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# Multiple ion beam irradiation for the study of radiation damage in materials



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### ABSTRACT

The effects of transmutation produced helium and hydrogen must be included in ion irradiation experiments to emulate the microstructure of reactor irradiated materials. Descriptions of the criteria and systems necessary for multiple ion beam irradiation are presented and validated experimentally. A calculation methodology was developed to quantify the spatial distribution, implantation depth and amount of energy-degraded and implanted light ions when using a thin foil rotating energy degrader during multi-ion beam irradiation. A dual ion implantation using 1.34 MeV Fe<sup>+</sup> ions and energy-degraded D<sup>+</sup> ions was conducted on single crystal silicon to benchmark the dosimetry used for multi-ion beam irradiations. Secondary Ion Mass Spectroscopy (SIMS) analysis showed good agreement with calculations of the peak implantation depth and the total amount of iron and deuterium implanted. The results establish the capability to quantify the ion fluence from both heavy ion beams and energy-degraded light ion beams for the purpose of using multi-ion beam irradiations to emulate reactor irradiated microstructures.

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#### 1. Introduction

Ion irradiation experiments using light and heavy ion beams have been used for decades to explore radiation damage processes. Only recently have they been applied as surrogates for reactor irradiation. Proton irradiations with damage rates of  $10^{-5}$  dpa/s have demonstrated much success in determining the mechanistic behavior of materials under light water reactor relevant conditions [1]. Heavy ion irradiation experiments with damage rates of  $10^{-3}$  to  $10^{-4}$  dpa/s have replicated the microstructure observed in reactor irradiated materials also with good success [2,3]. These experiments have provided optimism for using ion irradiations as a surrogate for reactor irradiation.

Helium is known to play a role in the development of the irradiated microstructure with modifications to cavities [4–8], dislocations [9–11], and secondary phases [12–16]. Previous studies on the effects of helium using ion irradiation have used either pre-implanted or co-injected helium. The swelling behavior under ion irradiation is influenced by the mode of helium injection

[17,18]. Kohyama et al. [8] used several ion irradiation schemes to assess the impact of helium injection mode on the development of cavities. Pre-injection of helium was postulated to produce a high number density of essentially immobile defects. The use of nickel ions to create a damaged microstructure followed by dual ion irradiation with nickel and helium resulted in the highest amount of swelling compared with dual ion (Ni + He) or single ion irradiation (Ni) separately. Hydrogen has been shown to modify the irradiated microstructure. Zhanbing et al. [19] irradiated a 12Cr-ODS ferritic steel to 15 dpa using dual-beam irradiation of hydrogen ions and electrons. The results showed that the dislocations were introduced at the initial stage of irradiation and were enhanced by the presence of hydrogen before developing into dislocation networks. To capture all of the effects of transmutation gas products, both helium and hydrogen need to be injected into the sample simultaneously with damage production.

The combination of helium and hydrogen together suggest a synergistic effect on the evolution of the microstructure in materials under irradiation [20–25]. The work of Wakai et al. demonstrated this synergistic effect under simultaneous displacement damage from 10.5 MeV Fe<sup>3+</sup> ions with the co-injection of 1.05 MeV He<sup>\*</sup> and 0.38 MeV H<sup>\*</sup> in the ferritic-martensitic steel

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F82H. Swelling was increased with larger cavities developing with an accompanying increase in microhardness in a ferriticmartensitic steel as compared to displacement damage combined with either helium or hydrogen co-injection alone. From these works and others [26], it is clear that multi-ion beam irradiations facilities are required to capture the synergistic behavior of gas injection and radiation damage that occurs in reactor.

Several multi-beam irradiation facilities around the world have provided significant insight into radiation damage processes, such as the facilities at TIARA [27], DuET [28], HIT [29], FZ Rossendorf [30], FSU Iena [31], LANL [32], JANNUS [33,34], the former facility at Oak Ridge National Laboratory [35] and others listed in Table 3 of reference [36]. These facilities use multiple ion accelerators in which one provides heavy ion beams to induce damage and the others to inject light ions across a range of depths in the target. A common method of controlling the light ion distribution in the target is to use multiple foils with different thicknesses on a rotating wheel [28]. A second method uses a single foil rotated in front of the beam [27]. To study the effects of simultaneous radiation damage with multiple ion beam injection, several conditions must be met and demonstrated with high fidelity. This work presents the description and validation of the systems required to perform multi-ion beam irradiation experiments for the purposes of emulating reactor environments. The major components and methodologies used at the Michigan Ion Beam Laboratory are described, including the multi-beam irradiation chamber, the irradiation stage, the thermal control systems, the dosimetry, and the thin foil energy degrader. Experiments to benchmark and validate the methods are presented as a proof-of-concept.

#### 2. Experimental design

To allow for rigorous studies of simultaneous radiation damage and ion injection using multiple ion beams, the following conditions must be met:

- i. Ion beams from multiple accelerators must be aligned so that they overlap on a single plane (the target surface).
- ii. The ion beams need to be uniform across the irradiated area.
- iii. The temperature of the irradiated area must be monitored and controlled to minimize the variability over the duration of the experiment, as quantified by the  $2\sigma$  variation in temperature.
- iv. The measurement of the total ion fluence for each species must be accurate.

#### 2.1. Accelerators and laboratory layout

The Michigan Ion Beam Laboratory includes three accelerators: a 3 MV NEC Pelletron accelerator, a 1.7 MV General Ionex Tandem accelerator, and a 400 kV NEC Ion Implanter shown in Fig. 1. The 3 MV NEC Pelletron is a tandem accelerator used primarily for ion irradiations. The accelerator is equipped with a Peabody PS120 sputter source to generate negative ions from solid targets through sputtering and a high brightness Toroidal Volume Ion Source (TORVIS) capable of producing hydrogen and deuterium ions. The 1.7 MV General Ionex Tandem accelerator has both an Electron Cyclotron Resonance (ECR) ion source for production of positive ions with any gas, and a Multi-Cathode Source of Negative Ions by Cesium Sputtering (MC-SNICS). The 400 kV NEC Ion Implanter is a single ended accelerator where positive ions formed in the Danfvsik 921A ion source are passed through a 90° bending magnet before acceleration in the accelerator tube. The ion source can operate in three modes: gas mode with a direct gas feed, liquid mode with ions produces through the vaporization of volatile compounds in a separate oven, or sputtering mode with ions produced from a solid target with argon ions. The accelerators are housed in the accelerator room and the beamlines pass through a 1.2 m thick wall into the target room that houses multiple end stations.



Fig. 1. The 3 MV NEC Pelletron accelerator "Wolverine", 1.7 MV General Ionex Tandem accelerator "Maize", and 400 kV NEC Ion Implanter "Blue" in the Michigan Ion Beam Laboratory.

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