material as do higher temperatures, which may accelerate the creep considerably. Consequently, under-temperatures should be cause for rejection only when the time at that temperature significantly alters the test results.

14.5.4 *Tubular Creep Test*:

14.5.4.1 All of the test procedures described in 14.5.3 are applicable to the tubular creep tests. The following additional guidelines are provided:

(1) Load Application—The biaxial strain in the tubular specimens is generated by the internal gas pressure. Inert gases, argon or helium, should be used to minimize reaction of the gas and the cladding. A bottled gas with a precision pressure gage and regulator is satisfactory. The pressure gage should have an accuracy of ± 35 MPa. Apply the axial load using a constant load-lever-arm creep machine or a standard tensile test machine. In either case, the machine should be equipped with an automatic beam-leveling device.

(2) Strain Meassurement—The strain can be measured in a number of ways, depending on the information needed and the accuracy desired. Use micrometers and profilometers to measure the diametral strain where accuracy isn't too important and where total plastic strain is desired. These devices are used during post-test measurements and can only obtain data on total plastic strain. Measure axial strain with micrometers or by

measuring the distance between fiducial marks. The axial strain (plastic plus elastic) can also be measured by using cross head travel. The use of strain gages is recommended to obtain very accurate data for code development. Use both axial and circumferential gages. They should have an accuracy of ± 0.005 %, preferably ± 0.001 %. Take special care in attaching the gages to ensure that the adhesive is compatible with the cladding material, the gage can measure the amount of strain anticipated, and the adhesive can withstand the test temperature. An automatic data acquisition system is recommended for recording and evaluating the data.

14.5.5 *Fatigue Test*:

14.5.5.1 Conduct the temperature control and measurement in accordance with Practice E 139 as follows:

(1) Report the indicated test temperature continuously by using a suitable device during hours when the test is unattended.

(2) Indicated temperature variations along the gage length of the specimens should not exceed the following limits:

Up to 540°C, incl	±2°C
540 to 1090°C, incl	±3°C
Over 1090°C	±4°C

Overheating prior to attaining the limits specified shall not exceed 1 % above the indicated nominal test temperature; the duration of such heating should not exceed 10 min.

(3) Conduct strain measurement, if applicable during test, in accordance with Practice E 606.

14.5.6 *Bend Test*:

14.5.6.1 Conduct temperature control and measurement in accordance with Test Methods E 8.

14.5.6.2 Measure strain by using strain gages (room temperatures) and a device to measure the radius of curvature in the gage section at elevated temperature. For modulus determinations, use an ASTM Type A extensometer. For routine testing use ASTM Type B-1 or B-2. Optical and mechanical devices are acceptable for the above.

14.5.6.3 Measure the dimensions of the cross section of the specimen to within ± 0.03 mm.

14.5.6.4 Load the specimen continuously at an initial outer fiber strain rate of 0.005 ± 0.002/min at room temperature. If the ductility of the sample exceeds 5 %, gradually increase the strain rate on subsequent tests of similar material to 0.05 ± 0.2/min after 0.6 % offset or 1.0 % total strain is reached. For elevated temperature testing, use an initial strain rate of 0.005 ± 0.002/min until a total strain of 1.0 % is reached and then change the rate to 0.05 ± 0.02/min.

14.5.6.5 In the elastic region, the stresses induced in a bending beam, σ, in megapascals, are given by the flexure formula as follows:

$$\sigma = 3Pa/bt^2 \quad (5)$$

where:
P = load, N
a = distance, mm
b = width of gage section, mm, and
t = thickness of gage section, mm.

14.5.6.6 Beyond the elastic limit or proportional limit, the stress is no longer merely a function of the load P, but rather is a function of the variation or change in load with the change in strain of outer fibers. The stress calculated in the outer fibers reaches a maximum before the maximum load is applied. Use the following formula to calculate the stresses beyond the proportional limit:

$$\sigma = (2/bt^2)\,[P_2a + (A/2)\,(\Delta P/\Delta\Sigma)\,(\Sigma_2)] \quad (6)$$

where:
ΔP = $P_2 - P_1$(corrected load), N
$\Delta\Sigma$ = $\Sigma_2 - \Sigma_1$(corrected strain), mm/mm,
t = thickness, mm,
P = load, N, and
a = distance, mm.

14.5.6.7 Calculate the strain from the following formula:

$$t = 4st/l^2 \quad (7)$$

where:
s = deflection reading, mm
t = thickness, mm, and
l = gage length, mm.

14.5.6.8 Plot a stress-strain diagram using the information in 14.5.6.5-14.5.6.7.

14.5.7 *Transient Test*:

14.5.7.1 Obtain cladding mechanical property data for analysis of fuel pin transient behavior under experimental conditions in which the temperature ramps associated with reactor transients are simulated. The purpose of simulated transient testing of fuel cladding specimens is to define the failure stress and strain for cladding over a wide temperature range for various heating rates.

14.5.7.2 The equipment utilized for a transient test must provide for rapid heating of the specimen under a controlled rate. Commonly, an induction generator is used with closed-loop control. A thermocouple spot-welded to the specimen or an equivalent temperature-sensing device such as an optical pyrometer provides the feedback for the control. The response rate of the temperature sensor must be rapid enough to keep pace with the various heat rates involved. During the test, monitor both the specimen pressure and temperature. Accomplish pressure measurement conveniently with a strain-gage pressure transducer, whose output can be monitored with a recorder. Measure the temperature with the controlling device. Additional thermocouples or other temperature sensing devices may also be used. Generate the temperature ramps by a function generator that is calibrated to the output of the temperature sensing device or thermocouple. All temperature and pressure measuring equipment should be calibrated and should have a response time compatible with the transient.

14.5.7.3 When induction heating is used, considerable care is required in the design of the heating coil. The coil must provide a uniform temperature along the length of the sample for the duration of the test. If different heating rates are to be used, different coils may be required. Insulate the coil from the specimen with an appropriate insert or coating.

14.5.7.4 For other heating methods, use similar cladding temperature profiles and conditions required by 14.5.7.3. Conduct the test according to the following sequences:

(1) If using a thermocouple, spot-weld it to the specimen with the wires spaced about 1.5 mm apart. Check the continuity and resistance of thermocouple.

(2) Place the specimen in the RF coil and connect the thermocouple and gas pressure line. Pressurize the specimen to approximately 80 % of the test pressure and check for leaks.

(3) Heat the specimen to the desired initial temperature and adjust the pressure to the desired level. Hold the specimen at this temperature until equilibrium has been attained. Check all equipment to make sure that the proper settings have been achieved.

(4) Perform the test and terminate when burst occurs. Note readings for test time, temperature, and pressure.

(5) After the specimen has cooled, locate the rupture and record the location.

(6) Measure the diameter of the specimen at increments along the axis.

14.6 *Report:*

14.6.1 *General*—The report, regardless of the mechanical property test, shall include the following:

14.6.1.1 Description of the material tested as recommended in 14.1,

14.6.1.2 Description of the test specimen, size, shape, etc., and if other than a standard specimen is used, include the data correlating the specimen used with a standard specimen,

14.6.1.3 Location and orientation of the test specimen in the parent material,

14.6.1.4 Data on radiation environment as recommended in Section 4:

(1) Irradiation temperature,

(2) Neutron fluence,

(3) Reactor location, and

(4) Coolant or environment.

14.6.1.5 Trade name and model of testing machine, gripping devices, extensometer, and recording devices used in the test, methods, and results of calibrations of the apparatus,

14.6.1.6 Speed of testing, and method of measuring and controlling the speed,

14.6.1.7 Temperature of the specimen during testing,

14.6.1.8 Atmosphere or medium surrounding the specimen during the test,

14.6.1.9 Photographs of the fractured specimens,

14.6.1.10 Temperature gradients and method of determination, and

14.6.1.11 Test specimen axial or radial restraint.

14.6.2 *Specific*—In addition to the information required in 14.5, the following information shall be reported for each specific test:

14.6.2.1 *Tension Test*:

(1) Complete stress-strain curves (if a group of specimens exhibits similar stress-strain curves, a typical curve may be reported for the group),

(2) Yield strength or yield point and method of measurement,

(3) Tensile strength,

(4) Fracture strength and fracture stress. (Fracture strength is the load at fracture divided by the initial cross-sectional area. Fracture stress is the load at fracture divided by the cross-sectional area at the time of fracture.),

(5) Uniform elongation and method of measurement,

(6) Total elongation, and

(7) Reduction of area.

14.6.2.2 *Burst Test*:

(1) Yield strength,

(2) Hoop strength,

(3) Location of rupture with regard to any surface defects, weld locations, or any irregularities in the tubing, and

(4) Method by which the fuel was removed from the tubing, where applicable.

14.6.2.3 *Creep Tests*:

(1) Stress in megapascals,

(2) Time in hours for total or plastic strains of 0.1, 0.2, 0.5, 1.0, and up to 5 % if available,

(3) Minimum creep rate in percent per hour,

(4) Transition to third stage creep in hours,

(5) Test duration hours indicating whether rupture occurred,

(6) Total extension for creep tests,

(7) Elongation and reduction of area for rupture tests,

(8) Primary creep behavior,

(9) From stress-rupture curves, determine the stress to rupture for times of 1, 10, 100, 1000, 10 000, and 100 000 h. If extrapolation is involved, the report must include the ranges of rupture times and creep rates upon which the extrapolation is based, and

(10) Stress for designated creep rate of 0.000 01 and 0.0001 %/h. If extrapolation is involved, the report must include the ranges of rupture times and creep rates upon which the extrapolation is based.

14.6.2.4 *Fatigue Tests*:

(1) Speed of cycling in cycle per unit time,

(2) Description of type of control used on fatigue machine and accuracy of this control,

(3) Number of cycles to failure for each test condition,

(4) S-N diagrams, if data permits or if endurance limit is determined,

(5) Mean stress, stress range, mean strain, and strain range, and

(6) Any data or description of the test that will aid in comparison or interpretation of data, or both,

14.6.2.5 *Bend Tests*:

(1) Complete load-deflection or stress-strain curves,

(2) Yield strength or yield point and method of measurement,

(3) Tensile strength, if obtained.

14.6.2.6 *Transient Test*:

(1) Rate of heating,

(2) Failure temperature and pressure,

(3) Uniform failure strain, and

(4) Environmental conditions for specimen if irradiated prior to testing.

15. Keywords

15.1 fuel cladding; mechanical testing; nuclear reactor fuel; radiation