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Standard Practice for Examination of Fuel Element Cladding Including the Determination of the Mechanical Properties¹

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^{ε1} NOTE—Keywords were added editorially in August 1996.

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 Specimens³
- E 8 Test Methods for Tension Testing of Metallic Materials³
- E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases⁴
- E 21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials³
- E 45 Test Methods for Determining the Inclusion Content of Steel³

¹ This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications.

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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.

E 92 Test Method for Vickers Hardness of Metallic Materials³

E 94 Guide for Radiographic Testing⁵

E 112 Test Methods for Determining the Average Grain Size³

E 139 Practice for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials³

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

E 165 Test Method for Liquid Penetrant Examination⁵

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques⁷

E 263 Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Iron⁷

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 Materials³

E 606 Practice for Strain-Controlled Fatigue Testing³

F 77 Test Method for Apparent Density of Ceramics for Electron Device and Semiconductor Application⁹

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).

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.

Suggestions as a minimum guide in measuring the radiation 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 $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ 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 multi-channel 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 attenuator (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 specimen with