

## Enhancing irradiation tolerance of steels via nanostructuring by innovative manufacturing techniques

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The proposed project will involve neutron irradiation and post-irradiation examination of bulk nanostructured ferritic/martensitic steels that are anticipated to have enhanced irradiation tolerance and are produced by two innovative, low-cost manufacturing techniques, equal-channel angular pressing (ECAP) and high-pressure torsion (HPT). The objectives of our proposed research are to establish/enhance our fundamental understanding of irradiation effects in ultrafine-grained (100 nm < grain diameter < 1  $\mu$ m) or nanocrystalline (grain diameter < 100 nm) steels produced by ECAP or HPT, and to assess the potential applications of ECAP and HPT in fabricating materials for applications in current and advanced reactors. Improving the performance of currently used ferritic/martensitic steels through microstructural engineering via advanced manufacturing techniques provides high potential to improve radiation tolerance at relatively low cost compared to development of new alloys.

ECAP and HPT produce ultrafine or nanocrystalline grain sizes in metals and alloys through application of severe plastic deformation. It is relatively simple to perform ECAP and HPT at relatively low cost. Alloys produced by ECAP or HPT possess dramatically higher strength than their conventionally processed counterparts, owing to significant grain boundary strengthening, and have significantly enhanced irradiation tolerance due to significant volume fraction of grain boundaries that serve as sinks or recombination centers for radiation-induced defects. Ferritic steels are considered for application as fuel cladding materials for accident tolerant fuel for light-water reactors (LWRs), and ferritic/martensitic steels are leading fuel cladding and structural materials for advanced fast reactors. Life extension of LWRs and development of fast reactors require steels with enhanced irradiation tolerance and higher strength. This work will establish the performance of ultrafine-grained and nanocrystalline variants of reactor structural and cladding steels produced by ECAP or HPT, under neutron irradiation at relevant reactor operating temperatures, which has not previously been accomplished. Our proposed project will be focused on neutron irradiation and post-irradiation examination of ultrafine-grained or nanocrystalline ferritic/martensitic steels; these materials have been produced by our collaborators using ECAP or HPT.

In our proposed project, conventionally processed coarse-grained, ECAP processed ultrafine-grained and HPT processed nanocrystalline ferritic/martensitic Grade 91 steel will be subjected to neutron irradiation at different temperatures with different neutron doses. Conventionally processed steels provide a baseline for comparison to ECAP- and HPT-processed steels. All sample sets will be characterized in the un-irradiated and irradiated states.

Both the pre- and post-irradiation examinations will include mechanical testing and microstructural examination. Mechanical testing will include tension and creep. Researchers, including



the PI, have established that ultrafine-grained and nanocrystalline alloys can have good thermal stability and creep resistance, due to pinning of grain boundaries by solute segregation and precipitates at grain boundaries. In this proposed project, thermal stability study via annealing will be included in the pre-irradiation examination.

A variety of advanced microstructural characterization techniques will be employed for pre- and post-irradiation examination. Because the volume fractions of grain boundaries in the ultrafine-grained and nanocrystalline steels are significant, grain boundary characteristics (e.g., type, plane, and misorientation) are very important in influencing the behavior of the materials. Electron backscatter diffraction, transmission Kikuchi diffraction and precession electron diffraction derived ASTAR will be performed to study grain boundary characteristics at the micrometer, submicron and nanometer scales, respectively. (High resolution) transmission electron microscopy will be utilized to characterize the irradiation-induced defects and determine their structures. Scanning transmission electron microscopy, coupled with energy dispersive x-ray spectroscopy or electron energy loss spectroscopy, and energy-filtered transmission electron microscopy will be performed to generally identify solute segregation and precipitates, roughly determine their composition, and study their distribution. Atom probe tomography will be utilized to measure accurately and quantitatively the distribution and compositions of solute segregation, solute clusters, and precipitates.

Mechanical properties and microstructures of the steels after irradiation will be compared to those before irradiation so that the irradiation effects on microstructural evolution and mechanical properties can be determined, with focus on irradiation-induced hardening, solute segregation, and phase transformation. Irradiation effects in coarse-grained, ultrafine-grained and nanocrystalline steels will be compared, and irradiation tolerance will be studied as a function of grain size. The interaction between irradiation-induced defects and grain boundaries in the steels will be investigated in relation to the specific characteristics of grain boundaries, and the mechanisms by which grain boundaries reduce irradiation-induced defects and enhance irradiation resistance will be studied. The irradiation-induced solute segregation at grain boundaries will also be related to the specific characteristics of grain boundaries.

Multiscale modeling and simulation will be utilized to assist understanding experimental results on ultrafine-grained and nanocrystallline samples. In particular, molecular dynamics simulations will be used to study the interactions between grain boundaries and irradiation-induced defects. Irradiation-induced segregation will be investigated by kinetic Monte Carlo, with the defect - grain boundary interactions informed by molecular dynamics simulations. The effect of solute segregation at grain boundaries on the stability of nanocrystalline samples before and after irradiation will be evaluated via phase field modeling using the MARMOT code. Furthermore, molecular dynamics simulations will be applied to investigate possible irradiation-induced hardening.

The outcomes of the proposed research will be feasibility assessment of applications of two low-cost advanced manufacturing techniques (ECAP and HPT) in fabricating materials with improved performance for current and advanced reactors, with an established/enhanced understanding of the irradiation effects in ultrafine-grained and nanocrystalline ferritic/martensitic steels. The application of low-cost advanced manufacturing techniques in fabricating currently used materials in LWRs and advanced reactors to achieve microstructural engineering and improved performance will contribute to life extension of LWRs and facilitate development of advanced fast reactors. Hence, the proposed research is highly relevant to DOE-NE's Light Water Reactor Sustainability program and Advanced Fast Reactor program.