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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.).

¹ This guide is under the jurisdiction of ASTM Committee **E10** on Nuclear Technology and Applications and is the direct responsibility of Subcommittee **E10.05** on Nuclear Radiation Metrology.

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² 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 Degradator*—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