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Standard Test Method for Application and Analysis of Helium Accumulation Fluence Monitors for Reactor Vessel Surveillance, E706 (IIIC)[1]

This standard is issued under the fixed designation E 910; 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 test method describes the concept and use of helium accumulation for neutron fluence dosimetry for reactor vessel surveillance. Although this test method is directed toward applications in vessel surveillance, the concepts and techniques are equally applicable to the general field of neutron dosimetry. The various applications of this test method for reactor vessel surveillance are as follows:

1.1.1 Helium accumulation fluence monitor (HAFM) capsules,

1.1.2 Unencapsulated, or cadmium or gadolinium covered, radiometric monitors (RM) and HAFM wires for helium analysis,

1.1.3 Charpy test block samples for helium accumulation, and

1.1.4 Reactor vessel (RV) wall samples for helium accumulation.

1.2 *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:*

C 859 Terminology Relating to Nuclear Materials[2]

E 170 Terminology Relating to Radiation Measurements and Dosimetry[3]

E 184 Practice for Effects of High-Energy Neutron Radiation on the Mechanical Properties of Metallic Materials, E 706 (IB)[4]

E 244 Test Method for Atom Percent Fission in Uranium and Plutonium Fuel (Mass Spectrometric Method)[2]

E 261 Test Method for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques[3]

E 482 Guide for Application of Neutron Transport Methods for Reactor Vessel Surveillance, E 706 (IID)[4]

E 560 Practice for Extrapolating Reactor Vessel Surveillance Dosimetry Results, E 706 (IC)[4]

E 693 Practice for Characterizing Neutron Exposures in Iron and Low Alloy Steels in Terms of Displacements Per Atom (DPA), E 706 (ID)[4]

E 706 Master Matrix for Light-Water Reactor Pressure Vessel Surveillance Standards[4]

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E 706 (IIC)[4]

E 853 Practice for Analysis and Interpretation of Light-Water Reactor Surveillance Results, E 706 (IA)[4]

E 854 Test Method for Application and Analysis of Solid State Track Recorder (SSTR) Monitors for Reactor Surveillance, E 706 (IIIB)[4]

E 900 Guide for Predicting Neutron Radiation Damage to Reactor Vessel Materials, E 706 (IIF)[4]

E 944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance, E 706 (IIA)[4]

E 1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance, E 706 (IIIA)[4]

E 1018 Guide for Application of ASTM Evaluated Cross Section Data File, E 706 (IIB)[4]

E 1214 Guide for Use of Melt Wire Temperature Monitors for Reactor Vessel Surveillance, E 706 (IIIE)[4]

E 2005 Guide for the Benchmark Testing of Reactor Dosimetry in Standard and Reference Neutron Fields, E 706 (IIE-1)[4]

E 2006 Guide for the Benchmark Testing of LWR Calculations, E 706 (IIE-2)[4]

3. Terminology

3.1 *Definitions*—For definition of terms used in this test method, refer to Terminology C 859 and E 170. For terms not defined therein, reference may be made to other published glossaries.[5]

[1] This test method is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.05 on Nuclear Radiation Metrology.

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[2] *Annual Book of ASTM Standards*, Vol 12.01.

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

[4] *Annual Book of ASTM Standards*, Vol 12.02. The roman numeral-alphabetical designation at the end of this title indicates that a brief description of this standard may be found in Matrix E 706.

[5] See *Dictionary of Scientific Terms*, 3rd Edition, Sybil P. Parker, Ed., McGraw Hill, Inc.

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4. Summary of the HAFM Test Method

4.1 Helium accumulation fluence monitors (HAFMs) are passive neutron dosimeters that have a measured reaction product that is helium. The monitors are placed in the reactor locations of interest, and the helium generated through (n,α) reactions accumulates and is retained in the HAFM (or HAFM capsule) until the time of removal, perhaps many years later. The helium is then measured very precisely by high-sensitivity gas mass spectrometry **(1, 2)**.[6] The neutron fluence is then directly obtained by dividing the measured helium concentration by the spectrum-averaged cross section. Competing helium producing reactions, such as (γ,α) do not, except for ^{9}Be(γ,α), affect the HAFM results. The range of helium concentrations that can be accurately measured in irradiated HAFMs extends from 10^{-14} to 10^{-1} atom fraction. This range permits the HAFMs to be tested in low fluence environments yet to work equally well for high fluence situations.

4.2 Typically, HAFMs are either individual small solid samples, such as wire segments **(3)** or miniature encapsulated samples of small crystals of powder **(4)**, as shown in Fig. 1. As with radiometric dosimetry, different materials are used to provide different energy sensitivity ranges. Encapsulation is necessary for those HAFM materials and reactor environment combinations where sample melting, sample contamination, or loss of generated helium could possibly occur. Additionally, encapsulation generally facilitates the handling and identification of the HAFM both prior to and following irradiation. The contents of HAFM capsules typically range from 0.1 to 10 mg.

4.3 Following irradiation, encapsulated HAFMs are cleaned and identified in preparation for helium analysis. Helium analysis is then accomplished by vaporizing both the capsule and its contents and analyzing the helium in the resulting gases in a high sensitivity mass spectrometer system **(5)**. The amount of ^{4}He is determined by measuring the ^{4}He-to-^{3}He isotopic ratio in the sample gases subsequent to the addition of an accurately calibrated amount of ^{3}He "spike." Unencapsulated HAFMs, for example, pure element wires, are usually etched to remove a predetermined layer of outer material before helium analysis **(3)**. This eliminates corrections for both cross contamination between samples and α-recoil into or out of the sample during the irradiation.

4.4 The ^{4}He concentration in the HAFM, in general terms, is proportional to the incident neutron fluence. Consideration must, however, be made for such factors as HAFM material burnup, neutron self-shielding and flux depression, α-recoil, and neutron gradients. Corrections for these effects are discussed more fully in Section 13. Generally, they total less than 5 % of the measured helium concentration. Since the individual corrections are usually known to within 50 %, the total error from these corrections amounts to ≤2 %. Sources of uncertainty also lie in the HAFM material mass, isotopic composition, and mass spectrometric helium analysis. As indicated in Section 13, however, these uncertainties generally contribute less than 1 % of the total uncertainty for routine analyses.

4.5 Applying the above corrections to the measured HAFM helium concentration, the total incident neutron fluence (over the energy range of sensitivity of the HAFM) can be obtained directly from a knowledge of the spectrum-integrated total helium production cross section for the particular irradiation environment. At the present time, the uncertainty in the derived neutron fluence is mainly due to uncertainty in the spectrum-integrated cross section of the HAFM sensor material rather than the combined uncertainties in the helium determination process. This situation is expected to improve as the cross sections are more accurately measured, integrally tested in benchmark facilities **(6)**, and reevaluated.

5. Significance and Use

5.1 The HAFM test method is one of several available passive neutron dosimetry techniques (see, for example, Methods E 854 and E 1005). This test method can be used in combination with other dosimetry methods, or, if sufficient data are available from different HAFM sensor materials, as an alternative dosimetry test method. The HAFM method yields a direct measurement of total helium production in an irradiated sample. Absolute neutron fluence can then be inferred from this, assuming the appropriate spectrum integrated total helium production cross section. Alternatively, a calibration of the composite neutron detection efficiency for the HAFM method may be obtained by exposure in a benchmark neutron field where the fluence and spectrum averaged cross section are both known (see Matrix E 706 IIE).

[6] The boldface numbers in parentheses refer to the list of references appended to this test method.

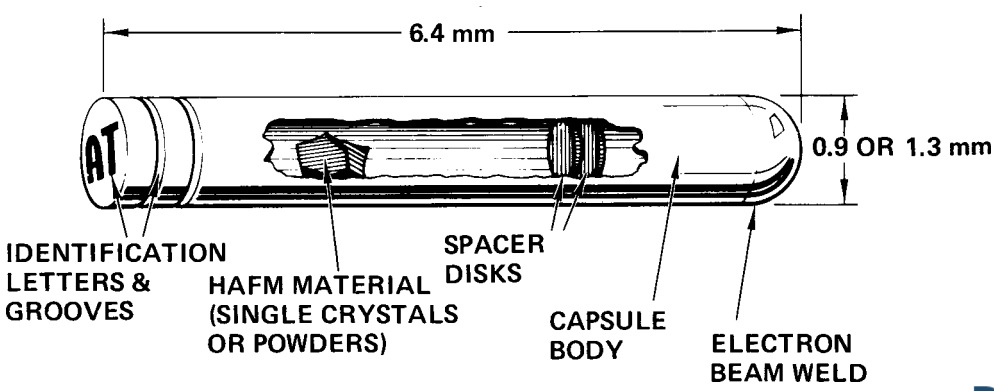

FIG. 1 Helium Accumulation Fluence Monitor Capsule

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5.2 HAFMs have the advantage of producing an end product, helium, which is stable, making the HAFM method very attractive for both short-term and long-term fluence measurements without requiring time-dependent corrections for decay. HAFMs are therefore ideal passive, time-integrating fluence monitors. Additionally, the burnout of the daughter product, helium, is negligible.

5.2.1 Many of the HAFM materials can be irradiated in the form of unencapsulated wire segments (see 1.1.2). These segments can easily be fabricated by cutting from a standard inventoried material lot. The advantage is that encapsulation, with its associated costs, is not necessary. In several cases, unencapsulated wires such as Fe, Ni, Al/Co, and Cu, which are already included in the standard radiometric (RM) dosimetry sets (Table 1) can be used for both radiometric and helium accumulation dosimetry. After radiometric counting, the samples are later vaporized for helium measurement.

5.3 The HAFM method is complementary to RM and solid state track recorder (SSTR) foils, and has been used as an integral part of the multiple foil method. The HAFM method follows essentially the same principle as the RM foil technique, which has been used successfully for accurate neutron dosimetry for the past 20 to 25 years. Various HAFM sensor materials exist which have significantly different neutron energy sensitivities from each other. HAFMs containing ^{10}B and ^{6}Li have been used routinely in LMFBR applications in conjunction with RM foils. The resulting data are entirely compatible with existing adjustment methods for radiometric foil neutron dosimetry (refer to Method E 944).

5.4 An application for the HAFM method lies in the direct analysis of pressure vessel wall scrapings or Charpy block surveillance samples. Measurements of the helium production in these materials can provide in situ integral information on the neutron fluence spectrum. This application can provide dosimetry information at critical positions where conventional dosimeter placement is difficult if not impossible. Analyses must first be conducted to determine the boron, lithium, and other component concentrations, and their homogeneities, so that their possible contributions to the total helium production can be determined. Boron (and lithium) can be determined by converting a fraction of the boron to helium with a known thermal neutron exposure. Measurements of the helium in the material before and after the exposure will enable a determination of the boron content (**7**). Boron level down to less than 1 wt. ppm can be obtained in this manner.

5.5 By careful selection of the appropriate HAFM sensor material and its mass, helium concentrations ranging from $\sim 10^{-14}$ to 10^{-1} atom fraction can be generated and measured. In terms of fluence, this represents a range of roughly 10^{12} to 10^{27} n/cm^2. Fluence (>1 MeV) values that may be encountered during routine surveillance testing are expected to range from $\sim 3 \times 10^{14}$ to 2×10^{20} n/cm^2, which is well within the range of the HAFM technique.

5.6 The analysis of HAFMs requires an absolute determination of the helium content. The analysis system specified in this test method incorporates a specialized mass spectrometer in conjunction with an accurately calibrated helium spiking system. Helium determination is by isotope dilution with subsequent isotope ratio measurement. The fact that the helium is stable makes the monitors permanent with the helium analysis able to be conducted at a later time, often without the inconvenience in handling caused by induced radioactivity. Such systems for analysis exist, and additional analysis facilities could be reproduced, should that be required. In this respect, therefore, the analytical requirements are similar to other ASTM test methods (compare with Test Method E 244).

6. Apparatus

6.1 *High-Sensitivity Gas Mass Spectrometer System*, capable of vaporizing both unencapsulated and encapsulated HAFM materials and analyzing the resulting total helium content is required. A description of a suitable system is contained in Ref (**5**).

6.2 *Analytical Microbalance for Accurate Weighing of HAFM Samples*, minimum specifications: 200-mg capacity with an absolute accuracy of ±0.5 µg. Working standard

TABLE 1 Neutron Characteristics of Candidate HAFM Materials for Reactor Vessel Surveillance

HAFM Sensor Material	Principal Helium Producing Reaction	Thermal Neutron Cross Section, (b)	Fission Neutron Spectrum	
			Cross Section, (mb)[A]	90 % Response Range, (MeV)[A]
Li	$^{6}Li(n,\alpha)T$	942	457	0.167–5.66
Be	$^{9}Be(n,\alpha)^{6}He$;ra^{6}Li	...	284	2.5–7.3
B	$^{10}B(n,\alpha)^{7}Li$	3838	494	0.066–5.25
N	$^{14}N(n,\alpha)^{11}B$	...	86.2	1.7–5.7
F	$^{19}F(n,\alpha)^{16}N$	...	27.6	3.7–9.7
Al[B]	$^{27}Al(n,\alpha)^{24}Na$	...	0.903	6.47–11.9
S	$^{32}S(n,\alpha)^{29}Si$	...	...	...
Cl	$^{35}Cl(n,\alpha)^{32}P$	...	~13 (Cl)	2.6–8.3
Ti[B]	$^{47}Ti(n,\alpha)^{44}Ca$	...	0.634 (Ti)	6.5–12.8
Fe[B]	$^{56}Fe(n,\alpha)^{53}Cr$	...	0.395 (Fe)	5.2–11.9
Ni[B]	$^{58}Ni(n,\alpha)^{55}Fe$	...	5.58 (Ni)	3.9–10.1
Cu[B]	$^{63}Cu(n,\alpha)^{60}Co$	...	0.330	4.74–11.1
316–SS PV Steel Charpy Block	Helium Production Largely from ^{56}Fe and ^{58}Ni			

[A] Evaluated ^{235}U fission neutron spectrum averaged helium production cross section and energy range in which 90 % of the reactions occur. All values are obtained from ENDF/B-V Dosimetry File data. Bracketed terms indicate cross section is for naturally occurring element.

[B] Often included in dosimetry sets as a radiometric monitor, either as a pure element foil or wire or, in the case of aluminum, as an allaying material for other elements.

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masses must be traceable to appropriate national or international mass standards. Additionally, a general purpose balance with a capacity of at least 200 g and an accuracy of 0.1 mg is required for weighing larger specimens.

6.3 *Laminar flow (optional) clean benches*, for use in the preparation of HAFM samples and capsules.

6.4 *Stereo microscope*, with 7 to 30 magnification, a ~0.1-mm graticule, and an optical illuminator.

6.5 *Electron beam welder*, with moveable platform stage, for sealing HAFM capsules, minimum specifications: variable beam power to 0 to 1 kW, variable beam size capable of focusing down to a diameter of 0.5 mm. Controls must also be available to permit automatic control of beam duration and onset and offset beam power slopes.

6.6 High temperature vacuum furnace for out-gassing HAFM materials, capsules, and mass spectrometer system furnace components. Minimum specifications: 1000°C at a maximum pressure of 10^{-5} Torr.

6.7 *Micro-sand blaster/cleaner*, for cleaning mass spectrometer vacuum furnace parts.

6.8 *X-ray machine*, for quality assurance test of HAFM capsules. Minimum specifications; 300 kV, 10 mA, 4-mm spot size with control of source distance to 1.0 m and exposure time to 5 min.

6.9 *General Laboratory Supplies*:

6.9.1 *Ultrasonic Cleaner*—100 to 200 W,

6.9.2 *Heat Lamp*—250 W, and

6.9.3 *Optical Pyrometer*—700 to 2000°C.

6.10 *Radioactive Material Handling*:

6.10.1 Lead shielding,

6.10.2 Portable radioactive (β-γ) counters (0.01 mrem/h to 100 rem/h), and

6.10.3 Radioactive waste disposal capability.

6.11 *Reagents and Materials*:

6.11.1 *Hydrochloric Acid* (HCl), (37 %),

6.11.2 *Hydrofluoric Acid* (HF), (48 %),

6.11.3 *Nitric Acid* (HNO_3), (70 %),

6.11.4 *Sulfuric Acid* (H_2SO_4), (96 %),

6.11.5 *Acetone* [$(CH_3)_2CO$]—Reagent grade (>99.7 %),

6.11.6 *Alcohol* (C_2H_5OH)—Pure (200 proof),

6.11.7 *Chloroform* ($CHCl_3$)—Reagent grade (>99.2 %),

6.11.8 *Distilled and Deionized Water*, and

6.11.9 *Detergent Cleaning Solution* (Alconox[7] or equivalent).

7. HAFM Materials

7.1 *General Requirements*—The general requirements concerning the characteristics of HAFM materials fall into two broad categories: (*1*) nuclear properties and (*2*) chemical properties. These two categories are discussed separately below.

7.2 *Nuclear Properties*:

7.2.1 *Helium Production Cross Section*—Consideration must be made for the energy range or energy sensitivity of the (n, total helium) cross section of the potential HAFM sensor material. For any given neutron environment, the set of HAFMs or combination of HAFMs, RM, and SSTR multiple foils must be chosen to cover the entire neutron energy range (refer to Guide E 844). The majority of potential HAFM materials fall into the threshold reaction category. That is, below the threshold energy (usually in the 1–10 MeV range), these materials produce essentially no helium from neutron reactions. Above this energy, however, the (n, total helium) cross section generally rises fairly rapidly to a plateau from where it continues to rise relatively slowly. Generally, the higher the threshold energy, the lower the total cross section. The threshold reaction HAFM isotopes presently identified as being most suitable for reactor vessel surveillance are ^{9}Be, ^{14}N, ^{19}F, ^{27}Al, ^{32}S, ^{35}Cl, ^{56}Fe, ^{58}Ni and ^{63}Cu (see Table 1).

7.2.1.1 The two stable isotopes that have significant nonthreshold helium production cross sections are ^{6}Li and ^{10}B. The cross sections of these two isotopes, which are large and well known, vary inversely with the neutron velocity below about 0.1 MeV. Above 0.1 MeV, the cross section behavior becomes more irregular, with the ^{6}Li exhibiting a significant resonance near 0.24 MeV.

7.2.1.2 Other stable isotopes exist which have nonthreshold helium production cross sections, but all are much less than 1 barn (10^{-24} cm^2). Of the radioactive isotopes, ^{59}Ni, which has a ~12 barn thermal neutron (n,α) cross section, is the only one important for HAFM neutron dosimetry through the two-stage reaction ^{58}Ni(n,γ)·^{59}Ni(n,α)^{56}Fe. Also included in Table 1 are additional potential HAFM materials which are already included in the standard specified RM foil and metallurgical sets (refer to Matrix E 706) and thus may serve a double purpose (see 11.1). These materials include the natural elements Ti, Fe, Cu, and Ni; stainless steel dosimetry capsule material, RV steel; and Charpy block metallurgical specimens. Relevant characteristics of the various HAFM isotopes and materials are listed in Table 1. Aluminum is also often included in RM sets in the form of alloys of Co and Au.

7.2.2 *Activation Cross Sections*—Also to be considered in the selection of HAFM materials is their relative activation cross sections in typical reactor vessel neutron fields. Although activation reactions in general do not interfere with helium production (exceptions are cases of two-stage reactions as with $_{58}$Ni, and cases where daughter products have contributing (n,α) reactions such as ^{9}Be(n,α)^{6}He →^{6}Li), the resulting radioactive decay contributes to post-irradiation handling and analysis difficulties and, to this extent, should be minimized.

7.2.3 *Neutron Self-Shielding*—High cross section isotopes, such as ^{6}Li and ^{10}B, exhibit significant neutron self-shielding and surface flux depression in thermal and epithermal neutron environments. In order to apply these isotopes to reactor surveillance dosimetry, dilution of these materials by alloying is required to reduce their effective isotopic concentrations. Suitable alloying materials for boron and lithium at the 0.1 to 0.5 weight percent level are vanadium, niobium, and aluminum. Additional details on self-shielding are given in Section 13.

7.2.4 *Neutron Screening at Low Energies*—An alternate technique, or one that can be used in conjunction with alloying to reduce neutron self-shielding, is to protect the dosimetry

[7] Alconox is a registered trademark of Alconox Inc., 215 Park Ave. South, New York, NY 10003.

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