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Sub-size tensile specimen design for in-reactor irradiation and post-irradiation testing

Maxim N. Gussev *, Richard H. Howard, Kurt A. Terrani, Kevin G. Field

Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

HIGHLIGHTS

- New specimen geometry for post-irradiation testing (PIE) was designed.
- The new geometry performance was evaluated for several commercial and model alloys.
- Scale factor role was discussed in detail.

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ABSTRACT

The present work aims to provide a complete engineering solution, an appropriate experimental database, and a brief physical background on designing a miniature specimen geometry suitable for irradiation in materials test reactors such as the High Flux Isotope Reactor and post-irradiation out-of-hot cell testing. The physical limits of specimen miniaturization and a background of the scale factor effect are discussed, and principal limitations are defined. The advantages of modern test methods like digital image correlation, as well as some limitations connected to small specimen size, are analyzed. A sub-sized specimen geometry, "SS-Mini," is designed; the geometry employs existing irradiation capsules leading to the reduced cost of any irradiation campaign. The new geometry performance is evaluated using a commercial 304 L stainless steel, an aluminum alloy including advanced 3D-printed material, a high nickel 718-alloy, tungsten, and an advanced fuel cladding FeCrAl alloy. Mechanical tests are conducted to compare the engineering mechanical properties (yield and ultimate tensile stress, uniform and total elongation values) and plastic behavior of the proposed miniature specimen with common specimen types for irradiation testing.

1. Introduction

There is a constant and growing interest in the mechanical testing of small specimens and analyzing material behavior at small scales. In general materials science, this interest is driven by introducing smaller devices and exploring the micro- and nanomechanics areas (Connolley et al., 2005; Jaya and Alam, 2013). The goal is to analyze the properties and performance of microparts and components in detail while understanding materials behavior at lower length scales.

At the same time, the nuclear industry often has a different goal (Klueh, 1985; Kohno et al., 2000; Hindley et al., 2015; Viehrig et al., 2015): testing of miniature samples taken from nuclear installations should provide information that is applicable to bulk mechanical properties or at a minimum yield coherent scaling parameters from small scale testing. The motivation for small-scale mechanical testing in the nuclear industry is to best take advantage of the limited experimental space in materials test reactors and minimize the radiological dose produced from activated materials that directly scales with their volume.

Additionally, recent progress in different in-situ test methods, where small scale testing is routinely needed, is impressive (Dehm et al., 2006), and the next decade is likely to see a considerable rise in the in-situ test techniques for general materials science and nuclear engineering. For instance, micro-pillar testing (Dehm et al., 2006; Shin et al., 2015), high-resolution EBSD (Jiang et al., 2013), advanced tools for 3D structure reconstruction, and other techniques are quickly providing new, unique, and—most importantly—insightful data. Employing these methods is an attractive idea for the investigation of radiation effects in materials, including plastic strain at small scales (Greer and De Hosson, 2011).

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* Corresponding author at: PO Box 2008, Oak Ridge, TN 37831, USA.
E-mail address: gussevmn@ornl.gov (M.N. Gussev).

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Moreover, the development of innovative nuclear systems constantly requires new materials, which, in turn, means increasing needs for advanced comprehensive testing methods (Fazio et al., 2011; Yvon et al., 2015), like in-situ corrosion testing (Klecka et al., 2015), while simultaneously reducing the time to achieve such data and analysis.

The deployment of the advanced in-situ techniques on neutron irradiated materials is of special importance. Although significant progress was made to use light ion and heavy ion irradiations to simulate neutron irradiation while producing samples with little or no radioactivity [e.g., see (Was et al., 2002)], the discrepancies in the observed microstructure and mechanical properties still exist. Hence, data for neutron irradiated materials are still essential to fully develop a nuclear-grade alloy ripe for commercial deployment.

Irradiation may stimulate specific processes not observed without irradiation, including swelling, increased diffusion and creep, radiation-induced segregation (RIS), and irradiation-assisted stress corrosion cracking (IASCC), just to name a few. Advanced in-situ tools may bring new insights into well-known processes. For instance, a preliminary, not-on-purpose analysis of the deformation localization in neutron irradiated austenitic steels revealed specific phase transformation at the irradiated specimen's surface (Gussev et al., 2014b) along with areas of localized deformation and high dislocation density spots (Field et al. 2014a) in addition to the expected and well-known defect-free channels. It is important to be able to test the irradiated specimen in-situ, registering, for example, the evolution of dislocation density and the evolution of local misorientation (Jiang et al., 2013) with direct measurements of acting stress or observations of stress corrosion crack appearance and propagation. These data, coupled with modern modeling tools, allow for complex analysis of material behavior, including constitutive crystal plasticity approaches. However, to reach these goals, one has to at least have direct access to the specimen for preparing the appropriate quality surface.

Miniature sub-size specimens for post-radiation testing have a long history and are widely used for investigating mechanical properties and deformation-hardening behavior of irradiated and non-irradiated metals and alloys (Klueh, 1985; Panayotou et al., 1986; Kohyama et al., 1991; Kohno et al., 2000; Gussev et al., 2014a), weldments (Field et al., 2014b), nanostructured materials, and different composites. A variety of different sizes and geometries are used in nuclear materials science (Klueh, 1985; Pierron et al., 2003; Wakai et al., 2011), with new geometries and testing methods being constantly developed (Rickerby and Fenici, 1986; Džugan et al., 2014; Hurst and Matocha, 2015; Rund et al., 2015). Specimen miniaturization often involved other methods, not only tensile testing. Thus, Jung et al., (1996) offered a miniature tensile specimen with gauge section of 5 × 1 × 0.4 mm; additionally, a number of testing methods for miniature specimens (tensile, facture toughness, punching, impact testing) were discussed in detail (Jung et al., 1996) focusing on the detailed correlation between miniature and bulk behavior. The offered tensile specimen geometry was intensively used in a number of projects (see for instance, (Dai and Bauer, 2001)).

Through the past few decades, there has been a tendency to decrease the overall specimen size and volume (Klueh, 1985). Prior to this work, two miniature tensile specimen geometries were commonly used for irradiations in the High Flux Isotope Reactor (HFIR): the SS-3 and the SS-J type specimens (Klueh, 1985; Gussev et al., 2014a). These two geometries provide acceptable mechanical properties compared to standard-scale specimens (Gussev et al., 2014a). However, the post-irradiation activity level is usually too high for nuclear reactor metals after discharge, and this limits the post-irradiation analysis to testing within a hot cell facility only, especially when moderate to high damage dose (dpa) samples are of interest. Reaching the acceptable level of radioactivity may require unacceptable waiting times that exceed the lifetime of the funding program or project interested in such data.

Thus, there is a need for a specimen geometry capable of supporting the modern in-situ testing methods on moderate to highly neutron irradiated materials, while still providing repeatable bulk-scale mechanical properties. It is important for the new geometry to provide appropriate mechanical test results and keep a continuity and comparability with the existing databases in the literature. If suitable, these small-scale mechanical testing specimens impact scientific productivity in the nuclear materials area in two important ways. First, by avoiding in-cell testing, a whole host of new characterization techniques too costly or simply inviable for in-cell deployment—where high radiation fields detrimentally interact with electronic components—suddenly become available for utilization. Second, the costs associated with in-cell testing are often very high (e.g., at least a factor of 2–3x higher at ORNL) and would, in turn, limit the depth and breadth of the R&D project.

The circumstances discussed above motivated the development of a new specimen geometry allowing for: (1) in-HFIR irradiation within the existing "rabbit capsule" geometry and specifications, (2) out-of-hot cell testing with short "cool-down" (reduction in radioactivity by decay) time, and (3) the capability to perform in-situ measurements using advanced techniques. The present work discusses the design, geometry, and implementation of a miniature sub-size specimen that meets these requirements.

2. The background of scale and size effects

The scale factor and size effects usually become apparent when the specimen size approaches the characteristic length scale of the material microstructure (Connolley et al., 2005). For common steels and alloys, this tends to be the grain size. Another size effect, often observed in brittle materials (Bažant, 1999), occurs when the size of the specimen becomes small compared to the spatial distribution and density of critical defects. Additionally, size effects may appear due to physical, chemical, or corrosion processes that are negligible for relatively large objects but become critical when the size of the test specimen decreases. Most of these processes, like oxidation, usually do not appear in common mechanical tests; however, some important physical phenomena sensitive to the scale factor are briefly analyzed below.

Considering the scale factor role, researchers most often focus on the uniform strain area, where the uniaxial stress state exists, and on the necking behavior, where the complex stress state and the stress triaxiality play an important role.

In the first case, under uniaxial stress, the specimen is often presented as a "multilayer" object with a layer of near-surface grains. The ratio of the "near-surface grains" to the "bulk grains" impacts the specimen deformation behavior and strain hardening rate (Wang et al., 2013; Shin et al., 2015). The near-surface grains (or the near-surface layer if a single crystal specimen is considered) have smaller dislocation density and internal back stress level compared to the bulk grains (Keller et al., 2010; Shin et al., 2015) because of the dislocation escape through the free surface. Also, the surface plays a key role in crack initiation during fatigue (Signor et al., 2016) and stress corrosion cracking (SCC). To handle very thin specimens (10–20 μm sheets), some special test method(s) may be needed. For instance, Hoffman and Hong (Hoffmann and Hong, 2006) offered an aero-bulging test method for testing thin foils of different materials.

It is worth noting that the near-surface layer is not necessarily "weak;" that is to say, it does not necessarily have a decreased strength. Often, specimens are produced by electric discharge machining (EDM) followed by mechanical grinding or polishing