Graphical Abstract

Intermediate energy proton irradiation: rapid, high-fidelity materials testing for fusion and fission energy systems

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Highlights

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- Proton-induced recoils have the relevant energy range for emulating fusion and fission reactor conditions
- Proton-induced helium and hydrogen production is controllable and able to match a wide range of reactor conditions
- Intermediate-energy proton irradiation allows higher dose-rates (0.1–1 dpa/day) than fission reactors with bulk samples (100-300µm), less radioactivity, and minimal temperature gradients.
- 12 MeV proton irradiation and tensile testing is shown to mimic previous reactor irradiation data.
Intermediate energy proton irradiation: rapid, high-fidelity materials testing for fusion and fission energy systems

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\textbf{ABSTRACT}

Fusion and advanced fission power plants require advanced nuclear materials to function under new, extreme environments. Understanding the evolution of mechanical and functional properties during radiation damage is essential to the design and commercial deployment of these systems. To address shortcomings of existing methods, we propose a new technique - intermediate energy proton irradiation (IEPI) - using beams of 10 - 30 MeV protons to rapidly and uniformly damage bulk material specimens before direct testing of engineering properties. We show that IEPI achieves high fidelity to fusion and fission environments in both primary damage production and transmutation, often superior to nuclear reactor or typical (heavy or low-energy light) ion irradiation. Modeling demonstrates that high dose rates (0.1–1 DPA per day) can be achieved in bulk material specimens (100–300 µm) with low temperature gradients and induced radioactivity. We demonstrate the capabilities of IEPI through a 12 MeV proton irradiation and tensile test of 250 µm thick tensile specimens of a nickel alloy (Alloy 718), reproducing neutron-induced data. These results demonstrate that IEPI enables high throughput assessment of materials under reactor-relevant conditions, positioning IEPI to accelerate the pace of engineering-scale radiation damage testing and allow for quicker and more effective design of nuclear energy systems.

1. \textbf{Introduction: opportunities for intermediate-energy proton irradiation}

Future fusion power plants and advanced nuclear fission reactors are being pursued as clean and affordable energy sources to mitigate global climate change. Fully realizing the potential of these technologies will require the development and qualification of materials that satisfy demanding operational requirements while surviving in extreme environments \cite{1,2,3}. Perhaps the most challenging aspects of materials in these systems are the exposure to large fluxes and fluences of radiation on the structural and functional materials used in the core of each plant. For example, materials surrounding the core of a fusion power plant are expected to receive total neutron fluxes of $10^{14}$–$10^{15}$ cm$^{-2}$ s$^{-1}$, equivalent to up to 15 displacements/atom (DPA) per year \cite{4}. Such high levels of neutron exposure will cause undesirable property changes such as hardening, embrittlement, and decreases in thermal conductivity \cite{5}. In both technologies, materials play a decisive role in determining the performance, maintainability, safety, and lifetime of the plants, which ultimately sets the economic viability of each technology \cite{6,7,8}.

Understanding and mitigating radiation effects requires the irradiation of materials with neutrons or ions in dedicated experimental facilities. These facilities are flexible platforms for studying a wide range of radiation-induced phenomena. A key objective in irradiation experiments is high fidelity to the application environment to maximize the relevance of the measured material response and our ability to predict true response of nuclear materials in service.

Radiation damage in materials has long been studied with neutron irradiation in fission test reactors. Due to the long mean-free path of reactor-spectrum neutrons (~ cm) in materials, the damage induced is inherently bulk, and the induced changes can be assessed in two complementary ways: first, through the use of macro-scale techniques such as tensile, fracture, and impact testing to directly extract engineering-scale properties; and second, through the use of techniques like electron microscopy, atom-probe tomography, and x-ray diffraction, that provide physical insight into the underlying microstructural evolution. The opportunity to experimentally link changes in the engineering properties to the changing microstructure is an critical advantage of reactor irradiation.

Despite the advantages of reactor irradiation, its high costs and time requirements limit its use in studying radiation damage. Because advanced fission and fusion energy system designs can have material dose requirements approaching 200 DPA, thermal test reactors (~5 DPA/year) and even fast test reactors (~20 DPA/year) are not practical options for frequent high dose experiments \cite{10}. Irradiation in test reactors typically leads to high levels of radioactivity, requiring significant post-irradiation dwell times (months – years) and specialized safety equipment during post-irradiation analysis. Due to the complexities of operating fission reactors, fission reactor experiments can cost millions of dollars and can require years or even a decade to complete \cite{10}. Reactor irradiation struggles to meet the wide range of irradiation conditions (e.g. dose, temperature) and material characteristics (composition and mechanical/thermal processing) needed for the development and qualification of new materials for next generation fission and fusion systems \cite{10}.

The difference in neutron energy spectra between current test reactors and future (e.g. fusion) reactors is an obstacle to predicting material performance. For example, a tungsten...
Intermediate-energy proton irradiation

Figure 1: Ion ranges in four common elements demonstrate the ability of intermediate-energy protons to achieve bulk penetration, allowing exploration of irradiated material properties at the macroscopic engineering scale. Data from SRIM [9].

Component in a fusion reactor is expected to produce several atomic-parts-per-million (appm) of helium per year [4, 11], which will stabilize voids and promote swelling [12]. Both thermal and fast reactors lack the high energy neutrons required to generate comparable amount of helium [13–15]. Conversely, the large fraction of thermal neutrons in test reactors, which are not present in fusion first wall materials, cause artificially high production of dissolved impurities and the formation of embrittling precipitates [5]. These inherent differences in neutron energy spectra limit the accuracy with which future fusion and fission reactor conditions can be emulated with current test reactor technology.

The irradiation of materials with ion beams has been widely adopted by the nuclear materials community as a faster, more flexible tool for studying radiation damage effects [10]. Large ion currents (1 µA to 1 mA) enables dose rates on the order of DPA/day-DPA/hr [16] providing access to the life-time doses expected in advanced fission and fusion systems in practical experimental times. Most ion irradiations are performed with relatively low energy (∼ MeV) ions and avoid any radiation-producing nuclear reactions. This allows relatively low-cost and flexible experimental facilities and accelerates post-irradiation testing. While neutron irradiation has remained the gold-standard for assessing materials, proton irradiation has reproduced the material response of neutron irradiation across a variety of microstructural effects (e.g. segregation and loop formation) and property changes (e.g. hardening) [16].

A principal limitation of ion irradiation is the microscopic thickness of the damaged region achievable given the limited range of ion beams. Due to technology and cost limitations, the vast majority of ion accelerators available for materials irradiation provide relatively low energy light ions (e.g. protons ≤ 5 MeV) or high Z ions (e.g. self-ions, swift heavy ions) [17]. The ranges of such beams are typically micrometers or less, with sharp spatial gradients in amount of damage. This range limitation complicates the ability to extract engineering properties, restricting applicable tests to micro- or nano-scale techniques such as indentation testing and preventing bulk measurements of strength, ductility, toughness, and creep. The limited micro-scale testing available to ion-irradiation experiments is susceptible to distortion by the effects of denuded zones [16], injected ions [18], and carbon contamination [19, 20], which are artifacts of ion irradiation. Polycrystalline, multi-phase, or composite materials are difficult or impossible to study with conventional ion techniques because the material length scales of interest are much larger than the irradiation regions.

Intermediate-energy (10–30 MeV) protons offer the possibility of combining the benefits of ion irradiation (e.g. high dose rate, flexible irradiation conditions) and reactor neutron irradiation (uniform/bulk irradiation, direct engineering testing). As the lightest ion, protons have the lowest stopping power and greatest range in materials. As shown in Figure 1, protons above 10 MeV are able to penetrate more than 100 µm in typical metals - a length scale at which direct testing of mechanical properties becomes feasible - without inducing the the confounding effects associated with low-range ions. As with all ion irradiation, high dose rates are achievable and can be enhanced by increasing the ion currents.

Traditionally, accelerators providing protons with energy above 10 MeV (and beam currents above ∼1 µA) have been rare due to their high capital and operating costs, large phys-
ical size, electricity consumption, and staffing requirements. These machines are dedicated almost exclusively to nuclear and astrophysical research [21] or the production of isotopes for research and medicine [22]. Recent accelerator advances, particularly in commercially availability of ultracompact superconducting cyclotrons [23] are enabling the production and utilization of intermediate-energy ion beams of relatively high current (10 – 100 uA) in university-scale facilities dedicated to materials research [24].

In this paper, we present a foundation for the irradiation of materials using intermediate-energy protons in three parts: first, analysis of how protons can emulate the expected response of materials to fission and fusion systems; second, a presentation of the technique’s practical limitations; and third, a demonstration of the ability to measure bulk property changes. The paper is structured as follows: Section 2 describes the fidelity with which protons can represent the recoil energies and damage cascades expected in materials during fission or fusion reactor operation; Section 3 presents the ability of protons to represent the levels of helium and hydrogen production expected in fission and fusion materials; Section 4 examines the relationship between the achievable dose-rates with proton irradiation, the degree of temperature uniformity, and the irradiated sample thickness; Section 5 investigates the radioactive hazard produced by intermediate-energy protons and compares the irradiation and dwell times to neutron irradiation; Section 6 presents the first irradiations and tensile tests of a bulk structural material with intermediate-energy protons, validating the proposed advantages of the technique; Section 7 discusses the results in detail and presents next steps. Together, these pieces show the capability for intermediate-energy protons to enable the rapid design and validation of materials solutions for fusion and advanced fission energy systems.

2. Irradiation fidelity through recoil energy spectra matching

In a collision between an irradiating particle and a primary knock-on atom (pka), the amount of energy transferred can vary over many orders of magnitude, from eV to MeV, leading to drastically different damage cascades and subsequent primary damage production [25]. The inherent difference between charged particle interactions and neutron interactions drives different amounts of energy transfer to pkas. For example, 1 MeV protons in copper have a much lower average recoil energy (500 eV) than 1 MeV neutrons (45 keV). Likewise, the proton recoils are evenly distributed over three orders of magnitude in pka energy (20 eV – 50 keV), while over 95% the neutron recoils fall within a single order of magnitude (5 – 50 keV) [26]. This difference in recoil energies is often used to argue for the benefit of heavy ions, which have a higher average recoil energy than protons (but have an similarly wide distribution in energies). However, in order to make an accurate comparison to true reactor conditions, charged particle pka energies must be compared to a reactor spectrum of neutron energies, rather than neutrons of only a single energy.

2.1. Generating weighted recoil spectra

In order to evaluate the similarity of proton, neutron, and other ion irradiation techniques to realistic conditions, a case study of recoil energies was performed using tungsten as an example of a fusion-relevant material. Recoil energy distributions are calculated for mono-energetic proton and self-ion exposure, along with a fast fission reactor neutron spectrum and a hypothetical fusion reactor neutron spectrum. These recoil spectra are weighted according to a common energy-based weighting and compared graphically.

The initial recoil spectra are each taken from previously published work and given a consistent weighting. Ion-induced recoil energy spectra were produced from the code DART, which is informed by binary collision approximations [27]. The fast reactor recoil energy spectra was taken from the FISPACT Materials Handbooks for fast reactors [15] using the simulation of in-core conditions. Likewise the fusion reactor recoil spectrum was taken from the FISPACT Handbook for fusion reactors [11], representing a fusion first-wall component. Spectra are weighted according to the NRT formula [28], which predicts the number of defects created by each recoil based on the recoil energy. The FISPACT software assumes a tungsten displacement energy of 55eV [29], and this value was also used in all other calculations.

Several weighted recoil spectra for tungsten are plotted in figure 2, both as cumulative distribution functions (CDF) and as probability distribution functions (PDF). The CDF and PDF are two equivalent ways for presenting the spectrum of pka energies that are produced by each irradiating particle; While the CDF is a more common way to present pka energy ranges, the PDF allows more easy comparison of the similarity between irradiation types, which visually represented as the overlap between the two spectra.

In figure 2, we can see that both the fusion neutron spectrum and the fast breeder spectrum lead to pka energy distributions spanning several orders of magnitude, which contrast with the narrower distribution expected from monoenergetic neutrons. Due to the wide spread in neutron recoil energies, we see that intermediate-energy protons have similar recoil spectra to the fusion and fast fission reactor cases. Intermediate-energy protons are able to create recoils fairly uniformly distributed across the range of recoils expected in fission and fusion reactors (up to 100 eV – 300 keV). Lower energy recoils (~1 MeV) are only able to create low energy recoils (up to 10s of keV). Likewise, 10 MeV tungsten self-ions drastically over-produce very high energy recoils (up to MeVs) which are not expected from fusion or fission reactors. From figure 2, we can gauge a rough similarity between the recoil spectrum, but we can not systematically evaluate this similarity over a wide range of ion energies.

2.2. Optimizing the ion energy for recoil similarity

The recoil energy spectrum for a given ion species depends strongly on its incident kinetic energy, as demonstrated by the difference between the 1 MeV and 10 MeV proton curves in figure 2. Thus, an ion irradiation experiment can
be tailored to better match the expected material response in the real world application by carefully selecting the incident ion kinetic energy. In order to make a more general comparison between protons and heavy or self-ions it is beneficial to establish metrics for recoil spectrum similarity that can be compared across many potential irradiation scenarios. The weighted average recoil energy for each irradiation scenario was calculated by:

$$\bar{E}_{pka} = \int_0^\infty E \cdot P(E) \cdot dE$$ \hspace{1cm} (1)

where $\bar{E}_{pka}$ is the weighted average energy, $E$ is the pka energy, and $P$ is the weighted probability distribution function. Along with the weighted average energy, a spectrum range was calculated as the energy bounds within the middle 90% of recoils fall. Both the weighted average and the energy ranges are plotted in figure 3 for the tungsten-fusion case. As with section 2.1, the neutron pka spectra [11, 15] and ion pka spectrum [27] are taken from previously published work and weighted by the NRT formula [28].

Figure 3 demonstrates an intrinsic and limiting trade off in self-ion irradiation as well as the lack of fidelity in reactor irradiations. At low irradiation energies (200 keV), the recoil spectra of tungsten in self-ion irradiation provide a close match to the material response expected for tungsten in the fusion environment; however, the range of tungsten ions at 200 keV is less then 20 nanometers, resulting in challenging irradiation and post-irradiation measurement constraints and complicating the extrapolation of results to larger length scales of interest in engineering applications. To combat this, tungsten self-ion experiments are routinely performed at higher energies, such as 20 MeV [31–34] to provide radiation uniformity over larger sample depths. At such high ion irradiation energies, the weighted average pka energy is more than an order of magnitude higher than in the fusion environment, inducing cascades of many thousands of displacements with high energy self-ion irradiation compared to the fusion application with cascades of tens to hundreds. A similar problem in matching the pka response of tungsten in a fusion environment exists for emulating fission reactors, although in this case the recoil energy spectra is lower by an order of magnitude as shown by the fast breeder reactor case in figure 3). In contrast, proton irradiation with 10 – 30 MeV simultaneously achieves both high fidelity matching to the pka response of tungsten in a fusion environment with uniform irradiation depths over 500 microns, over seven order of magnitude larger than self-ion irradiation. This enables straightforward bulk irradiation of macroscale specimens suitable for direct engineering assessments like tensile tests with high confidence in the fidelity of the material response to a fusion environment.

2.3. Discussion

This case-study demonstrates that intermediate-energy protons can have a high degree of similarity in recoil energies to future reactor conditions. Protons produce recoils with a wide distribution in energy, and the range of that distribution can be tailored by changing the proton energy. Similarly, realistic reactor conditions also produce a wide distribution in recoil energies, with a comparable energy range and mean pka energy to intermediate-energy protons. Other ions, such as self-ions, can also produce tailored recoil spectra, but the desired recoil spectra come from lower ion energies and leading to much more limited range. While self-ion experiments are inherently a compromise between increasing the ion range and maintaining a representative recoil energy spectrum, proton irradiation allows both extended range and recoil similarity to occur simultaneously.

While this case-study focused on tungsten as a fusion plasma-facing material, the methodology is extensible any hypothetical fusion power plant or advanced nuclear reactor component by optimizing the proton energy to achieve fidelity to the neutron spectrum for the new material of interest the material. Therefore, this analysis not only indicates some general trends between proton and self-ion irradiation, it also established a framework for quantitative comparison of recoil similarity between many different irradiation scenarios.
### 3. Proton induced transmutation: fidelity to nuclear environments

In addition to displacing atoms, neutrons from fission and fusion reactors also cause damage by introducing impurity atoms into the irradiated materials. Neutrons are capable of generating nuclear reactions, including \((n,\alpha)\) and \((n,p)\) reactions, which transmute the original atoms into new ones, often including low levels of helium and hydrogen, in the range of atomic parts per million (appm). Appm concentrations of helium are well known to exacerbate radiation damage effects, promoting the formation of voids, causing high temperature helium embrittlement, and weakening grain boundaries at low temperatures [35]. Additionally, hydrogen has been shown to further promote the formation of voids through synergistic interaction with helium [36]. The combined effect of helium and hydrogen on radiation damage is not fully understood, and are the study of ongoing research using highly specialized techniques such as multiple-simultaneous-ion-beam experiments with coincident heavy ions, helium ions, and protons. While these existing techniques allow insight into He and H damage effects, they produce damage that is shallow, nonuniform, and not representative of bulk neutron damage [35].

Intermediate energy protons also possess the ability to generate transmutation products including helium and hydrogen simultaneously with displacement. Common nuclear materials have the potential for nuclear reactions such as \((p,\alpha)\) and \((p,p')\), resulting in the helium and hydrogen generation analogous to neutron reactions. Cross sections for proton induced nuclear reactions vary with proton energy; therefore, there is the potential for the amount of transmutation produced to be controlled by modifying the incident proton energy. In order to evaluate if intermediate-energy protons could serve as a source of radiation damage with the reactor-relevant levels of transmutation, there is a need to compare proton and neutron induced transmutations.

In this section, a comparison of transmutation products is made between fission reactor, fusion reactor, and intermediate-protons. Transmutation calculations normalized across irradiation types by the number of displacements and were performed on metals that are common nuclear materials. A wide range of proton energies were used to evaluate the degree to which proton irradiation can be tailored to match a given neutron environment.

#### 3.1. Predicting proton-induced transmutation

The proton-induced transmutation was calculated using the inventory and activation code FISPACT [29]. In these calculations, the proton irradiation was modeled as mono-energetic protons with a consistent flux of \(6.24 \times 10^{14} \text{cm}^{-2}\text{s}^{-1}\) and irradiation time of 1 day (a representative flux and irradiation time for intermediate-energy proton experiments [24]). Helium and hydrogen levels were extracted at the end of the simulated irradiation, yielding the average amount of transmutation per unit of proton fluence. The damage efficiency (number of DPA per proton fluence) was calculated at each proton energy using the DART program, assuming the same displacement energies as the FISPACT software (55eV for tungsten, 40 eV for copper, nickel, and iron) [29]. Using this transmutation yield and damage efficiency data, the transmutation was normalized to displacements (appm He and H per DPA) each proton energy and element.

The neutron-induced transmutations performed with FIS-
Intermediate-energy proton irradiation

Figure 4: Transmutation calculations demonstrate that intermediate energy protons can generate fission and fusion relevant levels of helium and hydrogen during irradiation.

PACT using example reactor neutron spectrum provided in the FISPACT Handbooks [11, 14, 15]. These simulations were performed up to 1 DPA, and the helium and hydrogen impurity levels were recorded at the end of the simulated irradiation. These simulations were checked against published simulation data in the FISPACT handbooks to ensure their validity.

3.2. Comparison to nuclear environments

In figure 4, the normalized production of helium and hydrogen in materials are compared for three neutron environments (fast breeder reactors (FBR), high flux reactors (HFR) and prototypical fusion power plant first wall (Demo)) and proton irradiation. There are three important takeaways from this figure. First, there are orders of magnitude difference in the production of helium and hydrogen across neutron environments with a fusion power plant typically producing one to two orders of magnitude more than either fission reactor environment due to the presence of 14.1 MeV neutrons from the deuterium-tritium fusion reaction. Material irradiation in fission reactor are insufficient to reproduce the important effect of H and He accumulation found in fusion materials; any high-fidelity irradiation technique must be able to produce H and He over the range required to achieve fusion relevant levels of these transmutation gases.

The second takeaway is that due to the nuclear reaction thresholds that generate H and He in charged particle reactions with the irradiated material - low energy proton irradiation (less than approximately 10 MeV) and self-ion irradiation for materials such as tungsten cannot generate transmutation gases that important to capturing the full material response of fusion materials.

The final and most important takeaway is the capability of intermediate energy protons to produce fusion-relevant quantities of H and He transmutation gases over three to four order of magnitude in concentration simply by tuning the irradiation energy. For each element, the amount of helium or hydrogen produced could be matched to the application with a proton energy of less than 20 MeV. This matches closely with the energy range required to achieve high fidelity in the pka spectrum recoil distribution and irradiation of bulk specimens suitable for direct engineering testing discussed in Section 2. Because the helium and hydrogen generation are coupled, it is difficult to simultaneously match both the helium and hydrogen production to the application. However, proton irradiation does allow, in general, a closer match to a fusion environment than either of the fission reactor environments, which produce much less He and H in each case. Additionally proton irradiation at several energies would produce comparable irradiations with a varied level of He and H production, allowing a controlled study of the impact of helium and hydrogen production. Therefore, intermediate-energy proton irradiation can be tailored to study the transmutation effects expected in fusion and future fission reactors with a much greater range than multiple-beam techniques and much more flexibility than fission irradiation.

4. Predicting proton irradiation constraints: dose rate, sample thickness, and temperature uniformity

Because protons directly heat their samples during irradiation, the dose-rate, sample thickness, and temperature uniformity are inherently coupled for a proton irradiation experiment. While this beam-heating phenomena is well
known, we lack information about the inherent dose-rate limits set by heating. To address this knowledge gap, modeling of interaction of protons with sample materials was performed to understand what experimental conditions are achievable. This modeling captures the deposition of heat into a sample by a 12 MeV proton beam, the conduction of that heat under ideal circumstances, and the amount of damage created by the protons. This establishes bounds on what dose-rates and sample thicknesses are achievable for 12 MeV proton irradiation, as a representative case for all intermediate-energy ion irradiation. A 12 MeV beam was used for this analysis because it is the beam energy provided by the superconducting cyclotron available in the irradiation facilities of the authors [24]; however, the methodology and modeling tools developed for this assessment can be easily applied to any irradiation energy.

4.1. Modeling of heat and damage production by protons

The first step in this analysis was to model the heat deposition by the proton beam into a sample. Range vs. Energy data was extracted from SRIM tables [9] to model the proton’s energy loss through the sample, and was interpolated to create a table of proton energy values as a function of depth (beginning with an incident proton energy of 12 MeV). All energy lost by the beam was assumed to translate to heat production locally in the material, ignoring the small fraction that remains as defects.

With the knowledge of heat production, the heat transfer is then modeled. Heat conduction is assumed to be purely 1-dimensional - the ideal for minimizing temperature differences in a sample - and heat is assumed to conduct along the direction of the incident protons. The increase in temperature is tracked through the sample thickness, using the room temperature thermal conductivity of the material. With these assumptions, the temperature difference from one end of a sample to the other can be calculated by:

\[
\Delta T = T(x_{\text{max}}) - T(0) = \int_0^{x_{\text{max}}} \frac{dT}{dx}(x)dx = \int_0^{x_{\text{max}}} \frac{q(x)}{k} dx
\]

\[
= \frac{\Phi}{k} \int_0^{x_{\text{max}}} (E(x) - E(0)) dx
\]

(2)

where \(\Delta T\) is the total temperature difference across the sample, \(T(x)\) is the temperature at a depth \(x\), \(E(x)\) is the proton energy, \(\Phi\) is the incident flux of protons, \(k\) is the thermal conductivity, and \(q(x)\) is the heat flux. A representative temperature profile is shown in figure 5, demonstrating how \(\Delta T\) is calculated.

With a given \(k\), \(E(x)\), and \(x_{\text{max}}\), Equation 2 yields the maximum flux allowed for a specific temperature variation in a sample. This method was used in this analysis to calculate the maximum flux for a 5 K temperature difference across a range of thicknesses (0 – 400 µm) for four pure metal elements (Fe, Cu, W, Ni). This conservative limit of 5 K was set as an optimistic case for temperature uniformity, knowing that any real experimental system will have greater difference due to imperfect conduction, and as a representative temperature difference that could have a significant impact on defect mobility [citation needed]. Because equation 2 presents a linear relationship between temperature difference and proton flux, these results can be scaled linearly to any other desired temperature limit.

Using TRIM simulations, the maximum allowable flux values were converted to maximum allowable dose-rates. For each material, a TRIM simulation was constructed simulating 12 MeV protons, as shown in figure 5. Each TRIM simulation output the density of vacancies created, per unit of ion-fluence, at each depth location into the sample. This damage profile was converted into an average dose rate per unit flux by equation 3:

\[
\frac{R_D(x_{\text{max}})}{\Phi} = \frac{1}{x_{\text{max}}} \int_0^{x_{\text{max}}} \frac{R_D(x)}{\Phi} dx
\]

\[
= \frac{1}{x_{\text{max}} * \rho_N \Psi} \int_0^{x_{\text{max}}} \rho_V(x) dx
\]

(3)
4.2. Dose-rates and thickness bounds for representative metals

As shown in figure 6, for each example material, 12 MeV proton irradiation is able to access a range of thicknesses greater than 100 µm and dose-rates on the order of 1 DPA/day without causing temperature differences greater than 5 K through the thickness of the sample. The exact shape of the accessible area is dependent on many material properties, including thermal conductivity, displacement threshold energy, and the stopping power of protons in the material (which depends on density, atomic number, etc.). Despite these differences, each material allows irradiation to rates greater than 0.1 DPA/day in relatively thick samples (200 – 300 µm) and irradiation to rates as high as ∼1 DPA/day in thinner samples (∼100 µm). Furthermore, irradiating any of the example materials to dose-rates in excess of 10 DPA/day would require either very thin samples (<50 – 100 µm) and/or large temperature inhomogeneity in the sample (in addition to a ~mA source of intermediate-energy protons). From this analysis, we can see that the range of 0.1 – 1 DPA/day with samples thickness of 100 – 300 µm is readily accessible to proton irradiation without compromising temperature homogeneity.

4.3. Discussion

The results of this case-study of 12 MeV protons presents the basic trade-off between achievable experimental conditions for all intermediate-energy protons. While extended range is desired for easier measurement of bulk properties after irradiation, it comes with an approximately quadratic reduction in the dose-rate achievable (e.g. doubling range decreases doserate to one-fourth). Increasing the incident proton energy is an option to further increase range, but that increased range will further decrease the achievable doserates, which is likely the limiting factor for the speed of experiments. Instead, many proton experiments will benefit from designing for the thinnest samples that can be easily measured, maximizing the doserate and temperature uniformity achievable.

Additionally, this work also presents a novel framework for understanding the limitations of proton (or other light-ion) irradiation under any arbitrary scenario. With modification of the inputs to TRIM and SRIM and to the thermal conductivity, this framework could be adapted to any material, proton energy, and irradiation temperature. As such, it is a flexible method for predicting the fundamental limits of irradiation experiments, due to the inherent characteristics of the ion and materials chosen. Therefore, this framework can help determine the fundamental limitations of intermediate-energy proton irradiation in any future scenario.

5. Predicting and reducing irradiation-induced radioactivity

Both neutron and intermediate-energy proton irradiation experiments can cause radioactivity in irradiated materials and risk to personal and environmental safety. Neutrons and protons (above an isotope-dependent energy threshold ∼ 1-10 MeV) lead to formation of new isotopes through the process of transmutation. The delayed release of radiation through the decay of long-lived radioisotopes poses a prolonged obstacle to the safe evaluation of irradiated samples. Addressing the hazard of radioactive samples often requires long times (months - years) to allow radioisotopes to decay, expensive equipment (e.g. gloveboxes, dosimeters) to protect personnel, and limited examination of the material response (requiring remote handling and limiting time near samples). Mitigating this radioactive hazard is paramount to allowing frequent and cost-effective measurements of radiation damage.

This section compares intermediate-energy proton and reactor activation of irradiated materials in order to understand the capability for protons to enable low-activation, bulk irradiation experiments. Predicting proton and neutron activation of materials is complex, requiring precise knowledge
of the incident radiation energy spectra, energy-dependent cross-sections for each isotope present, and half-lives of each radioisotope produced. As a result of strong variations in each of these quantities, it is challenging to make general claims about the levels of radioactivity across different reactor or proton irradiation scenarios. In order to make a direct and representative comparison of reactor and intermediate-energy proton irradiation, a case-study of induced radioactivity was performed across a set of four engineering materials. In this study, the irradiation-induced activation of each material was calculated for each irradiation scenario: 12 MeV proton, high flux reactor, and fast reactor exposure, each to an equivalent level of exposure. The decay of this activation over time after the irradiation was predicted to quantify both the level of hazard and the potential time-cost across techniques.

5.1. Simulating activity after irradiation

Both neutron and proton simulations were performed using the FISPACT code [29]. These simulations require inputs including an irradiating particle energy spectrum, irradiation flux, irradiation time, and material composition. The simulations output a variety of data relating to transmutation, activation, and radiation exposure in the irradiated material.

Representative values for neutron energy spectra and fluxes were taken from the FISPACT materials handbooks for fast breeder reactors [15] and high flux reactors [14]. Initial simulations output a doserate in DPA/day, which was used to calculate the time of irradiation needed to reach 1 DPA. This irradiation time to 1DPA was used as the time input for further simulations.

Proton spectra and fluxes were set to be representative of a realistic proton irradiation experiment, as is described later in section 6. Proton spectra were calculated assuming a sample thickness of 150um, which is shown in section 4 to enable bulk property measurement while limiting temperature differences due to beam-heating. SRIM range tables were used to determine how much material was exposed to each proton energy as the proton beam degraded from its initial energy (12 MeV) to its exit energy (~3-5 MeV). These input spectrum files can be found in the appendix [to be added].

Proton fluxes were set to 6.24 × 10^{14}cm^{-2}s^{-1} to represent a beam current density of 1μA/mm² (representative of values used in [24]). Dose-rates were calculated from TRIM simulations using the methodology described by equation 3, and using the displacement energies specified in the FISPACT user manual [29]. These dose-rates were then converted into time of irradiation needed to reach 1DPA which was used as an input to FISPACT.

Materials were chosen to be representative of a wide span of engineering-relevant materials for future nuclear applications. Two structural materials - a stainless steel and a nickel alloy - and two functional, high-heat-flux materials - a high-purity tungsten and a precipitate-hardened copper alloy - were selected as a subset of relevant materials. Material compositions were taken from published work on fusion reactor relevant materials: compositions for ITER-grade (IG) stainless steel 316, ITER-grade CuCrZr, and ITER-grade tungsten were each taken from ITER materials handbooks [40], using the maximum allowable impurity levels. The composition for Incoloy 908 was taken from a data handbook published by the Plasma Science and Fusion Center at Massachusetts Institute of Technology [41], using maximum allowable impurity limits. Each of these materials has tightly controlled impurity limits in order to be minimally activating. Material compositions are summarized in table 1.

The FISPACT simulations yielded data representing the radioactivity of irradiated samples while those samples decay after irradiation. Specifically, this analysis extracted the dose to a human 30 cm away from 1 gram of irradiated material, and scaled those results by representative volumes and densities. A one-to-one comparison of neutrons and protons used results scaled by the material density and the volume of an SS3 tensile specimen (33mm³), which a common miniature specimen used in neutron irradiation experiments [42]. A second set of proton results were scaled by the size of the irradiated region in a proton irradiated tensile specimen (1.5mm³), as described in section 6, representing a realistic reduction in volume that can be achieved with proton irradiation.

| Material name       | Major elements | Minor elements | Impurities                        |
|---------------------|---------------|---------------|-----------------------------------|
| Tungsten ITER-grade | W 99.94       |               | C 0.01, O 0.01, N 0.01, Fe 0.01, Ni 0.01, Si 0.01 |
| CuCrZr ITER-grade   | Cu 98.98      | CR 0.75, Zr 0.11 | Nb 0.1, Co 0.05, Ta 0.01         |
| Stainless steel 316* | Fe 64.6, Cr 17.5 | Mo 2.5, Mn 1.8 | C 0.023, N 0.007, Ti 0.15, Si 0.5, Cu 0.3 |
| Incoloy 908         | Ni 49.5, Fe 40.7 | Cr 3.9, Nb 3.0, | Si 0.15, Mn 0.04, C 0.01, Cu 0.01, Mo 0.02, Ta 0.01 |

*other trace elements included, see Appendix

Table 1
Material compositions for the four fusion relevant materials used in the FISPACT activation simulations
5.2. Radioactivity case-study for proton and neutron comparison

The radioactivity curves in figure 7 contain an equal-volume comparison between the two reactor scenarios (HFR and FBR) and the proton scenario, which demonstrate that radioactivity is very material-specific. In the incoloy and copper alloy cases, protons performed similarly to high flux reactors on practical experimental time scales (days to months). In the steel case, protons generated much more radioactivity than the high flux reactors, while in the tungsten case the reverse was true. Similarly, fast reactors allow a large decrease in radioactivity in the copper and tungsten cases, but a much more modest decrease in the steel and Incoloy cases.

Figure 7 also displays the benefit of reducing the proton sample volume. In the dark shading, we see a reduced-volume proton case, which outperforms the full volume HFR radioactivity in every material and across the full range of times scales. This reduced volume case even outperforms the FBR in every material but the copper alloy, where both cases represent relatively low levels of activation.

In order to quantify the potential for time savings in a proton irradiation experiment, irradiation times and cooldown times was extracted from the FISPACT and TRIM simulations, and plotted in figure 8. In this figure, the irradiation time to a fixed dose (1DPA) is plotted along with the cool-down time required to bring a sample below the Nuclear Regulatory Commission definition of a high radiation area (0.1 Rem/hr at 30 cm away from the source) \[39\]. This represents the amount of time needed to irradiate and wait for sample cool-down without regulations requiring that the irradiated materials remain in strictly controlled areas during post-irradiation examination. Additionally, this is the level of radioactivity in which a worker would reach their yearly maximum exposure (5 rem \[43\]) within 50 hours of being 30 cm away from a sample. Therefore, this doserate is a representative value for high radioactivity that presents difficulties during experimentation.

From figure 8 we can see a distinct advantage in experimental time required for proton irradiation compared to reactor irradiation. High flux reactors require extend irradiation times, requiring months to reach single-DPA doses, and creating high levels of radioactivity in samples that require years to decay away. FBRs allow a significant acceleration relative to HFRs, but still require weeks of irradiation to reach 1 DPA and months of cooldown in the steel...
and nickel alloy cases. Protons further reduce the irradiation time to days, and reduced sample volumes shorten the cooldown times across all four materials.

5.3. Discussion

The above activation analysis compares radioactivity across a representative set of materials and irradiation scenarios. From this analysis, we see that there is not an inherent advantage in reduced activity present from the use of intermediate-energy protons. While there can be a distinct advantage in some materials (e.g. tungsten), there can also be a distinct disadvantage in others (e.g. stainless steel), when compared across equal volumes. However, this equal volume comparison presupposes a sample size that has been optimized for use in reactors, not in proton experiments.

When activation is compared using a test geometry that has been customized for a set of proton experiments, we see a nearly universal decrease in activity across all materials. This contrast is especially stark when compared to high flux reactors, a prevalent tool for use in materials irradiation testing that can be accessed in North America [44], Europe [45, 46], and Asia [47]. Fast reactors represent a best case-scenario for reactor irradiation and are a faster, lower-activation alternative to high flux reactors. However fast test reactors are much less common [48, 49], with none currently operating outside of Asia and Russia. Even compared to fast reactors, protons offer a considerable acceleration in radiation damage and - with custom test geometries - a further reduction in activity and required cool-down times. Therefore, proton irradiation with custom test geometries has advantages in reducing the cost, time, and risk associated with radioactivity generated from materials irradiation.

6. Intermediate-energy proton irradiation demonstration

Sections 2 - 5 establish principles by which intermediate-energy proton irradiation can be used as a rapid and flexible tool for bulk, radiation-damage testing. This section provides an initial demonstration of bulk, intermediate-energy proton irradiation and the extraction of irradiated material mechanical properties through tensile testing of a metal relevant for nuclear energy systems: Alloy 718.

6.1. Experimental method for proton irradiation

The experiment comprises the irradiation of Alloy 718, a high strength nickel, with 12 MeV protons followed by tensile testing. Irradiation doses and temperatures were controlled to replicate those used in a similar set of neutron irradiations performed in a high flux reactor [50]. Solution annealed Alloy 718 was chosen for its sensitivity to low levels of radiation damage and the availability of irradiated tensile test data. Solution annealed alloy 718 shows substantial hardening at doses as low as $6 \times 10^{-4}$ DPA with yield strength increases of 100s of MPa [50]. The compositions of the Alloy 718 varied slightly this experiment and the referenced neutron irradiations, as shown in Table 2.

Proton irradiation was performed with 12 MeV protons produced an Ionex ION-12SC cyclotron [23]. Samples were actively water cooled during irradiation to control the temperature; sample temperature was directly monitored by thermocouples mounted to the sample surface. Irradiation temperature was kept between 80°C and 100°C and was controlled by varying the intensity of the proton beam onto the sample. The sample was irradiated to a dose of $3 \times 10^{-4}$ DPA uniformly throughout the bulk of the sample’s tensile test specimen gauge region. Details of the experimental equipment - including the cyclotron, irradiation sample, and in-
Intermediate-energy proton irradiation

|        | Ni  | Fe  | Cr  | Nb  | Mo  | Ti  | Al  | Co  | Mn  | Si  | Cu  | C   | S   | ref |
|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Proton | 52.5| 18.5| 19  | 5.13*| 3.05| 0.9 | 0.5 | 0.18| 0.18| 0.15| 0.04| 0.008|     |     |
| Neutron| bal | 18.3| 18.13| 5.07| 3.0 | 1.1 | 0.54| 0.4 | 0.21| 0.13| 0.05|     |     |     |

*Nb + Ta

Table 2
Material compositions for proton and neutron irradiation experiments

![Stress-Strain Curve](image)

Figure 9: Low dose irradiation of a nickel-alloy highlights the sensitivity of this facility to radiation-induced property changes.

6.2. Bulk property measurement: tensile test

A post-irradiation tensile test of the Alloy 718 specimen was performed to extract the change in strength and ductility resulting from irradiation-induced changes to the material microstructure. These tensile tests allow the extraction of a full stress-strain curve, which includes information such as the yield strength, ultimate tensile strength, ductility, and toughness. Tests were performed at room temperature, using a constant displacement rate, and with a digital-imaging-based strain measurement system. Details on the tensile testing system and technique can be found in [24].

The results of the tensile test are shown in Figure 9, which shows stress-strain curves for two Alloy 718 samples, one pristine and one irradiated to $3 \times 10^{-4}$ DPA. The irradiated samples show expected changes in the stress strain curve indicative of radiation damage induced alteration of the material microstructure. In particular, the yield strength of the material increased by 20 percent, from 424 MPa in the pristine case to 511 MPa in the irradiated case, which quantitatively matches results in previous irradiation studies of Alloy 718 at these dose levels. Importantly, the uncertainty in the yield strength measurement with the specimen geometry and tensile tester used for this experiment is less than 10 MPa [24], well below the measured yield strength change at such low radiation dose. This provides confidence that the resulting yield strength change is a direct result of irradiation and not due to the equipment or uncertainty. This experiment also demonstrates the high sensitivity to material property changes of the technique even at relatively low radiation doses in materials like Alloy 718. We note also that no significant change in ultimate tensile strength and ductility are observed in this sample, consistent with previous studies of irradiated Alloy 718 at such low doses.

An important final result from this experiment is how closely the hardening (e.g. change in yield strength) of the Alloy 718 at $3 \times 10^{-4}$ DPA induced by 12 MeV protons in this experiment qualitatively and quantitatively matches the hardening induced by reactor spectrum neutrons of the same material [50]. Preliminary irradiations with 12 MeV protons to higher doses indicate similarly close changes in other mechanical properties, such as ductility and ultimate tensile strength. This replication of neutron irradiation-induced material property changes with 12 MeV protons suggests that the microstructural changes to the material in both neutron and proton cases are highly similar. More detailed irradiations are now being carried out on a larger selection of materials and to a wider range of total doses. Macroscopic testing such as tensile tests for engineering material properties are being combined with nano- and micro-scope techniques such as indentation and imaging (SEM, TEM) to provide quantitative comparison between the microstructural similarity and evolution of neutron and intermediate energy proton irradiated materials. Those results, and the comparison between neutron and proton irradiated materials, will be presented in an upcoming publication. If close comparison are found, this would start to confirm the capability of proton irradiation - with the beam energy tuned to replicate the neutron-induced material response - to produce material changes with high fidelity to the changes materials would experience in advanced fission and fusion energy neutron spectra, as discussed above in Section 2. The use of intermediate energy proton irradiation would then provide a rapid, high fidelity tool to achieve understanding, down selection, and qualification of materials for advanced nuclear energy systems.
7. Conclusions and impact

In this article, intermediate-energy protons have been identified as a uniquely flexible tool for studying radiation damage for nuclear power technology. Unlike other ion-based techniques, 10 – 30 MeV protons are capable of damaging samples with thicknesses of hundreds of microns, enabling bulk property measurement. The recoil energies produced by these protons are well matched to those of fission and fusion reactor neutrons, ensuring similar radiation damage cascades and primary damage production. The production of helium and hydrogen through transmutation is controllable based on the incident proton energy, and can be matched to any of the wide range of neutron environments. This combination of bulk irradiation, representative recoil energies, and controllable transmutations cannot be found in any other irradiation damage technique.

Intermediate-energy protons are also a fast, low-cost tool for radiation damage testing. With dose-rates that are many times higher than test reactor conditions, protons allow time savings of months or years compared to reactor irradiation. The ability to reduce sample activation leads to similar time savings of months or years compared to reactor irradiation.

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With new accelerator technology, proton experiments can be performed in small laboratories with testing equipment. With new accelerator technology, proton experiments can be performed in small laboratories with testing equipment. With new accelerator technology, proton experiments can be performed in small laboratories with testing equipment.

The ability to reduce sample activation leads to a similar time savings of months or years compared to reactor irradiation. Protons also allow time savings of months or years compared to reactor irradiation.

With both experimental flexibility and the potential for more abundant data, intermediate-energy protons are able to evaluate critical radiation damage effects in a way that is infeasible with other techniques. Protons enable well controlled single-effects studies, such as the ability to determine the impact of varied amounts of helium and hydrogen transmutation on bulk mechanical properties, an important effect for steels and other structural components. Protons also allow bulk mechanical effects to be studied with finer resolution, enabling key phenomena like onset of void swelling and the radiation-induced shift in ductile/brittle transition temperature to be better resolved under simulated reactor conditions. As a very high-range ion, intermediate energy protons create the possibility for a family of bulk, in-situ materials testing techniques, such as in-situ irradiation creep testing, that are not feasible under typical ion or neutron experiments.

Not only does this technique lower the cost and time needed to develop new nuclear systems through faster and cheaper testing of nuclear materials, it also has the potential to dramatically improve the design of these systems. The ability to test more variations of existing materials with finer data can lead to a finer understanding between the trade offs in performance, cost, and lifetime, and subsequently a better optimized design. Once a material is selected, more realistic testing of component lifetimes can have a large impact on the system performance, minimizing down-times and operational costs. As the discovery of new nuclear materials continues, the ability to rapidly and reliably test many combinations of composition, processing, and irradiation conditions has a huge potential to speed up the development and deployment of these new, high-performance materials. Therefore, the wide-spread deployment of intermediate-energy protons as a radiation damage testing technique could have a profound impact on the materials selections and overall design of fission and fusion power technology.

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Data Availability

The raw data and processed data required to reproduce these findings are available to download from http://dx.doi.org/10.17632/ndj55thg39.1.

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