Designation: E942 – 16

Standard Guide for
Investigating the Effects of Helium in Irradiated Metals[1]

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

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
E706 Master Matrix for Light-Water Reactor Pressure Vessel Surveillance Standards, E 706(0) (Withdrawn 2011)[4]
E910 Test Method for Application and Analysis of Helium Accumulation Fluence Monitors for Reactor Vessel Surveillance, E706 (IIIC)

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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[1] This guide is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.08 on Procedures for Neutron Radiation Damage Simulation.

Current edition approved Dec. 1, 2016. Published January 2017. Originally approved in 1983. Last previous edition approved in 2011 as E942 – 96 (2011). DOI: 10.1520/E0942-16.

[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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

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 µA 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 µA. 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 Degrader*—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. 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 ±5 % can be achieved by superposition of the depth profiles produced by 25-µm increments in the thickness of aluminum beam degrader foils. Uniformity of ±10 % is recommended for all material experiments. 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-cm^2 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 flux 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 flux on the axis to the average flux 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 10^6:

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

$$M_{\mathrm{H}} = N_0 \rho_{\mathrm{H}}/A_{\mathrm{H}} \tag{2}$$

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 flux since the total area under a normal distribution curve is equal to σ2π. If N_T is the total number of particles incident on the surface per unit area (fluence) then:

$$M_{\mathrm{He}} = \frac{N_T}{\sigma\sqrt{2\pi}} \exp - \frac{\left(R - \bar{R}\right)^{-2}}{2\sigma^{2}} \tag{3}$$

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

$$M_{\mathrm{He}} = N_T / \sigma\sqrt{2\pi} \tag{4}$$

therefore:

$$C_{\mathrm{ppm}} = N_T A_{\mathrm{H}} / \left(\sigma\sqrt{2\pi}\, N_0 \rho_{\mathrm{H}}\right) \cdot 10^{6} \tag{5}$$

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

$$N_{\mathrm{T}} = C_{\mathrm{ppm}} N_0 \rho_{\mathrm{H}} \sigma \sqrt{2\pi} / A_{\mathrm{H}} \cdot 10^{-6} \tag{6}$$

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:

$$Q = 3.2 \times 10^{-25}\, C_{\mathrm{ppm}} N_0 \rho_{\mathrm{H}} \sigma \sqrt{2\pi} / A_{\mathrm{H}} \tag{7}$$

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.[5]

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. The following list covers the most practical sources that are recommended for use in this application (**24**):

Source	Half-Life	αEnergy, MeV	γ Radiation, MeV	Spontaneous Fission Half-Life Yr
$_{94}{}^{238}$Pu	87.7 year	5.50 (72 %) 5.46 (28 %)	0.099 (0.008 %) others	Yes, 4.77 × 10^{10}
$_{96}{}^{244}$Cm	18.1 year	5.80 (77 %) 5.76 (23 %)	0.043 (0.02 %) others	Yes, 1.35 × 10^{7}
$_{84}{}^{208}$Po	2.90 year	5.1	0.285 (0.003 %) 0.060 (0.006 %) others	No
$_{96}{}^{242}$Cm	163 days	6.11 (74 %) 6.07 (26 %)	0.044 (0.04 %)	Yes, 6.09 × 10^{6}

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 other elements, so that 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 (**25**). A source of $^{244}\mathrm{CmO_2} + {}^{244}\mathrm{Cm_2O_3}$ was evaporated on a 25.4-mm diameter titanium disk substrate to a thickness of 3 to 4 mg/cm^2. 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

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[5] 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**).

the example cited in 5.2.4 (**25**) 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 (**26**). 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 radioactive decay of tritium by the reaction $^{3}_{1}T \rightarrow {}^{3}_{2}He + {}_{1}\beta\left(\text{half} - \text{life of } 12.34 \text{ years}\right)$, 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* (**27**)—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* (**28**)—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* (**29**, **30**)—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 (**29**) 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 at 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:

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

where:

t = decay time,
$[\mathrm{He}_{\mathrm{appm}}]_t$ = He content at decay time t in atomic parts per million, appm,
$[T_{\mathrm{appm}}]_i$ = 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:

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

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:

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

$$n_T = n_{T_2} \left(M_{T_2} / M_T\right) \tag{11}$$

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

therefore:

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

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 ^{3}He required per gram of niobium are:

$$\frac{50\times10^{-6}}{\text{wk}}\times\frac{1}{92.9\ \text{g(Nb)/mol}}=5.382\times10^{-7}\ \frac{\text{mols}(\,^3\text{He})}{\text{g(Nb) wk}}\qquad(14)$$

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

$$1.097\times10^{-3}\ \text{atoms}\ (^3\text{He})/\text{atom}(T)/\text{wk}$$

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

$$\frac{[(5.382\times10^{-7}\ \text{mols}(\,^3\text{He})/\text{g(Nb)/wk})\times(22428\ \text{cm}^3(T_2)/\text{mol}(T))]}{[(1.097\times10^{-3}\ \text{atoms}(\,^3\text{He})/\text{atom}(T)/\text{wk})\times(2\text{N}_\text{A}\ \text{atoms}(T)/\text{mol}(T_2)/\text{N}_\text{A}\ \text{atoms}(\,^3\text{He})/\text{mol}(\,^3\text{He}))]}=\frac{5.50\ \text{cm}^3T_2}{\text{g(Nb)}}\qquad(15)$$

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:

$$\left[\left(\frac{40\ g(Nb)\ 5.5\ cm^3(T_2)}{g(Nb)}\right)+\left(\frac{10\ mm\times273\ K\times155\ cm^3}{760\ mm\times673\ K}\right)\right]\frac{1}{0.94}=235\ cm^3(T_2)STP\qquad(16)$$

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 (**31**) and (**32**). 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 implantation cup and transferred to the accelerator, again without exposure to air.

5.4.4.3 Helium ions are accelerated to 150 keV with a linear accelerator. The desired high-energy species are selected with a magnetic mass analyzer. The resulting ion beam is electrostatically steered in the vertical and horizontal planes to pass through an aperture and electrostatically steered to impinge on the target.

5.4.4.4 The powder, lying in the corner of the inclined cup, tumbles and mixes as the cup rotates, thereby exposing all powder particles to the ion beam. Scattered ions and ions passing through the outer layers of particles provide lower energy helium to distribute throughout the powder particle volume.

5.4.4.5 After helium implantation, the powder is sieved (down to −400 mesh) to break up aggregates. Analysis for residual and added gases is done by vacuum fusion extraction of the gases and mass spectrographic analysis. Duplicate powder samples are wrapped in platinum (which acts as a fluxing agent) and heated by induction in graphite crucibles. Blanks are run to account for the outgassing of the graphite, platinum, and apparatus.

5.4.4.6 Stainless steel tubes containing the helium implanted powder are evacuated to a pressure of about 10^{-2} Pa to remove all accessible absorbed or trapped gases. The evacuated stainless steel tubes are closed by crimping and the crimped sections are heated and hammered to bond opposing surfaces. These seals are reinforced with a weld bead. The sealed tubes are loaded in a hot isostatic press (HIP) which is first pressurized to 6.9 to 7.6 MPa (1000 to 1100 psi), and heated to about 500 °C, held for 2 h and cooled and depressurized.

5.4.4.7 The compacted powder can be worked to prepare microstructures for controlled precipitation of helium bubbles. Thermomechanical treatments can be designed that produce: (*a*) a reasonably homogeneous helium bubble distribution; (*b*) dislocation networks and precipitates which act as nucleation sites for gas precipitation; and (*c*) recrystallized microstructures with helium bubbles predominantly on the grain boundaries.

5.4.5 *Report*—The analyzed helium content and the processing history and heat treatments should be reported.

6. Techniques for the Analysis of Helium Content and Distribution

6.1 *Mass Spectrometric Analysis of Helium Content:*

6.1.1 *Summary of Method*—This section describes a recommended method for determination of the absolute helium concentration in any solid material using mass spectrometric analysis. The method utilizes milligram size samples to measure absolute amounts of either ^{3}He or ^{4}He, in concentrations ranging from percent levels down to 1 appt (atomic parts per trillion, 10^{-12} atomic fraction) with a total uncertainty (random or systematic, or both) of 1 to 2 % or better. The ^{4}He (or ^{3}He) content of the specimen is determined by vaporizing a small sample of the material in a furnace under vacuum, adding a precisely known amount of ^{3}He (or ^{4}He), and measuring the ^{4}He/^{3}He isotopic ratio.

6.1.2 *Limitations*—The main factors that determine the detection limits of the method are the background helium level from desorption of helium from the mass spectrometer system walls when the crucibles are heated, and permeation of helium through the walls, joints, and valves of the system. Helium contents of ~1 to 10×10^8 atoms of ^{4}He have been measured, which translates to a detection limit of from ~1 to 10 ppt (parts per trillion, ~1 to 10×10^{-12} atom fraction), depending on the sample mass.

6.1.3 *Apparatus*—The procedures described herein were performed on a custom-built apparatus (**33**) consisting of a gas mass spectrometer, high-vacuum system, high-temperature furnace, and calibrated volume spike system. The mass spectrometer has an all-metal tube with interior volume of approximately 1 L, an electron bombardment ion source, a permanent magnet, and an electron multiplier. The application of the system is discussed in Test Method E910.

6.1.3.1 A vacuum system capable of maintaining the mass spectrometer pressure as low as 10^{-7} Pa between analyses is required. The mass spectrometer must be capable of being operated in the "static" mode (**33**).

6.1.3.2 The helium in the sample to be measured is released by vaporization in resistance-heated tungsten-wire or graphite crucibles. Tungsten-wire crucibles are used for materials with melting points less than about 1800 °C. Graphite is used for higher melting point materials, up to and including carbon itself.

6.1.3.3 A system of getters is used to purify the helium gas sample before it is put into the mass spectrometer, and to maintain a high vacuum in the mass spectrometer while it is being operated in the static mode. The getters consist of liquid-nitrogen-cooled charcoal traps, followed by non-evaporable Zr-Al alloy getters operated at ambient temperature.

6.1.3.4 The "spike" system consists of a network of calibrated volumes that dispenses known quantities of ^{3}He and ^{4}He for calibration and for isotope dilution purposes. Separate systems provide exactly known spikes of ^{3}He, ^{4}He, and ^{3}He + ^{4}He in amounts ranging from ~1×10^{13} to ~3×10^{16}atoms. The ^{3}He + ^{4}He mixture is used as a "standard" with which to calibrate the relative sensitivity of the mass spectrometer for masses 3 and 4. Other "synthetic" combinations of ^{3}He and ^{4}He can be mixed to verify the relative sensitivity, and to cross-check the calibration and linearity of the mass spectrometer system. The volumes of the various sections of the spiking system were measured with an uncertainty of less than 0.02 % before final assembly of the system by filling the space between the stopcocks with mercury or water and by weighing the contained liquid.

6.1.4 *Analysis Procedure*—The first step in the procedure for analysis of a metallic specimen containing helium in the ppm range is to obtain a sample weighing ~1 to 5 mg by milling, cutting, drilling, etching, or otherwise sectioning the sample. Such samples contain much more helium than is necessary for the determination, but handling smaller specimens is difficult, and weighing them to better than 1 % accuracy becomes increasingly time consuming. Materials with melting points less than 1800 °C are cut into ~1-mg pieces and placed in tungsten crucibles. Materials with melting points above 1800 °C are cut into 1 to 5-mg pieces and placed in graphite crucibles. Samples whose helium concentrations are 0.1 ppb or lower are cut into pieces weighing ~20 to 100 mg and are loaded into graphite crucibles. The system is then evacuated by turbomolecular and ion pumping systems. A spike is added to the oven chamber just before vaporizing the sample. A current is passed through the tungsten or graphite crucible until the sample is observed to melt and vaporize. Complete mixing of the isotopes occurs in a few seconds.

6.1.4.1 Unwanted gases released during the vaporization of the sample are removed by passing the helium over the getters. It is important to perform the purification quickly so as to minimize contamination of the sample by background helium that could change the ^{4}He/^{3}He ratio before it is measured. The usual procedure is to allow the gas (or fraction of gas, for larger samples) to first expand into the liquid-nitrogen-cooled charcoal getter volume. After 20 s the getter volume is isolated for an additional 20-s period, following which the trapped gas is permitted to further expand into the alloy getter volume. Again, following a 20-s expansion time and a 20-s isolation time, the final trapped gas fraction is expanded into the mass spectrometer volume for isotopic analysis. The small amount of helium eventually admitted, about 3×10^{-14} m^3 STP, does not deleteriously affect the mass spectrometer vacuum.

6.1.5 *Calculation or Interpretation of Results*—Determination of the ratio of ^{4}He/^{3}He is accomplished by repeatedly changing the accelerating voltage so that the ^{4}He and ^{3}He ion beams are sequentially measured using a high-precision voltmeter and the ratio of amplitudes of the ^{4}He and ^{3}He peaks is determined. Multiple measurements of isotopic ratios are found to have a standard deviation of ≃0.5 %.

6.1.5.1 Gas samples from milligram size specimens whose helium concentrations are above 0.1 appm are sufficiently large that the very small permeation or desorption of ^{4}He into the mass spectrometer can be ignored. For smaller samples, this constant leak becomes perceptible and is carefully measured so that corrections to account for it can be made. Thus, in all analyses, the ^{4}He/^{3}He ratio is carefully examined for systematic increase; and if such an increase is found, the ratio is measured against time and extrapolated to the time the sample was admitted to the mass spectrometer volume. This gives the helium isotopic ratio at the time the sample was introduced, but does not account for the ^{4}He leakage into the sample line or crucible vacuum enclosure. By taking a second and third aliquot of gas from this enclosure, and analyzing them as described in 6.1.4, results can be extrapolated to the time the sample was vaporized to give the true amount of ^{4}He that was released from the sample. This time-correction procedure is used for samples with helium concentrations below ~0.1 appm (10^{-7} atom fraction).

6.1.6 *Precision and Accuracy*—The absolute accuracy of the helium measurements depends principally on the uncertainties of the sample mass and of the measured ratio of ^{4}He and ^{3}He. Both these uncertainties are less than ~0.5 % for samples weighing more than 0.3 mg and containing more than ~10 ppb He. Considering these and other possible systematic errors, the absolute (1 σ) standard deviation of an analysis is estimated to be 1 %. Duplicate analyses of the same specimen have confirmed that a reproducibility of about 0.5 % is regularly obtained. It is the usual practice to analyze duplicate specimens of each sample, not only to provide a measure of the reproducibility, but also to give bounds on the homogeneity of helium within the sample.

6.1.7 *Report*—Information to be reported from each specimen analysis should include the sample mass, number of helium atoms released, helium concentration, and uncertainty limits.

6.2 *Distribution of Helium Within a Specimen Using Alpha-Alpha Elastic Scattering:*

6.2.1 *Summary of Method*—The distribution of helium through the thickness of a foil specimen can be determined from the elastic scattering of an energetic alpha particle beam by helium atoms residing in the specimen (**34-36**). This technique, first used by B. L. Cohen, et al. (**37**) for measuring the depth profile of hydrogen in metal foils, consists of bombarding the foil containing helium with energetic alpha particles and detecting 45° elastic scattering events. These elastic scattering events are singled out from the multitude of other reaction products by coincidence detection of the scattered alpha particle and the recoiling helium nucleus in a pair of detectors placed at angles of ±45° to the beam direction. The total energy lost by this pair of scattered alpha particles is proportional to the depth of the scattering site. A frequency distribution of such elastic scattering events versus energy loss is simply related to the depth profile of helium concentration in the foil.

6.2.2 *Limitations*—The technique is limited by the thickness of material that can be penetrated by an energetic alpha particle beam and a practical time on cyclotron usage, a limit that corresponds to concentrations of about 1-ppm He.

6.2.3 *Apparatus:*

6.2.3.1 *Accelerator*—An accelerator, such as a cyclotron or Tandem Van de Graaff, capable of accelerating alpha particles to energies in the range from 15 to 60 MeV is required. Higher energies permit thicker samples to be profiled, but depth resolution improves for lower energies. Profiling is normally performed at one fixed energy. For the highest sensitivity, a beam energy should be selected where the elastic scattering cross-section is highest and remains nearly constant as the beam loses energy in penetrating the specimen (**38**).

6.2.3.2 *Beam Transport Equipment*—Beam handling equipment such as vacuum drift tube, defining slits, quadrupole magnets, and steering magnets are required to prepare and transport a well-defined beam to the target. Beam energy, direction, and divergence should be known within close limits. Beam intensities on the target to be profiled are of the order of 100 nA or less.

6.2.3.3 *Scattering or Target Chamber*—A suitable scattering chamber with three or four degrees of motion and that can be evacuated is required. The ability to change the elevation and angle of the target is useful for alignment and permits multiple target ladders. While one detector arm may be fixed at 45° with respect to the beam direction, it is desirable to be able to move the defining detector in order to check alignment.

6.2.3.4 *Collimators,* thick enough to stop the incident beam, are placed ahead of the target to define (and restrict) the area on the target that is struck by the incident beam (and therefore the area profiled). Collimators are placed in front of the detectors to ensure uniform detector efficiency and to define the angular acceptance.

6.2.3.5 *Beam Measuring Equipment*—A Faraday cup to collect the transmitted (unscattered) beam should be placed behind the foil to be profiled. Collected charge is read by a current integrator capable of reading nA currents.

6.2.3.6 *Detectors*—Two solid-state detectors, such as surface barrier detectors of 1000 to 2000-μm thickness, are required to measure the energies of the scattered alpha particles. It is useful for calibration purposes if the detectors are thick enough to stop the incident beam for the energy at which profiling is performed.

6.2.4 *Test Specimens*—The thickness of foils that can be profiled by alpha-alpha scattering is limited by the penetration depth of the incident beam and outscattering due to multiple scattering. Best results are obtained on thicknesses of 25 μm or less for A1, and 12 μm or less for stainless steel at 32.6 MeV.

6.2.5 *Procedure*—Foils to be profiled are mounted on a target ladder and centered on the center of target rotation. This ladder is then mounted in the scattering chamber so it can be raised and lowered remotely, as well as rotated. Defining apertures are placed ahead of the target to restrict the beam to a small but finite area. Detectors with their respective collimators are mounted at 45° with respect to the beam with the angle of one detector capable of being changed remotely.

6.2.5.1 The beam is carefully focused on the target position and aligned with an aperture of fluorescent screen in the target position (larger in size than the spot to be profiled) so that the beam passes through the aperture and is collected in the Faraday cup. The alignment can be checked easily by inserting a target with a helium concentration into position and observing the number of alpha-alpha elastic scattered events per µC as a function of the movable detector angle while the other detector remains fixed at 45°.

6.2.5.2 Energy of the incident beam is taken from the current in the analyzing magnet. Alpha particles scattered from a gold target should be used to get an energy calibration of the gain of the electronics and data accumulation systems. The thickness of the foils can be accurately measured by observing the energy loss of lower energy alpha particles. A pulser peak is adjusted in amplitude to appear in the spectra above the scattered alphas to measure the live time of the ADCs.

6.2.5.3 Data are then accumulated for alpha-alpha scattering events in the sample to be profiled until sufficient data are available to establish a 99 % confidence limit for the summed energy coincident spectra. The energy spectra for the two detectors are summed in the computer of the data accumulation system. This sum may be displayed on an oscilloscope for monitoring purposes, and the data may be stored on magnetic tape for later analysis.

6.2.6 *Calculation or Interpretation of Results:*

6.2.6.1 *Transformation of Energy Spectra to Depth Profile*—The loss of elastically scattered alpha particles by a subsequent scattering can be compensated for by the following exponential attenuation of radiation as a function of path length. S(N) is the summed energy coincidence distribution and C(N) is the corrected distribution:

$$\mathrm{C}(\mathrm{N}) = S(\mathrm{N})\ \mathrm{e}^{\lambda}\ (N_B - N) \tag{17}$$

where:

N_B = the channel number corresponding to the back or exit surface of the target foil, and

λ = an attenuation or scattering coefficient.

N_B can be measured directly by a transmission energy loss measurement in the beam, but the difficulty in reducing the beam current to ultra low levels so that the detector is not damaged makes this procedure difficult. An independent method of measuring the foil thickness must therefore be used. The energy loss in target, $\Delta E(E_o, \mathrm{THK})$ can then be calculated and

$$N_B = N_o - K_{EN}\,\Delta E(E_o, \mathrm{THK}) \tag{18}$$

where:

N_o = the channel number corresponding to the bombarding energy and the constant, and

K_{EN} = the energy-to-channel-number conversion factor.

The constant is determined from two alpha-alpha measurements on opposite faces of a helium implanted foil such that the helium layer is not on the centerline of the foil. Transformation of these two summed coincidence energy distributions must yield isomorphous distributions. This procedure for determining λ requires two measurements and is very time consuming, especially for low level concentrations. However, the constant λ can be determined by an auxiliary measurement using a high-level, known-concentration, surface-implanted foil placed ahead of and in contact with the front surfaces of the unknown specimen.

6.2.6.2 An alternate technique that is also recommended in deducing actual helium concentration profiles from summed energy coincidence distributions is the use of More's alpha-alpha diagnostic code (**39**). After ascertaining the channel numbers corresponding to scattering events from front and rear surfaces of the specimen, the maximum signal strengths (counts) and signal widths (channels at FWHM) of identical low energy helium markers are measured on front and back surfaces of reference foils with thicknesses comparable to that of the specimen.

6.2.6.3 The summed energy alpha-alpha coincidence spectrum is then analyzed as a series of overlapping markers (Gaussian signals whose amplitude and width are linear functions of depth [channel number]). For a real space helium distribution given by *N(y)*, the actual number of counts at any given depth, *X*, is then given by:

$$C(X) = \int_{\text{Front Surface}}^{\text{Back Surface}} A(y) e^{(-\ (x\ -\ y)2\ /\ 2\ [\beta\ (y)2])} n(y) dy \tag{19}$$

where: *A(y)* and β(*y*) are the functions describing the variation in signal strength (amplitude) and width (resolution) with position in the foil. In applying this technique it has been found easiest to simply integrate "guesses" on the helium distribution function *n(y)* and compare the calculated alpha-alpha coincidence spectrum and moments of the spectrum with the experimental measurements.

6.2.6.4 The conversion of the channel number to a depth coordinate requires that one first determine the channel number corresponding to the back (N_B) or exit surface as indicated above. Then the channel number corresponding to the front (N_F) or entrance surface is calculated from the total energy loss by a front surface scattering. This calculation also requires an accurate measurement of foil thickness.

6.2.6.5 *Calculation of Absolute Helium Concentration*—It has been shown that the depth distribution of helium in a foil can be measured using the technique of alpha-alpha elastic scattering. Since the alpha-alpha elastic scattering cross section is known (**40**), it is possible to convert the ordinate axis of the depth distribution to a helium concentration in parts per million.

6.2.6.6 The yield at 45° from an alpha-alpha elastic scattering in counts/channel is given as follows:

$$Y = C_{\mathrm{ppm}} \times 10^{-6} \frac{QL}{qA_H} \rho_{\ H} W\, d\Omega \frac{d\sigma}{de} \Big|_{\mathrm{LAB}} (E_o\ ,\quad \theta_{\mathrm{LAB}}\ =\ 45\ °) \tag{20}$$

where:

C_{ppm} = the helium concentration in parts per million of host material of atomic mass A_H and density ρ_H,

L = Avagadro's number,
Q = total charge in coulombs of alpha particles passing through the target,
q = 3.20×10^{-19} = the charge in coulombs carried by an alpha particle,
W = the width in centimetres corresponding to one channel in the alpha-alpha depth distribution,
$d\Omega$ = the laboratory solid angle of the defining detector in steradians, and

$d\sigma/de_{LAB}$ (E_o, $\theta_{LAB} = 45°$) = the laboratory alpha-alpha 45° elastic scattering cross section at an incident alpha energy of E_o.

6.2.6.7 Calculation of count yield from a helium containing foil allows one to estimate the time necessary to measure a helium concentration to given accuracy from the maximum allowable beam current. The yield from a 1-µm thick nickel foil containing 1 ppm helium, using 30-MeV alpha particles at which energy the 45° alpha-alpha elastic scattering cross section is 0.57×10^{-24} cm^2 per steradian, and assuming a detector solid angle of 2×10^{-3} steradians and that the channel width is equal to the foil thickness, is:

$$Y = 0.033 \text{ counts}/\mu \text{ coulomb} \quad (21)$$

Since the maximum alpha particle current on a thin foil is limited to about 0.1 µA due to high background count rate, it will take 300 s (5 min) to accumulate 1 count. In order to obtain 100 counts, which will provide 10 % accuracy, the foil must be irradiated for 500 min or 83 h. Thus, a helium concentration of 1 ppm is the practical limit to the minimum detection capability of the alpha-alpha technique.

6.2.6.8 Detection sensitivity can be doubled or tripled by doubling or tripling the number of pairs of detectors that view the target. Alternatively, the use of standards consisting of target foils of known thickness with shallow surface implants of known helium concentrations can be used to calculate actual helium concentrations in specimens. Concentrations on the order of 1 ppm can be measured on thin foils (>25 µm) but irradiation times as long as 10 h may be required. A depth resolving power of 0.3 µm in a 2 µm thick nickel foil has been obtained using 18 MeV alpha particles.

7. Keywords

7.1 helium analysis; helium effects; ion irradiation; radiation damage; tritium trick

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