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Standard Guide for Investigating the Effects of Helium in Irradiated Metals¹

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1. Scope

1.1 This guide provides advice for conducting experiments to investigate the effects of helium on the properties of metals where the technique for introducing the helium differs in some way from the actual mechanism of introduction of helium in service. Techniques considered for introducing helium may include charged particle implantation, exposure to α -emitting radioisotopes, and tritium decay techniques. Procedures for the analysis of helium content and helium distribution within the specimen are also recommended.

1.2 Three other methods for introducing helium into irradiated materials are not covered in this guide. They are: (1) the enhancement of helium production in nickel-bearing alloys by spectral tailoring in mixed-spectrum fission reactors, (2) a related technique that uses a thin layer of NiAl on the specimen surface to inject helium, and (3) isotopic tailoring in both fast and mixed-spectrum fission reactors. These techniques are described in Refs (1-6).² Dual ion beam techniques (7) for simultaneously implanting helium and generating displacement damage are also not included here. This latter method is discussed in Practice E521.

1.3 In addition to helium, hydrogen is also produced in many materials by nuclear transmutation. In some cases it appears to act synergistically with helium (8-10). The specific impact of hydrogen is not addressed in this guide.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standard-*

ization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 *ASTM Standards:*³

[C859 Terminology Relating to Nuclear Materials](#)

[E170 Terminology Relating to Radiation Measurements and Dosimetry](#)

[E521 Practice for Investigating the Effects of Neutron Radiation Damage Using Charged-Particle Irradiation](#)

[E910 Test Method for Application and Analysis of Helium Accumulation Fluence Monitors for Reactor Vessel Surveillance](#)

3. Terminology

3.1 Descriptions of relevant terms are found in Terminology C859 and Terminology E170.

4. Significance and Use

4.1 Helium is introduced into metals as a consequence of nuclear reactions, such as (n, α), or by the injection of helium into metals from the plasma in fusion reactors. The characterization of the effect of helium on the properties of metals using direct irradiation methods may be impractical because of the time required to perform the irradiation or the lack of a radiation facility, as in the case of the fusion reactor. Simulation techniques can accelerate the research by identifying and isolating major effects caused by the presence of helium. The word ‘simulation’ is used here in a broad sense to imply an approximation of the relevant irradiation environment. There are many complex interactions between the helium produced during irradiation and other irradiation effects, so care must be exercised to ensure that the effects being studied are a suitable approximation of the real effect. By way of illustration, details of helium introduction, especially the implantation temperature, may determine the subsequent distribution of the helium (that is, dispersed atomistically, in small clusters in bubbles, etc.).

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² The boldface numbers in parentheses refer to a list of references at the end of this guide.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

5. Techniques for Introducing Helium

5.1 *Implantation of Helium Using Charged Particle Accelerators:*

5.1.1 *Summary of Method*—Charged particle accelerators are designed to deliver well defined, intense beams of monoenergetic particles on a target. They thus provide a convenient, rapid, and relatively inexpensive means of introducing large concentrations of helium into thin specimens. An energetic alpha particle impinging on a target loses energy by exciting or ionizing the target atoms, or both, and by inelastic collisions with the target atom nuclei. Particle ranges for a variety of materials can be obtained from tabulated range tables (10-14) or calculated using a Monte Carlo code such as SRIM (15).

5.1.1.1 To obtain a uniform concentration of helium through the thickness of a sample, it is necessary to vary the energy of the incident beam, rock the sample (6), or, more commonly, to degrade the energy of the beam by interposing a thin sheet or wedge of material ahead of the target. The range of monoenergetic particles is described by a Gaussian distribution around the mean range. This range straggling provides a means of implanting uniform concentrations through the thickness of a specimen by superimposing the Gaussian profiles that result from beam energy degradation of different thicknesses of material. The uniformity of the implant depends on the number of superpositions. Charged particle beams have dimensions of the order of a few millimetres so that some means of translating the specimen in the beam or of rastering the beam across the specimen must be employed to uniformly implant specimens of the size required for tensile or creep tests. The rate of helium deposition is usually limited by the heat removal rate from the specimens and the limits on temperature rise for a given experiment. Care must be exercised that phase transformations or annealing of microstructural components do not result from beam heating.

5.1.2 *Limitations*—One of the major limitations of the technique is that the thickness of a specimen that can be implanted with helium is limited to the range of the most energetic alpha particle beam available (or twice the range if the specimen is implanted from both sides). Thus a stainless steel tensile specimen is limited to 1.2 mm thickness using a 70-MeV beam to implant the specimen from both sides. This limiting thickness is greater for light elements such as aluminum and less for heavier elements such as molybdenum.

5.1.2.1 One of the primary reasons for interest in helium implantation is to investigate the effects resulting from the production of helium by transmutation reactions in nuclear reactors. It should be appreciated that the property changes in irradiated metals result from complex interactions between the helium atoms and the radiation damage produced during the irradiation in ways that are not fully understood. Implantation of energetic alpha particles does produce atomic displacements, but in a manner atypical of most neutron irradiations. The displacement rate is generally higher than that in fast reactor, but the ratio of helium atoms to displaced atoms is some 10^3 times greater for implantation of stainless steel with a 50-MeV alpha beam.

5.1.3 *Apparatus*—Apparatus for helium implantation is usually custom designed and built at each research center and

therefore much variety exists in the approach to solving each problem. The general literature should be consulted for detailed information (16-20). Paragraphs 5.1.3 – 5.1.3.4 provide comments on the major components of the helium implantation apparatus.

5.1.3.1 *Accelerator*—Cyclotrons or other accelerators are used for helium implantation experiments because they are well suited to accelerate light ions to the high potentials required for implantation. Typical cyclotron operating characteristics are 20 to 80 MeV with a beam current of 20 μ A at the source. It should be noted, however, that the usable beam current delivered to the specimen is limited by the ability to remove heat from the specimens which restricts beam currents to a limit of 4 to 5 μ A. A beam-rastering system is the most practical method for moving the beam across the sample surface to uniformly implant helium over large areas of the specimen.

5.1.3.2 *Beam Energy Degradation*—The most efficient procedure for implanting helium with an accelerator, because of the time involved in changing the energy, is to operate the accelerator at the maximum energy and to control the depth of the helium implant by degrading the beam energy. This procedure offers the additional advantages that range straggling increases with energy, thus producing a broader depth profile, and the angular divergence of the beam increases as a consequence of the electronic energy loss process, thus increasing the spot size and reducing the localized beam heating nonuniformly. The beam energy degrader requires that a known thickness of material be placed in front of the beam with provisions for remotely changing the thickness and for removal of heat from the beam energy degrader. Acceptable methods include a rotating stepped or wedged wheel, a movable wedge, or a stack of foils. Beam degrader materials can be beryllium, aluminum, or graphite. The wedge or rotating tapered wheel designs provide a continuous change in energy deposition, so as to provide a uniform distribution of helium in the specimen but introduce the additional complexity of moving parts and cooling of thick sections of material. The stacked foil designs are simpler, can be cooled adequately by an air jet, and have well-calibrated thickness. The design must be selected on the basis of experiment purpose and facility flexibility. Concentrations of helium uniform to within $\pm 5\%$ can be achieved by superposition of the depth profiles produced by 25- μ m increments in the thickness of aluminum beam degrader foils. Uniformity of $\pm 10\%$ is recommended for all material experiments for both concentration and spatial uniformity including any increase in spot size from angular divergence. Distributing helium over more limited depth ranges (as, for example, when it is only required to spread helium about the peak region of heavy ion damage, in specimens that will be examined by transmission electron microscopy) can be done by cycling the energy of the helium-implanting accelerator (19) in place of degrader techniques.

5.1.3.3 *Specimen Holder*—The essential features of the specimen holder are provisions for accurately placing the specimen in the beam and for cooling the specimens. Additional features may include systems for handling and irradiating large numbers of specimens to improve the efficiency of the

facility and to avoid handling the specimens until the radioactivity induced during the implantation has had an opportunity to decay. Some method of specimen cooling is essential since a degraded, singly charged beam of average energy of 20 MeV and current of 5 μA striking a 1-cm² nickel target, 0.025 cm thick, deposits 100 W of heat into a mass of 0.22 g. Assuming only radiative heat loss to the surroundings, the resulting rise in temperature would occur at an initial rate of about 1300 K·s⁻¹ and would reach a value of about 2000 K. Techniques used for specimen cooling will depend on whether the implantation is performed in air or in vacuum and on the physical characteristics of the specimen. Conductive cooling with either air or an inert gas may be used if implants are not performed in vacuum. Water cooling is a more effective method of heat removal and permits higher current densities to be used on thick tensile specimens. The specimens may be bonded to a cooled support block or may be in direct contact with the coolant. Care must be exercised to ensure that metallurgical reactions do not occur between the bonding material and the specimen as a consequence of the beam heating, and that hot spots do not develop as a consequence of debonding from thermal expansion of the specimen. Silver conductive paint has been used successfully as a bonding agent where the temperature rise is minimal. Aluminum is recommended in preference to copper for construction of the target holder because of the high levels of radioactivity induced in copper.

5.1.3.4 Faraday Cup and Charge Integration System—A Faraday cup should be used to measure the beam current delivered to the target. A 600 mm long by 50 mm diameter aluminum tube closed on one end makes a satisfactory Faraday cup. An electron suppressor aperture insulated from the Faraday cup and positively charged is necessary to collect the electrons emitted from the degrader foils so as to give accurate beam current readings. Beam current density and beam profile can be determined by reading the current passed by a series of apertures of calibrated size that can be placed in the beam. The target holder assembly must be insulated from its surroundings, and deionized (low conductivity) water must be used for cooling purposes to permit an integration of current delivered to the target and thereby accurately measure the total helium implanted independent of fluctuations in the beam current. A negatively biased aperture must be placed between the target holder and the degrader foils to suppress secondary electrons emitted from the target that would give erroneously high values of total charge deposited on the specimen.

5.1.4 Procedure—Prior to the actual implantation of helium in a specimen, certain standardization and calibration procedures should be performed. The temperature rise to be expected from beam heating and the intended specimen cooling mode must be measured. Such measurements can be performed on dummy specimens using a thermocouple embedded in the sample behind the beam spot or with an infrared pyrometer capable of reading the surface temperature of an area the size of the beam spot. The thickness of the beam energy degrader must be accurately measured to determine the depth of the helium implant. This can be determined from a measurement

of the mean energy of the emergent particles from the degrader using a detector placed directly in the beam line behind the degrader.

5.1.4.1 The uniformity of the fluence rate on the surface of the specimen must be determined for the implant conditions and for each degrader thickness. This is easily done prior to implantation using a small-diameter aperture that can be moved into the centerline of the particle beam to compare the fluence rate on the axis to the average fluence rate on the specimen. The Faraday cup is placed behind this small aperture to measure the current, and the ratio of peak current density on the specimen to the average current density can then be determined for each degrader thickness since the ratio of the area of small aperture to the total implant area is known. An alternative is the use of a commercially available beam profile monitor.

5.1.4.2 The total charge deposited on the specimen by the incident alpha particles must be measured. Precautions must be taken to minimize leakage currents through the cooling water by the use of low conductivity water, to suppress collection of secondary electrons emitted from the target by a negatively biased aperture just ahead of the specimen, and to collect electrons knocked out of the exit surface of the degrader foil by collecting them on a positively charged aperture placed downstream from the beam degrader.

5.1.4.3 Following irradiation the specimens and specimen holder will have high levels of induced activity and precautions must be exercised in handling and storage of the specimens and target holder. Most of this activity is short-lived and decays within a day. The induced activity can be used advantageously to check the uniformity of the implant by standard autoradiographic techniques.

5.1.5 Calculation and Interpretation of Results—The ranges of energetic particles in solid media have been calculated (10-15) for a number of materials. The range increases with increasing energy and is affected by target parameters such as electron density, atomic density, and atomic mass. Ranges are stated in units of mg·cm⁻², which, when divided by the physical density of the target material, in g·cm⁻³ gives a distance in tens of μm . The total range is defined as the total path length from the point of entry at the target surface to the point at which the particle comes to rest. The projected range or penetration depth is defined as the projection of the total range along the normal to the entry face of the target, and is therefore a sensitive function of the angle of incidence of the α particle at the target surface. The concentration of helium in parts per million is defined as the ratio of the number density of helium nuclei to the number density of host material times 10⁶:

$$C_{\text{ppm}} = (M_{\text{He}}/M_{\text{H}}) \times 10^6 \quad (1)$$

$$M_{\text{H}} = N_0 \rho_{\text{H}} / A_{\text{H}} \quad (2)$$

where:

N_0 = Avogadro's number,

A_{H} = gram molecular weight of host material, and

ρ_{H} = its density, g·cm⁻³.

5.1.5.1 The quantity M_{He} (helium density) is a function of the range as given by the range-straggling formula. This

expression has been normalized to a unit particle fluence rate since the total area under a normal distribution curve is equal to $\sigma\sqrt{2\pi}$. If N_T is the total number of particles incident on the surface per unit area (fluence) then:

$$M_{\text{He}} = \frac{N_T}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(R - \bar{R})^2}{2\sigma^2}\right] \quad (3)$$

The peak number density which occurs at the mean range ($R = \bar{R}$) is:

$$M_{\text{He}} = N_T / \sigma\sqrt{2\pi} \quad (4)$$

therefore:

$$C_{\text{ppm}} = N_T A_H / (\sigma\sqrt{2\pi} N_0 \rho_H) \cdot 10^6 \quad (5)$$

Or solving for N_T will give the total number of alpha particles required to obtain a peak concentration of C_{ppm} :

$$N_T = C_{\text{ppm}} N_0 \rho_H \sigma \sqrt{2\pi} / A_H \cdot 10^{-6} \quad (6)$$

Since the alpha particle carries a charge of 3.2×10^{-19} coulombs, the total charge in coulombs delivered to the specimen per unit area is:

$$Q = 3.2 \times 10^{-25} C_{\text{ppm}} N_0 \rho_H \sigma \sqrt{2\pi} / A_H \quad (7)$$

5.1.5.2 A uniform helium depth profile can be approximated by injecting a sequence of helium layers whose mean range differs by the full-width-half-maximum of the range straggling distribution (FWHM = 2.35 σ). Under these conditions, the midpoint concentration will be equal to the peak concentration, whereas the summed peak concentration will be increased by 12 %. This increase is due to a 6 % contribution from the tail of each of the adjacent peaks.

5.1.6 Report—Information to be reported for helium implantation experiments should include the estimated helium concentration and its distribution in the material, the energy of the alpha particles employed, method for degrading the energy, beam current on the target, temperature rise, and total charge implanted.

5.2 Implantation of Helium Using α -Emitting Radioisotopes:

5.2.1 Summary of Method—The emission of α -particles during the radioactive decay of ^{238}Pu , ^{244}Cm , ^{208}Po , and ^{242}Cm can be used to implant helium concentrations of 10 to 100 appm in the surface layer of specimens in periods of one to two

months. The distribution of helium in the foil is controlled by the energy of the particle and the extent of shielding by the source material, and therefore is nonuniform. The source geometry is a thin sheet that conforms to the surface of the material to be implanted. The sources represent a potential health and contamination hazard, and therefore require handling in a glovebox facility with suitable shielding. The technique offers an inexpensive, simple method for implanting helium if surface implantation with a nonuniform profile is acceptable.⁴

5.2.2 Limitation—The major limitation of the technique is the depth to which helium can be implanted. The α -particles from usable sources have energies between 4 and 8 MeV and for a 6-MeV α -particle, the maximum penetration depth is about 30 μm in aluminum, about 12 μm in nickel, and about 20 μm in zirconium. The helium concentration profile will be nonuniform, varying from 0 helium just beyond the maximum range of the α -particles at normal incidence to some maximum value. Thickness of the source will affect the concentration profile if it is less than the self-absorption thickness.

5.2.3 Apparatus:

5.2.3.1 Source—Practical alpha sources are those unstable isotopes that decay and will give a target helium concentration of the order of 10 to 100 appm in a period of one to two months. Table 1 provides the recommended nuclear data for most of the practical sources that are recommended for use in this application (24).

5.2.3.2 Of these, ^{238}Pu represents the upper limit of half-life consistent with reasonable implantation time, and ^{242}Cm represents a lower limit of half-life below which consumption of the source may be undesirable. Some of these isotopes are also subject to spontaneous fission, creating neutrons and fission products, and some are sources of high [gamma] activity. All α sources are potential health hazards due to the toxic nature of ingested particles. Safety requirements dictate that these sources be handled in a glovebox, and some may require special licensing similar to that for handling of Pu. Metallic α sources are extremely reactive with oxygen and with most

⁴ A less flexible variant of this method is the examination of a microstructure in the helium "halos" generated around any naturally occurring boron-containing particles in metals (21). Boron has been deliberately introduced (22, 23), but this can introduce chemical alterations of the matrix or other alloy phases. These variants also entail studying the effects of lithium on microstructural development (22).

TABLE 1 Recommended Nuclear Data for Relevant Alpha Sources⁴

Source	Half-Life	Alpha (α) Emissions		Gamma (γ) Transitions		Spontaneous Fission Branching Ratio (%)
		Energy (MeV)	Probability (%)	Energy (MeV)	Probability (%)	
^{238}Pu	87.74 (3) a	5.49903 (20)	71.04 (6)	0.099852 (3)	0.00735 (8)	1.85 (5) E-7
		5.4563 (2)	28.85 (6)	0.043498 (1)	0.0397 (8)	
^{244}Cm	18.11 (3) a	5.80477 (5)	76.7 (4)	0.042824 (8)	0.0258 (7)	1.36 (1) E-4
		5.76265 (5)	23.3 (4)	0.098860 (13)	0.00136 (9)	
^{208}Po	2.898 (2) a	5.1149 (14)	99.9958 (4)	0.29181 (5)	0.00227	—
		4.220 (15)	0.00024 (7)	0.57013 (7)	0.00138 (17)	
^{242}Cm	162.86 (8) d	6.11272 (8)	74.06 (7)	0.04408 (3)	0.0330 (7)	6.36 (14) E-6
		6.06937 (9)	25.94 (7)	0.10192 (4)	0.00251 (14)	

⁴ Nuclear data from Ref (24), except ^{208}Po that is taken from Ref (25).

other elements, so their use in metallic form requires some form of protective atmosphere or a cladding envelope. The source strength is reduced if cladding is used to protect the surface. The reactivity of the metals used for sources also limits their use to temperatures below 500 °C. In the form of oxides they are more stable and can be used unshielded and at higher temperatures. However, it is recommended that even oxide sources should be clad or confined to minimize contamination of targets by spallation and to reduce health hazards.

5.2.4 Procedure—An example of the use of α sources for implantation is given in Ref (26). A source of $^{244}\text{CmO}_2 + ^{244}\text{Cm}_2\text{O}_3$ was evaporated on a 25.4-mm diameter titanium disk substrate to a thickness of 3 to 4 mg/cm². The target was placed in a recessed aluminum holder covered with a 5- μm thick aluminum cover foil to minimize contamination from the source. All operations were performed in a glovebox. A stainless steel spacer ring 25.4 mm in diameter and 1.5 mm thick was placed on top of the cover foil, and the source laid face down over the ring for the required implantation time. The ring holds the source away from the aluminum foil, preventing scratches and reaction products from damaging the source.

5.2.4.1 The use of a source whose thickness is less than the range of α -particles in source material makes possible a tailored profile in the target: a plateau preceding a linear decline. The depth of this plateau, at acceptable helium levels, is not likely to exceed half the maximum penetration depth. In the example cited in 5.2.4 (26), a zone 3.5 μm deep below the surface of a nickel target attained a uniform ~ 10 atomic ppm helium concentration after three days of exposure. Alternatively, two-sided implantation of specimen foils thinner than the maximum penetration depth can be used (27). The configuration selected for implantation should be consistent with the intended simulation (peaked distribution or uniform concentration).

5.2.5 Calculation and Interpretation of Results—The range of the α particles should be calculated from range tables using the procedures described in 5.1 for implantation using charged particle accelerators. Calculations of the rate of implantation of helium into a target and its final concentration must take into consideration the amount of α -emitter in the source, the age of the isotope, source thickness, contamination from other α -emitters, source density, and the range of α particles within the source. Some of these factors can be determined by chemical analyses, by precision weighing, and by radiation counting. It is recommended that the source be calibrated by implantation of a stack of 1 μm thick foils, analysis of the helium content of the individual foils, and then fitting the concentration profile to the calculated source characteristics.

5.2.6 Reporting of Results—Information to be reported should include the estimated helium concentration, α source characteristics such as isotope, activity, chemical species, physical dimensions, cladding, source calibration method, time of implantation, and the basic assumptions used to calculate the helium concentration.

5.3 Tritium Decay Charging:

5.3.1 Summary of Method—Helium is introduced into the metal specimen by diffusing tritium into the specimen, accumulating the desired concentration of helium from the radio-

active decay of tritium by the reaction $^3_1\text{T} \rightarrow ^3_2\text{He} + ^0_{-1}\beta$ (half-life of 12.34 years), and then heating the specimen in vacuum to remove the remaining tritium. The method offers the advantage of introducing helium into bulk specimens and into specimens with unusual contours.

5.3.2 Limitations—The distribution of helium in a specimen may be influenced by segregation or trapping of the tritium at internal sinks or by the formation of tritides. The use of this technique must be accompanied by characterization of the sample to ensure that a homogeneous distribution of helium has been achieved. An inherent characteristic of the technique for simulating the effects of transmutation-produced helium in neutron-irradiated specimens is the absence of radiation damage. The mobility of helium may change under irradiation because of changes in the diffusion mechanism when a steady-state concentration of interstitials and vacancies is present in the material during irradiation. The ratio of helium to dpa also may influence swelling and mechanical properties. The tritium decay method will not duplicate these effects and therefore should not be used in circumstances requiring both helium and displacement damage. It might, however, be considered an advantage in separating the effects due to helium from those of the associated displacement damage. Tritium is a radiological safety hazard, and suitable facilities for handling tritium must be available.

5.3.3 Apparatus—Depending on the method applied, the tritium charging system must be capable of evacuation to at least 10^{-3} Pa and capable of containing tritium at overpressures of a few tens of Pa. Elevated temperature capability to at least 500 °C is required for the charging system and higher if outgassing is done in the same system. If the radioactive decay stage is done at elevated temperatures, a temperature controller with a stability of ± 5 °C for periods of a month also will be required. Provision for measuring the tritium pressure over the specimens with sufficient accuracy to determine changes in pressure during the charging stage is required. Outgassing of the specimens following the decay period is required and may be done in either the charging system or another system with high-vacuum and high-temperature capabilities.

5.3.4 Procedure—Several procedures have been used to introduce helium into specimens by tritium decay; three will be mentioned here. The methods typically involve charging the specimens with tritium at elevated temperature and a final outgassing step, but differ in details such as the level of tritium overpressure and whether the tritium decay step is carried out at elevated temperature under a tritium pressure or whether it is done at room temperature with no tritium overpressure. Similar levels of helium content can be obtained with each method and in the absence of any obvious factor that would indicate a preference for one technique over the other, any of the methods may be acceptable for tritium (helium) charging.

5.3.4.1 Method A (28)—The first step in the process involves diffusion of tritium into specimen. The specimen is placed in a glass vacuum system that is subsequently evacuated to less than 10^{-3} Pa and is then pressurized with tritium to a pressure of 1.5 to 2.0 kPa by heating a uranium tritide bed. The section of the system containing the specimen is heated to 475 °C. The tritium pressure change in the system is monitored

to determine when tritium absorption in the specimen is essentially complete. This step usually takes from 2 to 3 h and the furnace is then cooled to room temperature. The pressure of the remaining tritium is measured at room temperature and compared with the original pressure to determine the amount of tritium absorbed by the specimen. This room temperature pressure is essentially the same as the final high-temperature pressure. Therefore, it is possible to charge a specific tritium concentration into a given sample by monitoring the pressure during absorption. The excess tritium remaining in the glass system is reabsorbed and stored on the uranium tritide bed. The second step involves aging of the specimen to allow time for transmutation of the tritium to helium. In Method A, the aging step is carried out at room temperature. The tritium decay time is determined from the final helium concentration desired in a given specimen, the tritium concentration charged into the specimen, and the tritium half-life (12.34 years). A typical initial tritium content of 95 000 appm yields a charging rate of 75-appm helium per month. The final step is removal of the tritium from the specimen. The specimen is placed in the original glass vacuum system, which again is evacuated to less than 10^{-3} Pa and heated to tritium outgassing temperatures of 875 to 925 °C. The evolved tritium is pumped into a calibrated volume chamber and pressure measurements are taken to determine the amount of tritium recovered. Typical pressure-volume measurements show recovery of 96 to 99+ % of the tritium calculated to be in the samples at the end of the aging period. The specimen is cooled to room temperature and the outgassed tritium is reabsorbed on the uranium tritide bed.

5.3.4.2 Method B (29)—The first step in the process again involves diffusion of tritium into the specimen. The specimen is weighed and placed in the charging vessel, the system is evacuated to 4 Pa, and heated to 400 °C. A known volume of tritium is metered into the charging vessel sufficient for that to be absorbed in the specimen and an equilibrium pressure of 1.33 kPa in the chamber. The charging vessel is valved off, and the temperature is maintained at 400 °C. The aging step in Method B is carried out at temperature and under the pressure of 1.33 kPa. The time at temperature is determined by the final helium concentration desired in the specimens. The tritium is removed from the specimen by evacuating the system for one week at 4 Pa. The temperature is held at 400 °C. The charging vessel is cooled and the specimens are placed in a high-vacuum system. The specimens are heated to 550 °C in a vacuum of about 1.33×10^{-4} Pa and outgassed for another week. The charging vessel is cooled and a small sample (about 0.05 g) is removed from the specimen. The sample is dissolved in acid, and an analysis for tritium is made. If the tritium level is above 0.3 to 1.0 C_i/g, the outgassing is repeated until these levels are achieved.

5.3.4.3 Method C (30, 31)—This method has been employed at the Savannah River National Laboratory for two kinds of studies on stainless steels and other alloys. The first kind of study involves measuring the effect of tritium and its decay product, helium, on the mechanical and fracture toughness properties of the alloy, while the second is for measuring the effects of only the helium decay product on the cracking properties of the steel at elevated temperature or during

welding. Both studies require samples that have been exposed to tritium gas at high pressures, up to 34 MPa, and temperatures up to 350 °C for two to three weeks. The temperature of 350 °C is high enough for tritium to diffuse into ~6-mm thick sections and obtain a uniform concentration but low enough to prevent significant changes to the preexisting microstructure. Tritium diffusion calculations (30) are used to estimate the amount of dissolved tritium. Helium concentrations in the range of ~1 to 20 appm are used for studies of helium effects on welding. The tritium gas pressure is chosen based on the amount of dissolved tritium and decay helium that is required. For most weld studies, the tritium is off gassed at 350 °C after the desired amount of helium has obtained from tritium decay. Following tritium exposure, samples are cooled and may be stored in air for long periods of time (years) at -50 °C. This temperature is low enough prevent tritium diffusion while the helium decay product can accumulate in the microstructure. Samples can be dissolved in an acid and tritium content measured, and the helium content is typically measured by vacuum extraction measurements such as those described in 6.1.

5.3.5 Calculations or Interpretation of Results:

5.3.5.1 Computation of Helium Content—The helium content of a tritium charged specimen is estimated from the tritium half-life using the radioactive decay equation $-dn/dt = \lambda n$ in the following form:

$$[\text{He}_{\text{appm}}]_t = [T_{\text{appm}}]_i (1 - \exp(-\lambda t)) \quad (8)$$

where:

t = decay time,
 $[\text{He}_{\text{appm}}]_t$ = He content at decay time t in atomic parts per million, appm,
 $[T_{\text{appm}}]_i$ = initial T concentration, appm, and
 λ = decay rate constant = $0.693/t_{1/2}$,

where:

$t_{1/2}$ = half-life.

For tritium, $t_{1/2} = 12.34$ years. The initial tritium content is either calculated from the experimentally determined tritium uptake during the tritium charging cycle (Method A), or it is assumed to be the equilibrium concentration determined from the metal-hydrogen phase diagram at the given tritium charging temperature and pressure (Method B). Calculation of the helium concentration in a specimen assumes a constant volume tritium charging apparatus and a single, initial tritium gas charge. The calculation for determining the helium content of a specimen after a given number of charging days is given as follows:

$$[\text{He}_{\text{appm}}]_t = [T_{\text{appm}}]_i (1 - \exp(-1.547 \times 10^{-4} t)) \quad (9)$$

for decay time t measured in days where the moles of tritium (as T_2) absorbed into the metal specimen are equal to twice the moles of tritium gas (as T_2) absorbed by the specimen, determined experimentally by the pressure drop in the constant volume charging system. The equations used to calculate the amount of tritium absorbed in atom parts per million are given as follows:

$$[T_{\text{appm}}]_i = (n_T/W_s/M_m) \times 10^6 \text{ ppm} \quad (10)$$

$$n_T = n_{T_2} (M_{T_2}/M_T) \quad (11)$$

$$n_{T_2} = ((\Delta P)V/RT)_{V,T} \quad (12)$$

therefore:

$$[T_{\text{appm}}]_i = \left[\frac{(\Delta P)V}{RT} \right] \left\{ \frac{M_{T_2}/M_T}{W_s/M_m} \right\} \times 10^6 \text{ ppm} \quad (13)$$

where:

n_{T_2} = number of moles tritium gas absorbed by the specimen,

n_T = number of moles T absorbed by the specimen,

ΔP = experimentally observed pressure drop during tritium charging,

V = charging system volume,

T = temperature of P measurement,

R = gas law constant,

M_{T_2} = molecular weight of tritium gas,

M_T = molecular weight of tritium,

M_m = molecular weight of specimen matrix, and

W_s = weight of specimen.

5.3.5.2 The Method B technique for charging a specimen with helium using the tritium decay method is based on a loading rate of 50 appm per week. The tritium required in the charging process is calculated below for an example using niobium. The moles ^3He required per gram of niobium are:

$$\frac{50 \times 10^{-6}}{\text{wk}} \times \frac{1}{92.9 \text{ g(Nb)/mol}} = 5.382 \times 10^{-7} \frac{\text{mols}(^3\text{He})}{\text{g(Nb) wk}} \quad (14)$$

The helium generation rate based on a half-life of 12.34 years is:

$$1.097 \times 10^{-3} \text{ atoms } (^3\text{He})/\text{atom}(T)/\text{wk}$$

The number of cm^3 of tritium at STP required per gram of niobium are:

$$\begin{aligned} & \left[\frac{(5.382 \times 10^{-7} \text{ mols}(^3\text{He})/\text{g(Nb)/wk}) \times (22428 \text{ cm}^3(T_2)/\text{mol}(T))}{[(1.097 \times 10^{-3} \text{ atoms}(^3\text{He})/\text{atom}(T)/\text{wk})} \right] \\ & \times (2N_A \text{ atoms}(T)/\text{mol}(T_2)/N_A \text{ atoms}(^3\text{He})/\text{mol}(^3\text{He})) \\ & = \frac{5.50 \text{ cm}^3 T_2}{\text{g(Nb)}} \end{aligned} \quad (15)$$

Parameters of the charging system are:

system volume = 128 cm^3 ,

charging vessel volume = 155 cm^3 ,

gas fill = 94 % T_2 ,

charging temperature = 673 °K, and

equilibrium gas pressure = 10 mm.

The total cm^3 of gas required for specimens weighing a total of 40 g are:

$$\left[\left(\frac{40 \text{ g(Nb)} \cdot 5.5 \text{ cm}^3(T_2)}{\text{g(Nb)}} \right) + \left(\frac{10 \text{ mm} \times 273 \text{ K} \times 155 \text{ cm}^3}{760 \text{ mm} \times 673 \text{ K}} \right) \right] \frac{1}{0.94} = 235 \text{ cm}^3(T_2) \text{ STP} \quad (16)$$

5.3.6 *Report*—Information to be reported should include the estimated helium concentration, residual tritium concentration, and pertinent details of the charging sequence.

5.4 *Introduction of Helium by Ion Implantation and Hot Isostatic Pressing of Metal Powders:*

5.4.1 *Summary of Method*—The specimen size limitations inherent in the alpha particle implantation methods described in 5.1 and 5.2 can be bypassed by implanting metal powders with a low energy alpha beam and then fabricating specimens from the powder using powder metallurgy techniques. The method falls conceptually into three steps: (1) ion implantation, (2) consolidation, and (3) thermomechanical processing. In the first step, helium is implanted in the individual particles of metal powder by ion bombardment. The second step involves fabricating a bulk solid from the helium-containing powder. The third step is intended principally to control the microstructure of the product and the distribution of helium within it.

5.4.2 *Limitations*—The technique is limited by the availability of powders in fine sizes and the degree to which the properties of the powder metallurgy product represent those fabricated by conventional techniques.

5.4.2.1 The displacement damage produced by the low energy implant may not be representative of damage produced by neutrons and, as with other alpha implant techniques, caution should be exercised in interpreting or extrapolating results where both helium content and displacement damage influence the effects to be simulated. Consolidation treatments require high pressures and temperatures near 0.5 T_m , which may result in formation of small helium bubbles.

5.4.3 *Apparatus*—The apparatus required for this technique includes a linear accelerator capable of accelerating alpha particles to energies of 150 keV, electrostatic deflection plates, a target chamber, and sample cup capable of rotation to mix the powders. Final consolidation requires a hot isostatic press.

5.4.4 *Procedure*—The procedure involves ion implantation of the powder, consolidation, and thermomechanical processing. Examples of the utilization of this technique for implanting AISI Type 316 stainless steel and molybdenum are provided in Refs (32) and (33). The powder particles used for the implant should have diameters approximately two times the range of the available helium ions. The particle size distribution should be determined by X-ray sedimentation analysis using a dilute water suspension of the powders. The suspensions should be ultrasonically dispersed for 30 min prior to analysis. Separated fines with a mean particle diameter twice the ion range and with 90 % of the particles having diameters less than four times the ion range can be obtained by this method. This provides a reasonably uniform distribution of implanted helium atoms because most of the volume of a spherical particle lies close to its surface.

5.4.4.1 Fine particle size powders are characterized by high chemical activity and tend to absorb relatively large amounts of oxygen when exposed to air. If not removed, this oxide “skin” forms an oxide grain boundary phase when the powder is pressed. To reduce the oxygen level, the powder is heat treated for 8 h at temperatures high enough to react with the oxide under slowly flowing dry hydrogen (dew point -60 °C). The effluent gas should be monitored for moisture content until the moisture level has dropped to the initial level of the source gas.

5.4.4.2 After the hydrogen treatment, the powder in the closed reaction vessel is transferred to an inert gas glovebox without exposure to air. The powder is then loaded into the