

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Guide for Neutron Irradiation of Unbiased Electronic Components¹

This standard is issued under the fixed designation F 1190; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide strictly applies only to the exposure of unbiased silicon (SI) or gallium arsenide (GaAs) semiconductor components (integrated circuits, transistors, and diodes) to neutron radiation from a nuclear reactor source to determine the permanent damage in the components. Validated 1-MeV damage functions codified in National Standards are not currently available for other semiconductor materials.

1.2 Elements of this guide with the deviations noted may also be applicable to the exposure of semiconductors comprised of other materials except that validated 1-MeV damage functions codified in National standards are not currently available.

1.3 Only the conditions of exposure are addressed in this guide. The effects of radiation on the test sample should be determined using appropriate electrical test methods.

1.4 This guide addresses those issues and concerns pertaining to irradiations with reactor spectrum neutrons.

1.5 System and subsystem exposures and test methods are not included in this guide.

1.6 This guide is applicable to irradiations conducted with the reactor operating in either the pulsed or steady-state mode. The range of interest for neutron fluence in displacement damage semiconductor testing range from approximately 10^9 to 10^{16} n/cm².

1.7 This guide does not address neutron-induced single or multiple neutron event effects or transient annealing.

1.8 This guide provides an alternative to Test Method 1017.3, Neutron Displacement Testing, a component of MIL-STD-883 and MIL-STD-750. The Department of Defense has restricted use of these MIL-STDs to programs existing in 1995 and earlier.

1.9 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- E 170 Terminology Relating to Radiation Measurements and Dosimetry²
- E 264 Test Method for Determining Fast-Neutron Reaction Rates by Radioactivation of Nickel²
- E 265 Test Method for Measuring Reaction Rates and Fast-Neutron Fluences by Radioactivation of Sulfur–32²
- E 668 Practice for Application of Thermoluminescence Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices²
- E 720 Guide for Selection and Use of Neutron-Activation Foils for Determining Neutron Spectra Employed in Radiation-Hardness Testing of Electronics²
- E 721 Method for Determining Neutron Energy Spectra with Neutron-Activation Foils for Radiation-Hardness Testing of Electronics²
- E 722 Practice for Characterizing Neutron Energy Fluence Spectra in Terms of an Equivalent Monoenergetic Neutron Fluence for Radiation-Hardness Testing of Electronics²
- E 1249 Practice for Minimizing Dosimetry Errors in Radiation Hardness Testing of Silicon Electronic Devices Using Co-60 Sources²
- E 1250 Test Method for Application of Ionization Chambers to Assess the Low Energy Gamma Component of Cobalt-60 Irradiators Used in Radiation-Hardness Testing of Silicon Electronic Devices²
- E 1854 Practice for Ensuring Test Consistency in Neutron-Induced Displacement Damage of Electronic Parts²
- F 980 Guide for the Measurement of Rapid Annealing of Neutron-Induced Displacement Damage in Semiconductor Devices.³
- F 1892 Guide for Ionizing Radiation (Total Dose) Effects Testing of Semiconductor Devices³
- 2.2 Other Documents:

2.2.1 The Department of Defense publishes every few years a compendium of nuclear reactor facilities that may be suitable for neutron irradiation of electronic components:

DASIAC SR-94-009, April 1996, Guide to Nuclear Weapons Effects Simulation Facilities and Techniques⁴

2.3 The Office of the Federal Register, National Archives and Records Administration publishes several documents that

¹ This practice is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.11 on Quality and Hardness Assurance.

Current edition approved Jan. 10, 1999. Published March 1999. Originally published as F 1190 – 88. Last previous edition F 1190 – 93.

² Annual Book of ASTM Standards, Vol 12.02.

³ Annual Book of ASTM Standards, Vol 10.04.

⁴ Available from Defense Special Weapons Agency, Washington, DC 20305-1000.

delineate the regulatory requirements for handling and transporting radioactive semiconductor components:

- Code of Federal Regulations: Title 10 (Energy), Part 20, Standards for Protection Against Radiation⁵
- Code of Federal Regulations: Title 10 (Energy), Part 30, Rules of General Applicability to Domestic Licensing of Byproduct Material⁵
- Code of Federal Regulations: Title 49 (Transportation), Parts 100 to 177^5

3. Terminology

3.1 *1 MeV equivalent fluence*—this expression is used by the radiation-hardness testing community to refer to the characterization of an incident neutron energy fluence spectrum, $\Phi(E)$, in terms of the fluence of monoenergetic neutrons at 1 MeV energy required to produce the same displacement damage in a specified irradiated material as $\Phi(E)$ (see Practice E 722 for details).

3.1.1 *Discussion*—Historically, the material has been assumed to be silicon (Si). The emergence of gallium arsenide (GaAs) as a significant alternate semiconductor material, whose radiation damage effects mechanisms differ substantially from Si based devices, requires that future use of the 1 MeV equivalent fluence expression include the explicit specification of the irradiation semiconductor material.

3.2 *silicon damage equivalent (SDE)*—expression synonymous with "1 MeV equivalent fluence in silicon."

3.3 equivalent monoenergetic neutron fluence $(\Phi_{eq,Eref, mat.})$ —an equivalent monoenergetic neutron fluence that characterizes an incident energy-fluence spectrum, $\Phi(E)$, in terms of the fluence of monoenergetic neutrons at a specific energy, Eref, required to produce the same displacement damage in a specified irradiated material, mat (see Practice E 722 for details).

3.3.1 *Discussion*—The appropriate expressions for commonly used 1 MeV equivalent fluence are $\Phi_{eq, 1 \text{ MeV}, \text{Si}}$ for silicon semiconductor devices and $\Phi_{eq, 1 \text{ MeV}, \text{GaAs}}$ for gallium arsenide based devices. See Practice E 722 for a more thorough treatment of the meaning and significant limitations imposed on the use of these expressions.

4. Summary of Guide

4.1 Evaluation of neutron radiation-induced damage in semiconductor components and circuits requires that the following steps be taken:

4.1.1 Select a suitable reactor facility where the radiation environment and exposure geometry desired are both available and currently characterized (within the last 15 months). A reasonably complete list is contained in DASIAC SR-94-009. Practice E 1854 contains detailed guidance to assist the user in selecting a reactor facility that is certified to be adequately calibrated.

4.1.2 Prepare test plan and fixtures,

4.1.3 Conduct pre-irradiation electrical test of the test sample,

- 4.1.4 Expose test sample and dosimeters,
- 4.1.5 Retrieve irradiated test sample,
- 4.1.6 Read dosimeters,
- 4.1.7 Conduct post-irradiation electrical tests, and

4.1.8 Repeat 4.1.4 through 4.1.7 until the desired cumulative fluence is achieved or until degradation of the test device will not allow any further useful data to be taken.

4.2 Operations addressed in this guide are only those relating to reactor facility selection, irradiation procedure and fixture development, positioning and exposure of the test sample, and shipment of the irradiated samples to the parent facility. Dosimetry methods are covered in existing ASTM standards referenced in Section 2, and many pre- and post-exposure electrical measurement procedures are contained in the literature. Dosimetry is usually supplied by the reactor facility.

5. Significance and Use

5.1 Semiconductor devices are permanently damaged by reactor spectrum neutrons. The effect of such damage on the performance of an electronic component can be determined by measuring the component electrical characteristics before and after exposure to fast neutrons in the neutron fluence range of interest. The resulting data can be utilized in the design of electronic circuits that are tolerant of the degradation exhibited by that component.

5.2 This guide provides a method by which the exposure of silicon and gallium arsenide semiconductor devices to neutron irradiation may be performed in a manner that is repeatable and which will allow comparison to be made of data taken at different facilities.

5.3 For semiconductors other than silicon and gallium arsenide, this guide provides a method that can improve consistency in the measurements and assurance that data from various facilities can be compared on the same equivalence fluence scale when the applicable validated 1-MeV damage functions are codified in National standards. In the absence of a validated 1-MeV damage function, the non-ionizing energy loss (NIEL) as a function incident neutron energy, normalized to the NIEL at 1 MeV, may be used as an approximation. See Practice E 722 for a description of the method.

6. Interferences

6.1 Gamma Effects:

6.1.1 All nuclear reactors produce gamma radiation coincident with the production of neutrons. Gamma rays are produced in the fission process directly and are emitted by fission products and activated materials. Furthermore, these gamma rays produce secondary gamma rays and fluorescence photons in reactor fuel, moderator, and surrounding materials. Consequently, degradation in piecepart performance may be produced by gamma rays as well as neutrons, and because of the softer photon spectra dose enhancement may be a problem. If a separation of neutron (*n*) and gamma ray (γ) degradation is desired, either the n/γ ratio must be increased to the point at which gamma effects are negligible or the test sample degradation must first be characterized in a "pure" gamma ray environment under zero bias conditions. The use of such data from a gamma ray exposure to separate neutron and gamma

⁵ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

effects obtained during a neutron exposure may be a complex task. If this approach is taken, Guide F 1892 should be used as a reference. Guides E 1249 and E 1250 should be used to address dose enhancement issues.

6.1.2 TRIGA-type reactors (Training Research and Isotope production reactor manufactured by General Atomics) deliver gamma dose during neutron irradiations that can vary considerably depending on the immediately preceding operating history of the reactor. A TRIGA-type reactor that has been operating at a high power level for an extended period prior to the semiconductor component neutron irradiation will contain a larger fission product inventory that will contribute significantly higher gamma dose than a reactor that has had no recent high level operations. The experimenter must determine the maximum gamma dose his experiment can tolerate, and advise the facility operator to provide sufficient shielding to meet this limit.

6.2 *Temperature Effects*—Annealing of neutron damage is enhanced at elevated temperatures. Elevated temperatures may occur during irradiation, transportation, storage, or electrical characterization of the test devices.

6.3 Dosimetry Errors- Neutron fluence is typically reported in terms of an equivalent 1 MeV monoenergetic neutron fluence in the specified irradiated material ($\Phi_{\rm eq,\ 1\ MeV,\ Si}$ or $\Phi_{eq, 1 MeV, GaAs}$) in units of neutrons per square centimeter. ASTM guidelines and standards exist for calculating this value from measured reactor characteristics. However, reactor facilities may not routinely remeasure the neutron spectrum, (using Guide E 720 and Method E 721) at the test sample exposure sites. A currently valid determination of the neutron spectrum is needed to provide the essential data to accurately ascertain the equivalent 1 MeV monoenergetic neutron fluence in the specified irradiated material. Lack of this critical data can result in substantial error. Therefore the experimenter must obtain a current valid determination of the 1 MeV equivalent fluence in silicon or GaAs, as needed, from the reactor facility operator. This may require a recharacterization of the reactor test facility, or the particular test configuration.

6.4 *Recoil Ionization Effects*—Ionization effects from neutron recoils within a semiconductor device may be significant for some device types at very high neutron fluences, although under normal conditions, ionization due to the gamma radiation from the source will be much greater than the ionization from recoils.

6.5 *Test Configuration Effects*—Extraneous materials in the vicinity of the test specimens can modify the environment at the test sample location. Both the neutron spectrum and the gamma field can be altered by the presence of such material even if these materials are not interposed between the reactor core and the test devices.

6.6 *Thermal Neutron Effects*—Fast Burst Reactor (FBR) neutron spectra have a small thermal neutron component; however, TRIGA reactors inherently produce a very large thermal neutron flux. Neutrons interact with the materials of the devices being irradiated causing them to become radioactive. Thermal neutrons generally induce higher levels of radioactivity. As a consequence, parts irradiated at TRIGA reactors to moderate or high levels should not be handled or

measured soon after exposure. It is therefore common practice at TRIGA reactors to shield test parts from the thermal neutrons with borated polyethylene or cadmium shields. Cadmium capture of thermal neutrons produces more gamma rays than boron capture, thus producing a lower n/γ ratio when such a shield is used. For this reason, borated polyethylene shields are preferred. While most facilities providing neutron irradiation for semiconductor parts will automatically provide the thermal neutron shields, it is the experimenter's responsibility to verify that such a shield is employed during the irradiation.

7. Procedure

7.1 Reactor Facility Selection:

7.1.1 Reactor Operating Modes and Fluence Levels—Two types of reactors are generally used for evaluating the displacement effects of neutrons on electronic components. These reactors, the FBR and the TRIGA types, can be operated in either a pulsed or a steady-state mode. The minimum pulse width for the FBR is approximately 50 µs and the TRIGA type has a nominal pulse width of 10 ms. No rate dependence of permanent displacement damage has been observed at these facilities. In the single-pulse mode, the FBR typically has a maximum fluence ($\Phi_{eq, 1 MeV, Si}$) up to 8×10^{13} n/cm² outside the core and 6×10^{14} n/cm² inside the core. TRIGA-type reactors have a maximum single pulse fluence that varies with the reactor and the exposure position within the core, but ranges from 5×10^{13} to 3×10^{15} n/cm². The in-core volume available for semiconductor components for most FBR reactors and TRIGA type reactors is on the order of 100 cm³. Significantly larger core volumes are available at some facilities. Higher fluences can be achieved by exposing the sample to multiple bursts or by operating the reactor in a steady-state mode. In the steady-state mode, the FBR can deliver fluxes on the order of 1.8×10^{11} n/cm²s outside the core and 7.8×10^{11} n/cm²s inside the core, while the TRIGA-type reactor can deliver maximum fluxes ranging from approximately 2.2×10^{11} to 2.5×10^{12} n/cm²s.

7.1.2 Neutron Fluence/Gamma Dose (η/γ) Ratio. In addition to a flux of neutrons, reactors produce a gamma-ray environment. In order to be sure that the observed radiation effects are due to neutrons, it is necessary that the η/γ ratio is sufficiently large that the gamma damage is small compared to the neutron displacement damage. In the pulse mode, the inherent η/γ ratios for the FBR and TRIGA-type reactors are approximately 4.5×10^9 and 3×10^8 [n/cm² per rad(Si)], respectively. These ratios can be increased or decreased by interposing shielding between the sample and the reactor. In general, the η/γ ratio decreases as the distance from the reactor is increased. The η/γ ratio tends to be lower for exposures using TRIGA-type reactors in the steady-state rather than pulse mode of operation, and also for exposures at lower rather than higher steady-state power levels as the fraction of the total gamma dose attributable to the preexisting fission product inventory increases as the total exposure time increases.

7.1.3 *Dosimetry and Field Mapping*. Mechanical supports or reactor control elements may cause localized perturbation of the neutron flux; therefore, mapping of the area in which samples are to be exposed is required to verify uniformity. Use sulfur or nickel dosimetry for mapping in accordance with Test

Method E 264 or E 265. Report the resulting neutron fluence in terms of the 1 MeV equivalent neutron fluence in the specified irradiated material ($\Phi_{eq, 1 \text{ MeV, Si}}$ or $\Phi_{eq, 1 \text{ MeV, GaAs}}$) in accordance with Practice E 722.

7.1.4 In the absence of a validated 1-MeV damage function, the non-ionizing energy loss (NIEL) as a function incident neutron energy, normalized to the NIEL at 1 MeV, may be used as an approximation. See Practice E 722 for a description of the method. Concurrent with the neutron mapping, determine the gamma total dose at the exposure location using Thermoluminescent Dosimeter (TLD) dosimetry. Practice E 668 provides good general guidance on the handling and use of TLDs; however, it specifically excludes use in a mixed neutron/ gamma exposure field. The facility operator should make appropriate independent measurements to derive a correction factor for the effect of neutrons in the TLD results should be obtained from the facility. Because the neutron energy spectrum extends to thermal energy levels and because lithium (⁶Li) has a large absorption cross section for thermal neutrons, the use of CaF₂ rather than LiF TLD's is recommended to avoid a potential error in the gamma dose measurement. CaF₂ is also a better match for energy absorption of semiconductor materials. Keep in mind the warning in 6.1.2.

7.2 Test Plan and Fixtures:

7.2.1 All reactor facilities require a test procedure or test plan. The procedure should specify the location of the test sample relative to the reactor core and the desired 1-MeV equivalent fluence. The test facility may need only the required fluence, from which the location of the sample and burst temperature will be determined by facility operating personnel. In the steady-state mode, the power level and duration of exposure are required. This too can be provided by facility operators if desired fluence is given. Plan the exposures such that placement of the test sample in the exposure area can be accomplished quickly with minimal reentry requirements to minimize exposure of test personnel.

7.2.2 Design test fixtures to enable accurate and repeatable positioning of the test sample for tests in which multiple exposures are made. Also design the test fixture with minimum mass to prevent perturbation of the flux field. Avoid hydrogenous materials because of the resulting degradation in n/γ ratio and the softening of the neutron spectrum. In addition, at FBR facilities large amounts of hydrogenous material will reflect neutrons back to the core and may require considerable experimentation by the facility operator in order to characterize, and hence control, the operation of the reactor with the test fixture in place. The experimenter should also be aware that certain materials, some of which are used as electrical insulation (for example, TFE-fluorocarbon), degrade in a reactor environment. Aluminum is commonly used to construct test fixtures.

7.3 Exposure of Test Sample and Dosimeters:

7.3.1 Mount test samples on panels of convenient dimensions for ease in handling. Sulfur (or nickel) and TLD dosimeters may be attached, as required, to the panels prior to exposure. In general, use an array of dosimeters if the nonuniformity of the environment is expected to exceed \pm 10 % over the test article or sample group. An exposure

geometry should be chosen such that the total variation in fluence observed at the test sample sites does not exceed \pm 20 %.

7.3.2 Semiconductor piece parts may be irradiated passively because displacement damage is independent of applied bias.

NOTE 1—Transient annealing of damage immediately following exposure of parts to pulsed neutron environment may be strongly affected by bias conditions following exposure. Refer to Guide F 980 for a method of characterizing rapid annealing effects. Mount the test samples unbiased. For MOS devices or any microcircuit containing an MOS element, all leads shall be shorted. For all other device technologies, the leads may be either open or shorted.

7.3.3 If static-sensitive parts are to be irradiated, use standard electrostatic discharge (ESD) protective procedures in handling these parts. However, as a general rule, protect the leads of *all* semiconductor devices during irradiation to prevent damage resulting from electrostatic discharge. This protection usually consists of placing the devices in conductive foam or shorted sockets.

7.3.4 All exposures shall be conducted at ambient temperatures between room temperature (that is, $24 \pm 6^{\circ}$ C) and 50° C unless otherwise specified.

7.3.5 The temperature of the sample devices should be maintained below 50° C from the time of the exposure until the post-electrical tests are made. If the temperature exceeds 50° C between the time of irradiation and the electrical measurements some correction may be required to account for annealing. The post-exposure electrical tests as specified shall be made within 30 days.

7.3.6 Significant postirradiation annealing of damage occurs immediately following irradiation (seconds through \sim 2 days depending on device composition and structure) and then continues at a much slower rate for months. The amount and rate of annealing depends on the semiconductor temperature and on the time duration at elevated temperatures. Such annealing will affect the results of the post-exposure electrical tests. It is recommended that postirradiation electrical tests be performed a minimum of 2 days following exposure.

7.4 Retrieval and Return of Test Sample:

7.4.1 Following exposure of the test samples, the parts may be retrieved after the radioactivity of the test chamber has reached a safe level. Facility Health Protection (Radiation Safety) personnel typically determine when re-entry is permissible based upon the hazard exposure limits in CFR 20. Retrieve the test samples and dosimeters quickly to minimize personnel exposure. Following retrieval, separate the dosimeters and turn them over to personnel responsible for reading the dosimeters. This service is generally provided by the reactor facility staff by prior arrangement.

7.5 Electrical Characterization:

7.5.1 Pre- and post-irradiation electrical measurements are made in accordance with the appropriate electrical test procedures. Selection of test parameters and bias conditions requires knowledge of basic radiation effects on the device technology being tested and may require knowledge of the intended application of the test device.

7.5.2 Electrical tests may be performed at the reactor facility or another facility. After exposure, the samples are likely to be radioactive for a period ranging from several days to months and must be handled in accordance with appropriate health safety procedures, referenced in 10 CFR 20, or until declared nonradioactive by a certified radiation health physicist.

8. Packaging and Package Marking

8.1 Radioactive test samples that are to be shipped to another facility must be packaged in accordance with applicable regulations pertaining to the shipment of radioactive material, referenced in 49 CFR 100-177. It is also important to note that the receiving facility must be licensed in accordance with governing Federal Regulations, referenced in 10 CFR 30, to receive radioactive material.

9. Report

9.1 In describing the results of a neutron irradiation report the following information:

9.1.1 Reactor identification,

9.1.2 Reactor operating mode-pulse or steady-state,

9.1.3 Core configuration used for irradiation,

9.1.4 Times and number of pulses or steady-state runs,

9.1.5 Shielding details,

9.1.6 Sulfur (or nickel) dosimeter readings,

9.1.7 TLD dosimeter readings, if any,

9.1.8 Fluence as determined using Practice E 772,

9.1.9 Total dose as determined using Practice E 668 and correlated to current exposure,

9.1.10 Sample identification (including part type number, serial number, manufacturer, controlling specification, the date code, and any other identifying numbers given by the manufacturer),

9.1.11 Date and time of exposure to neutrons,

9.1.12 Diagrams of the electrical parameter measurement circuits,

9.1.13 Electrical measurement data before and after irradiation,

9.1.14 Date, time, and temperature at which electrical measurements were made,

9.1.15 Date, time, and temperature history (particularly important is the maximum temperature and its duration) of any periods between the time of irradiation and the post-exposure electrical measurements where the samples exceeded 50° C.

9.1.16 Reactor facility and exposure site radiation environment characterization based on Practice E 1854, method used, and date completed,

9.1.17 Core configuration used for calibration,

9.1.18 Sulfur (or nickel) fluence multiplier used to calculate the 1 MeV equivalent fluence for the irradiated material associated with the current reactor facility calibration for the specific exposure site used, and

9.1.19 Geometry of radiation exposure.

9.1.20 Any anomalous incidents during the test shall be fully documented.

10. Keywords

10.1 dosimetry; electronic component; equivalent monoenergetic neutron fluence; fast burst reactor (FBR); gallium arsenide; gamma dose; gamma effects; irradiation; neutron fluence; neutron flux; nickel; 1 MeV equivalent fluence; radiation; reactor; semiconductor; silicon; sulfur; thermoluminescent dosimeter (TLD); TRIGA-type reactor

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.