

# Microstructure and mechanical behavior of neutron irradiated ultrafine grained ferritic steel



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## ABSTRACT

Neutron irradiation effects on ultra-fine grain (UFG) low carbon steel prepared by equal channel angular pressing (ECAP) have been examined. Counterpart samples with conventional grain (CG) sizes have been irradiated alongside with the UFG ones for comparison. Samples were irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) to 1.37 dpa. Atom probe tomography revealed manganese and silicon-enriched clusters in both UFG and CG steel after neutron irradiation. Mechanical properties were characterized using microhardness and tensile tests, and irradiation of UFG carbon steel revealed minute radiation effects in contrast to the distinct radiation hardening and reduction of ductility in its CG counterpart. After irradiation, micro hardness indicated increases of around 9% for UFG versus 62% for CG steel. Similarly, tensile strength revealed increases of 8% and 94% respectively for UFG and CG steels while corresponding decreases in ductility were 56% versus 82%. X-ray quantitative analysis showed that dislocation density in CG increased after irradiation while no significant change was observed in UFG steel, revealing better radiation tolerance. Quantitative correlations between experimental results and modeling were demonstrated based on irradiation induced precipitate strengthening and dislocation forest hardening mechanisms.

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## 1. Introduction

Advanced nuclear reactors employ higher temperature environments and require higher irradiation exposures for core structural components and fuel cladding compared to the current operating light water reactors (LWR) [1,2]. Thus, development of new materials with superior properties which can withstand such severe conditions is required to fill these needs [3–5]. Ferritic steels have been used widely as structural materials in light water reactors, but they suffer from irradiation hardening and embrittlement accompanied by increased ductile to brittle transition temperature (DBTT) and decreased upper shelf energy [6]. Previous studies have shown that major factors affecting low carbon steels under neutron irradiation are neutron fluence, irradiation temperature and chemical composition [7]. However, designing materials with tailored response that can sustain high amounts of radiation damage while maintaining their mechanical properties is a grand challenge in materials research [8]. During irradiation, point defects (vacancies and interstitials) are produced as

a result of displacement cascade [9–11]. These point defects can cluster to form other types of defects that will alter the mechanical properties of irradiated materials. One method to suppress accumulation of these point defects is by annihilating them at interfaces such as grain boundaries. Ultra-fine grained materials are expected to be more radiation tolerant since grain refinement increases the area of grain boundaries which can act as sinks for radiation induced point defects.

It has been theorized that the large amount of grain boundary area will help to prevent accumulation of defects which can adversely affect mechanical properties [12–20]. Singh [13] showed that void swelling in electron irradiated helium doped stainless steel decreases as the grain size decreases. Rose et al. [21] illustrated using TEM that the defect density in ZrO<sub>2</sub> irradiated by Kr ions reduces as the grain size decreases. Matsouka et al. [22] studied the effects of neutron irradiation on UFG SUS316L stainless steel and their TEM observations revealed defect-free zones along grain boundaries suggesting that the grain boundaries are acting as sinks for radiation induced defects. Kurishita et al. [23] showed that the density of voids in UFG W-0.5 wt %TiC is much lower than that in CG tungsten after neutron irradiation at 600 °C. Sun et al. [24] found that dislocation loops and He bubble densities in UFG Fe–Cr–Ni alloy after He ion irradiation are less than

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those in its CG counterpart. Computer simulation studies [14,25–27] demonstrated that materials with large surface area of interfaces or grain boundaries have a potential to increase irradiation resistance. The effect of a small grain size (large grain boundary area density) on radiation tolerance of a low-carbon ferritic steel is assessed in this study.

## 2. Materials and methods

### 2.1. Materials

Two material cases are considered in this study; ultra-fine grain low carbon steel with a composition of 0.1 C, 0.5 Mn, 0.27 Si with balance of Fe in wt% processed through ECAP [28–30] and their conventional coarse grain counterparts that were produced by annealing the UFG material at 800 °C for one hour [12]. UFG steel was made by ECAP using Bc route [31] with four passes where the material is rotated 90° in the same direction after each pass. This processing route ensures eventual restoration of the material cubic element [32]. More details about ECAP routes can be found elsewhere [33,34].

### 2.2. Irradiation experiment

The irradiation experiment was carried out in the E-7 position of the East Flux Trap (Fig. 1b [35]) in the Advanced Test Reactor (ATR) at Idaho National Laboratory. The materials were irradiated to a neutron fluence of  $1.78 \times 10^{25}$  neutrons/m<sup>2</sup> ( $E > 0.1$  MeV) corresponding to a dose of 1.37 dpa. Fig. 1a is a schematic of the irradiation test assembly consisting of the experimental basket, support rod and capsule assemblies. The support rod was inserted

at the bottom of the experimental basket to ensure that the test capsules are at the maximum flux location. The experimental basket of the test assembly is an aluminum tube designed for insertion in the capsule assembly in the ATR. The basket was designed such that there is adequate coolant circulation to prevent temperature distortions or mechanical effects, and that there is adequate mechanical support to secure the test capsule throughout the reactor insertion, irradiation, and removal from the reactor. Samples were cut from the bulk ECAP material and were prepared by grinding with a series of silicon carbide papers (600, 800 and 1200 grits) to optical flatness and then polished in colloidal silica resulting in deformation free surfaces. The prepared samples (UFG and CG steels) were loaded in sample holders (Fig. 2a) and a thin aluminum disc was tack-welded to the open end of each holder to position the samples inside it. Each group of sample holders was strung together using aluminum rods and was assembled into a sample train which was designed to hold the samples for easier removal following irradiation and also to help keeping the samples at the desired irradiation temperature (Fig. 2b, c and d). Finally, the sample trains were sealed in a stainless steel containment capsule that is essentially a sealed pressure vessel filled with helium (Fig. 2e). The integrity of the capsules was ensured using helium leak testing, dye penetrant testing and visual inspection.

Thermal analysis was performed using a detailed finite element model of the experiment using ABAQUS code [36]. MCNP code [37] was used to calculate the heat generation rate for each part of the experiment which was then used as an input to the finite element model (Fig. 3). The specimen temperature during irradiation was found to vary between 70 °C to less than and 100 °C depending on the sample position in the irradiation capsule. The low irradiation temperature is mainly due to the small helium gas gaps between specimens and holder, and between holder and capsule.

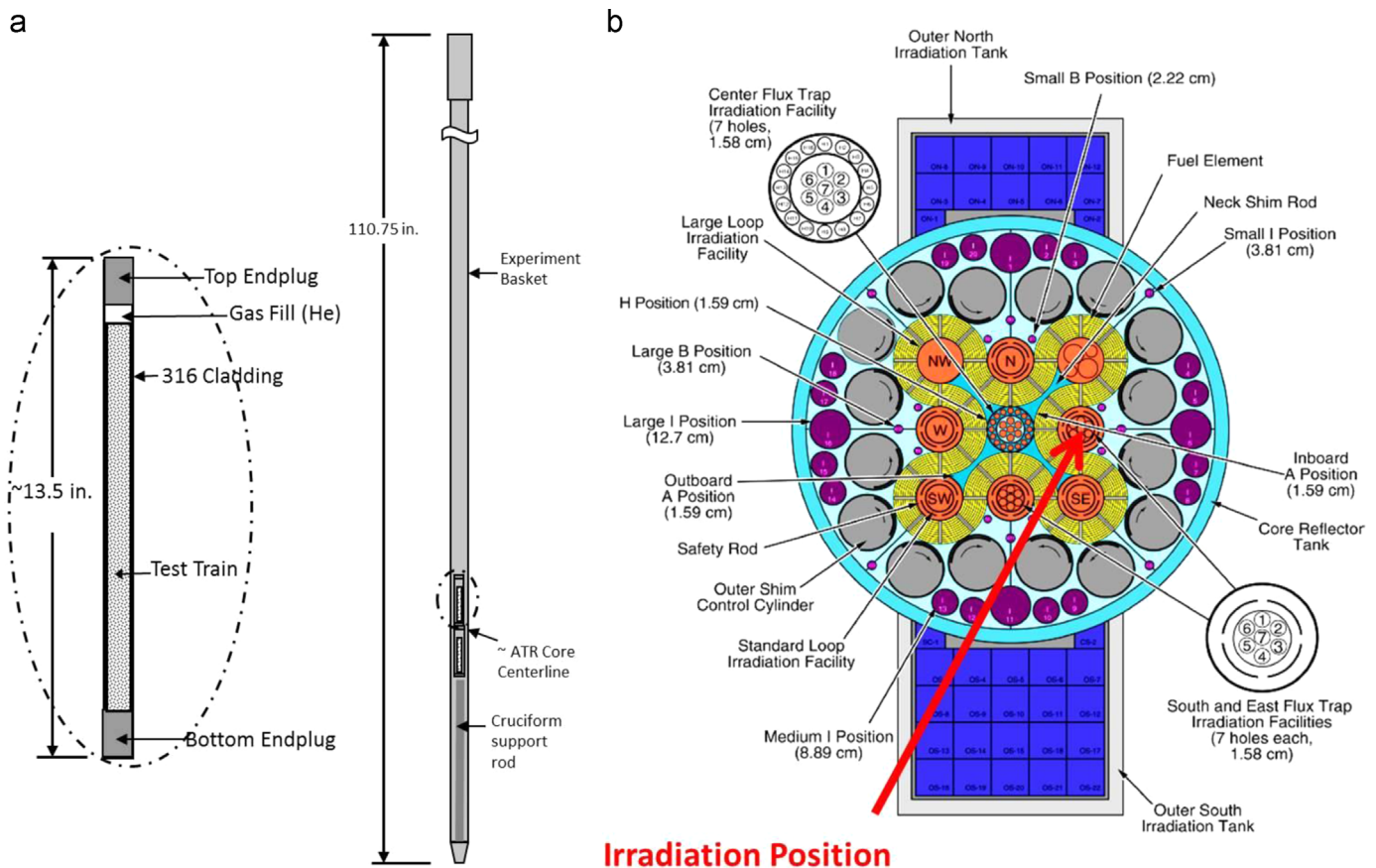


Fig. 1. Irradiation Test Assembly for ATR East Flux Trap Position (a) and Radial Cross Section View of the ATR Reactor Core, E-7 Irradiation Test Position (b) [35].

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