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E706 Master Matrix for Light-Water Reactor Pressure Vessel Surveillance Standards, E 706(0) (Withdrawn 2011)[4]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.).

---

[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: