ARTHEMIS

Assessment of Radiation damage and Transmutation Helium Effects on structural reactor materials via MIcromechanical testing and advanced analysis methodS

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Irradiation strategies using multi-ions

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Table of Contents

1. Introduction....3
2. Methodology....4
3. Experimental design....5
3.1. Multi Energy Sequential Irradiation Experiment Design for Thick Irradiated Layers ...6
3.2. Multi Ion Sequential Irradiation Experiment Design for Layers for coolant-material interaction studies....7
4. Ion irradiation 10
4.1. Instrumentation....10
4.2. Experiments....12
5. Summary....13
6. References 13

1. Introduction

Structural materials in the advanced nuclear systems - both fission and fusion - must operate under extreme conditions, including radiation and corrosive environments, and in many cases high production rates of transmutation helium. Helium formed within reactor internals can significantly degrade material performance, leading to hardening, embrittlement and ultimately a loss of structural integrity of the component. Investigating this phenomenon requires either powerful sources of fast neutrons or high-fluence ion-irradiation facilities capable of introducing sufficient amounts of helium into materials.

In most ion-irradiation studies, helium is implanted at a single energy, producing a thin, graded near surface modified layer. While useful for fundamental studies, this approach does not provide the homogeneous helium distribution or controlled helium-to-displacements-per-atom (He/DPA) ratios required for engineering-relevant assessments. Moreover, investigations on bulk specimens with relatively thick irradiated regions suitable for micromechanical testing remain scarce. This gap can be addressed by high-fluence, multi-energy ion irradiations, which allow modification of material volumes extending to several micrometers in depth, and enable the design of tailored helium depth profiles.

This report presents ion-irradiation strategies based on single-beam implantations at medium and high-energy ion accelerators, designed to generate quasi-uniform displacement damage across extended layer thickness with targeted He/DPA ratio, and helium-implanted, damaged layers providing suitable specimens for material–coolant interactions studies. The report details the technical aspects and implantation parameters of three specific irradiation experiments, supported by SRIM simulations of helium implantation profiles.

2. Methodology

Studies addressing the effect of transmutation helium require homogeneous helium concentration distribution within the irradiated volume. A single-step helium irradiation, performed at one ion energy, results in non-homogenous distribution within the irradiated depth, that can be often approximated by a Gaussian distribution. To achieve a homogenous distribution, box-like profile, we adopted a multi-step ion-irradiation approach. This means performing sequential helium implantation at different energies to create a constant concentration depth profile [1]. The final concentration profile is obtained by superimposing dopant box-profiles from a sequence of ion implantation steps with decreasing ion energies.

The single-energy implantation depth profiles were calculated using SRIM 2013 [2]. The ion ranges were fitted by bi-Gaussian functions to achieve the best fitting results (Figure 1). Since the materials investigated within this project are iron based alloys, SRIM calculations for ion ranges and displacement damage profiles were performed with 56Fe as target material. The number of energies employed is a free parameter and must be selected reasonably with respect to the accelerator parameters.

To reach the wanted level of concentration, the single profile components need to be optimized. The array of depth profiles serves as a basis function, and the ion fluence at individual energies is calculated of the following minimization problem:

$$\min_{u_1 \ldots n} \left\| \sum_{i=1}^{n} C_{He_i}(x) u_i - W(x) \right\| subject\ to \sum_{i=1}^{n} u_i = \int W(x) dx, \qquad (1)$$

where $C_{He_i}(x)$ represents the helium concentration profile for the i-th step, $W(x)$ denotes the desired concentration profile, u_i are the ion fluences at individual energies, and n is the number of implantation steps/energies. The resulting implantation profile is given by:

$$\hat{W}(x) = \sum_{i=1}^{n} C_{He_i}(x)\hat{u}_i \tag{2}$$

where, $\hat{u}_i$ are the calculated optimal fluences at individual energies.

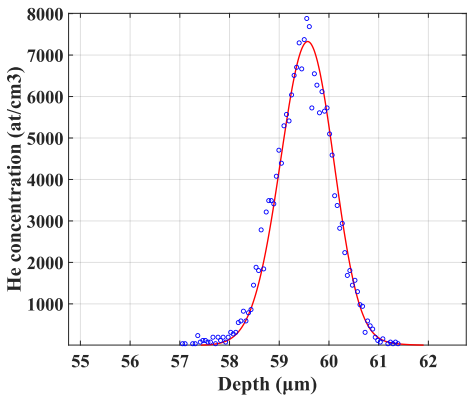


Figure 1. Fitting of individual He concentration profiles

Due to the small mass of helium ions, the displacement damage rate during implantation remains low. When higher damage level is required, multi-ion irradiation is adopted, where helium implantation is combined with heavy-ion irradiation. For the iron-based alloys we chose Fe ion to achieve the required level of DPA in the implanted region. Achieving equivalent ion ranges with heavier ions demands substantially higher energies compared to helium. Usually, for a given accelerator the maximum ion range for Fe is an order of magnitude lower than that of He ions, which limits the thickness of the implanted layers thickness achievable with this approach.

3. Experimental design

3.1. Multi Energy Sequential Irradiation Experiment Design for Thick Irradiated Layers

The aim was to design and perform unique single-beam, multi-step helium implantation experiment capable to producing quasi-uniform displacement damage and helium concentration over increased layer thickness of ≥60 μm. We aimed to reach at least 1000 appm helium concentration, as this level is expected to result in the formation of helium bubbles of a size detectable and quantifiable via various experimental techniques [3].

The availability of such thick irradiated layer will allow us to assess the mechanical properties applying micromechanical test methods such as micropillar compression. This thickness is sufficient for the use of conventional radioisotope-based positron sources in positron annihilation spectroscopy (PAS), which is an important characterization method used in the post-irradiation examination (PIE) of ion-irradiated materials.

To achieve the desired implanted layer properties, the final irradiation profile was assembled of 34 individual single ion profiles. The implantation energies started at 17 MeV down to 500 keV, in 500 keV decrements (Figure 2). This multi-energy design yields an approximately 65μm-thick irradiated layer containing 1000 appm helium quasi-homogeneously distributed in the irradiated volume, Figure 3.

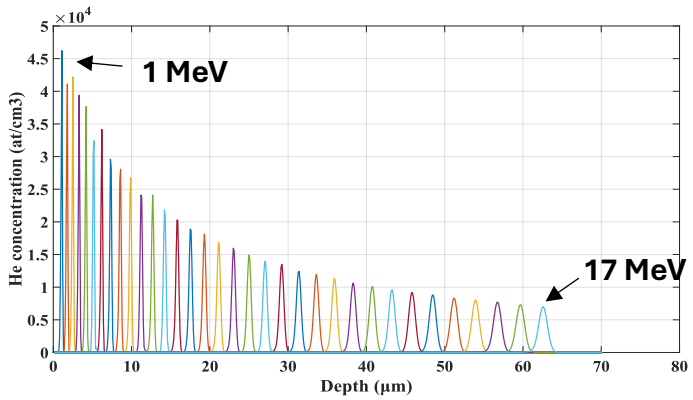


Figure 2. Helium concentration profile set of the 34-step irradiation

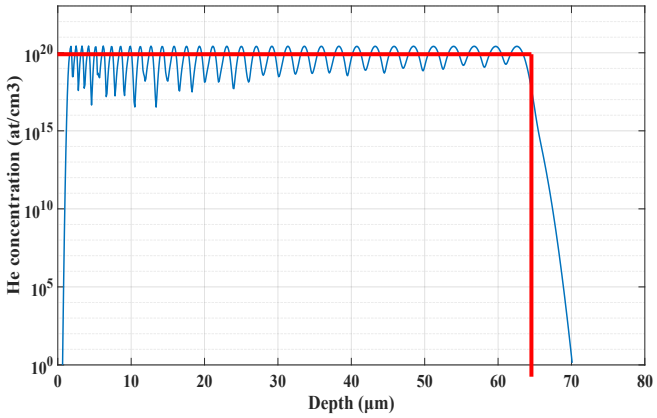


Figure 3. Projected helium concentration profile (corresponding to 1000 appm He).

3.2. Multi Ion Sequential Irradiation Experiment Design for Layers for coolant-material interaction studies

To investigate the synergetic effect of radiation damage, transmutational helium and environmental exposure, a dedicated ion-irradiation experiment was designed, to simulate end of life radiation conditions in an approximately 1µm thick subsurface region of the selected specimens. As mentioned in section 2, a multi-ion irradiation strategy was chosen to achieve that. The irradiation parameters were chosen to reflect the design specifications of the supercritical water-cooled small modular reactor (SCW-SMR) concept developed within the ECC-SMART Euratom project. Two helium concentration levels were targeted, 25 and 50 appm, corresponding to a radiation damage level of 10 DPA in both cases. The multi-step He irradiation was designed to yield a concentration profile with constant ratio of He/DPA, 2.5 appm/DPA and 5appm/DPA respectively.

The calculations were performed using SRIM 2013 (Stopping and Range of Ions in Matter), a Monte Carlo simulation package for simulating ion implantation. The DPA profile was calculated using the NRT (Norgett-Robinson-Torrens) method. For these calculations, iron (Fe) was used as both the incident ion and the target material to establish the baseline dpa profile in the iron substrate. The ion energy was set to 2.3 MeV, the displacement energy was set to 40 eV, binding energy to 0eV and surface energy also to 0 eV. The resulting DPA profile is introduced in Figure 4.

The helium implantation profile was designed in a way that the calculated concentration distributions copy the shape of the simulated DPA profile. The single energy helium concentration profiles were fitted with bi-Gaussian function to simplify further processing. A multi-energy ion implantation approach was used with nine energies (Table 1) and optimized to achieve the desired shape and target He/DPA ratio in the depth region spanning from 300 nm to 700 nm. The resulting helium concentration distribution is shown in Figure 5.

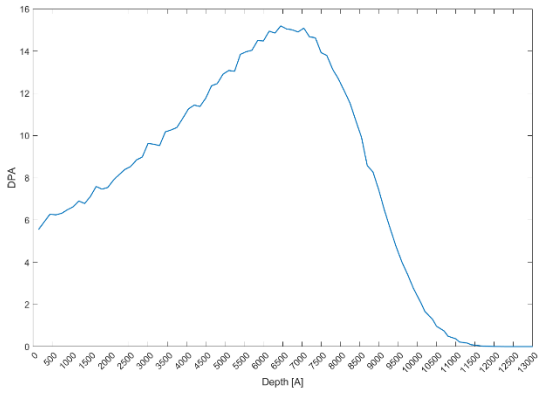


Figure 4. The simulated damage profile for single step Fe ion irradiation.

Table 1. He ion energies and corresponding fluences.

Energy [keV]	**Fluence [ions/cm^{-2}]**
15	1.16E+13
30	1.66E+13
50	2.66E+13
75	2.22E+13
100	2.68E+13
150	6.14E+12
200	8.50E+12
250	9.50E+12
320	5.83E+13

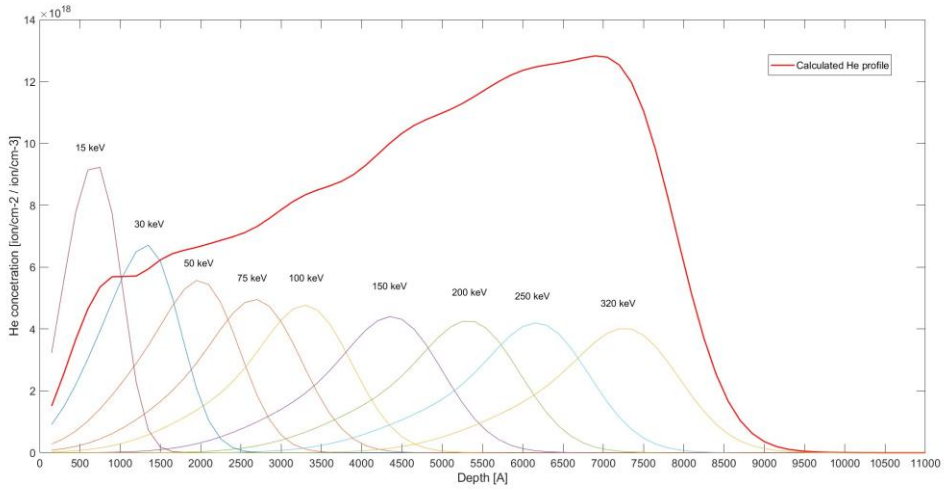


Figure 5. The resulting helium concentration profile assembled from 9 implantation steps.

Comparison of the calculated and target profiles reveals excellent agreement throughout the region of interest (Figure 6. Calculated vs. target He concentration profile.Figure 6). The maximum deviation from the target profile was less than 3%, Figure 7.

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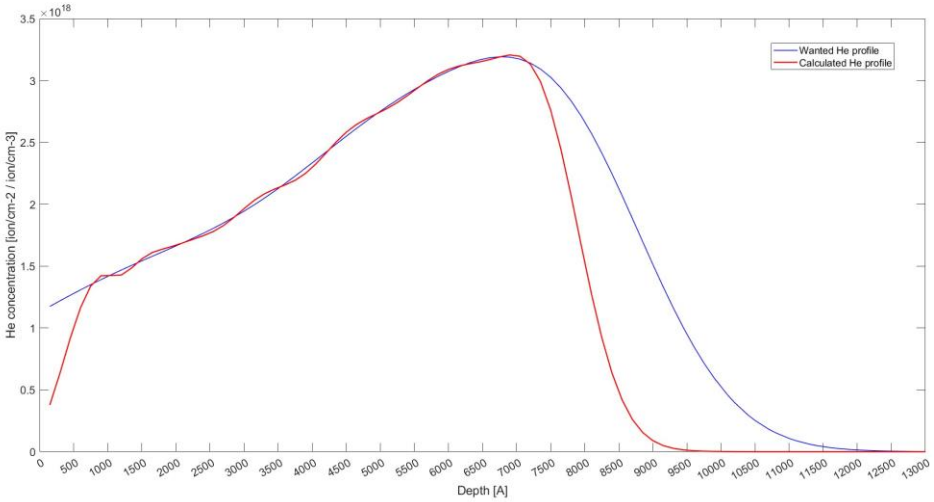


Figure 6. Calculated vs. target He concentration profile.

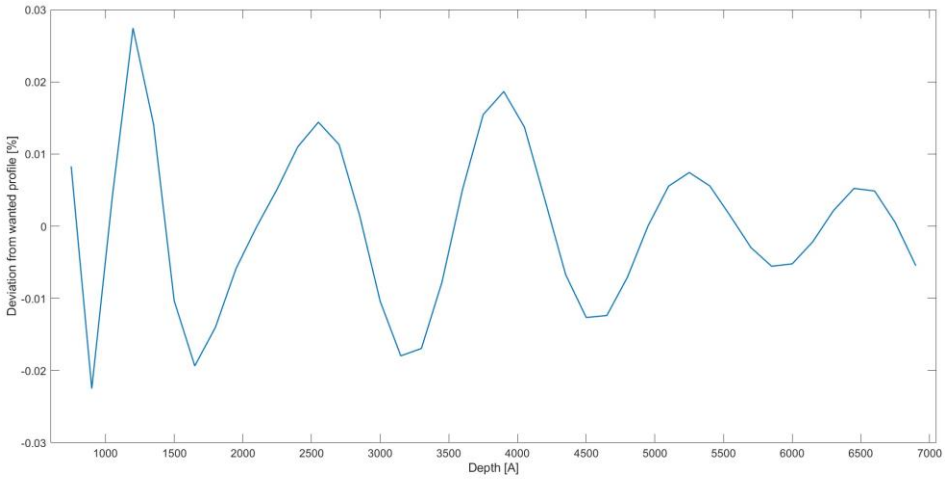


Figure 7. Deviation analysis showing less than 3% maximum fluctuation from target profile.

4. Ion irradiation

4.1. Instrumentation

The ion irradiation and implantation experiments at ATRI are facilitated by two advanced ion accelerators: the 6 MV Tandetron tandem ion accelerator and the 500 kV ion implanter.

The 500 kV High Voltage Engineering accelerator (Figure 8) is equipped with experimental end-station for ion implantation. The sample holder can accommodate samples up to Ø100 mm, and have water cooling and heating capability up to 1000 °C. The available ions range from H to U (excluding synthetic radionuclides and enriched source material), beam currents Up to 2 mA (gaseous elements), hundreds of µA for solid elements.

The 6 MV tandem accelerator serves to perform high-fluence irradiation experiments (Figure 9). It is equipped with HVEE 358 Duoplasmatron ion source with modified extraction optics and a new Na Charge-exchange canal (CEC) designed to deliver He beam currents up to 8 µA. This source is complemented by a HVEE 860 Cs sputtering ion source for heavy ion

beams. The available ions range from H to U (excluding synthetic radionuclides and enriched source material as well as noble gases heavier than Helium), Ion energies (with reasonable currents) from 600 keV to 100 MeV.

The implantation chamber, Figure 10, enables ion-irradiation of substrates with sizes up to Ø100 mm at room temperature and sample cooling down to LN2 temperatures. The heater with a Ø40 mm sample holder enables heating up to 1000 °C. The usual experimental setup for high-fluence experiments is a water-cooled Ø40 mm sample holder where the beam is rastered over an area of 36 cm^2.

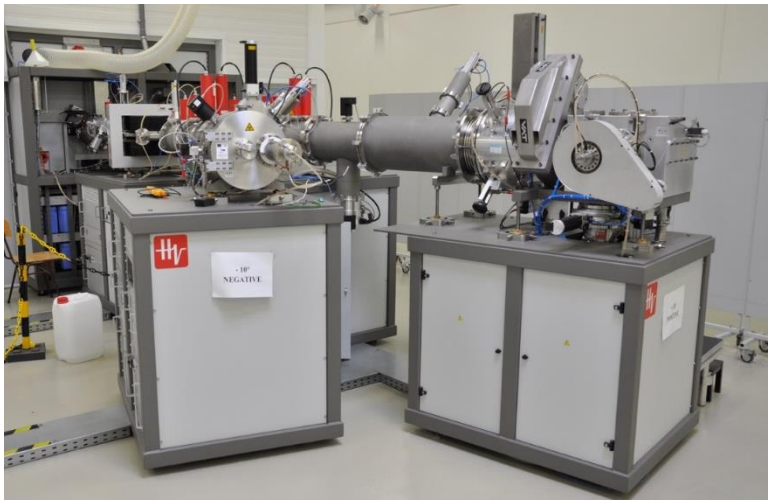


Figure 8. The 500kV ion implanter.

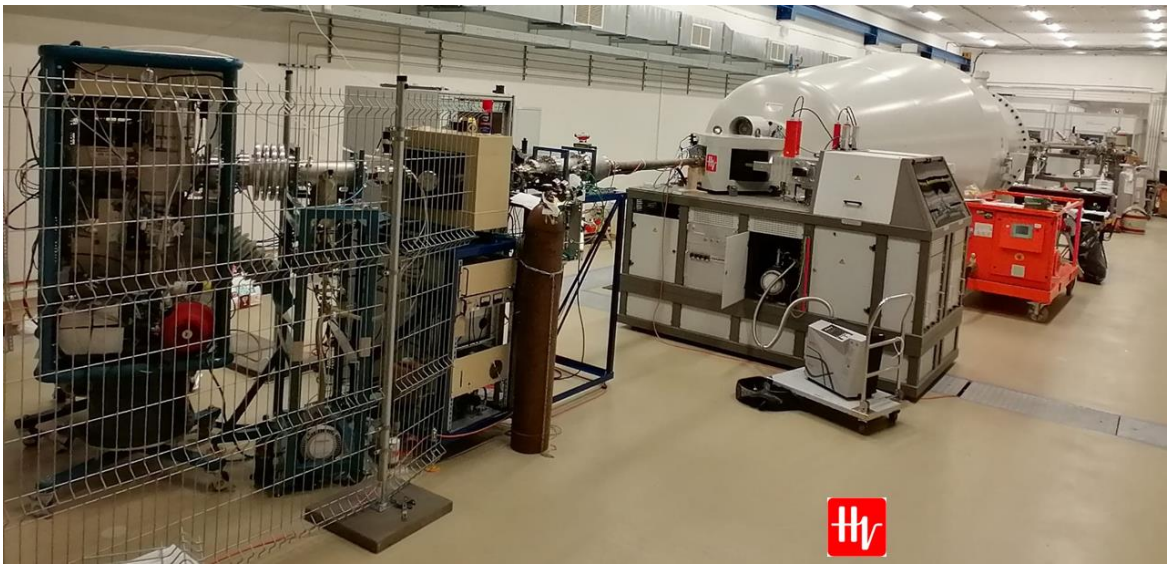


Figure 9. The 6 MV Tandem accelerator with ion sources.

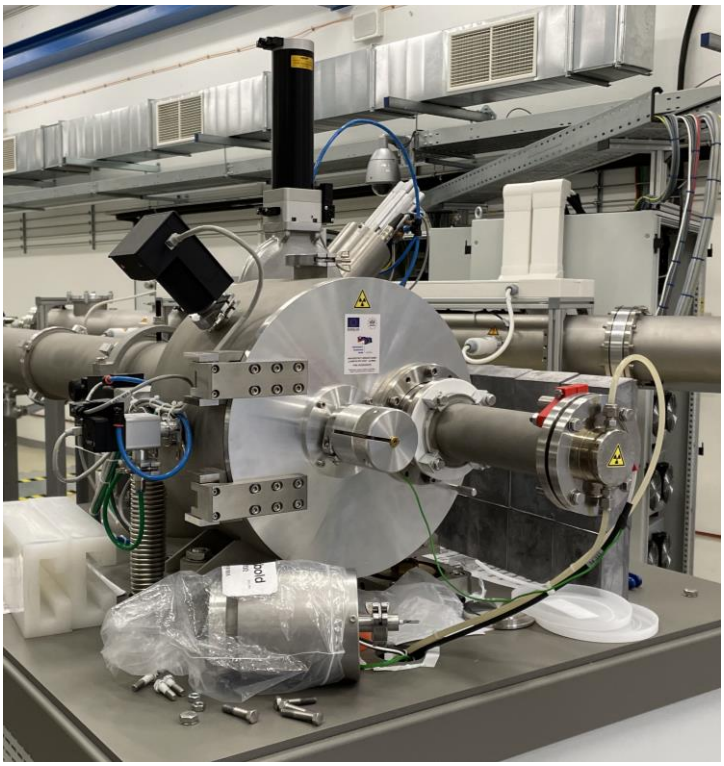


Figure 10. The irradiation end station on the 6 MV Tandem accelerator beamline.

4.2. Experiments

Specimens of Eurofer97 and 800H alloys, prepared in the form of 10x10x0.5 mm plate, were helium implanted based on the multi-energy design described in section 3.1 using the 6 MV Tandetron accelerator. The total implantation time was 1116 hours. During irradiation, the sample holder was water-cooled to keep room temperature and avoid unwanted thermal effects. The experiment achieved a helium concentration of 984 appm/cm^3 at a total fluence of 5.387 x 10^{17} ions/cm^2. The resulting displacement damage across the irradiated region was 0.116 dpa.

In parallel, corrosion coupons, with a size of 10x15x2 mm^3, fabricated from 800H alloy were implanted with helium at room temperature, using the parameters calculated in section 3.2. The experiments were performed with the 500 kV ion implanter, producing two sets of samples: one implanted with 25 appm He and the other with 50 appm He. Subsequently, the helium implanted specimens underwent iron ion irradiation at 500 °C using the 6 MV

Tandetron at an ion energy of 2.3 MeV, in order to introduce displacement damage consistent with reactor-relevant conditions.

5. Summary

This report has presented the design and execution of multi-ion and multi-energy irradiation strategies developed within the ARTHEMIS project. The methodologies were tailored to address key challenges in replicating reactor-relevant irradiation conditions, particularly the incorporation of transmutational helium into extended material volumes with controlled He/DPA ratios.

Two experimental designs were implemented. The first focused on producing thick irradiated layers (~65 µm) containing ~1000 appm helium, enabling micromechanical testing and positron annihilation spectroscopy. The second design targeted thin subsurface layers (~1 µm) with controlled helium concentrations (25 and 50 appm) and 10 DPA, optimized for subsequent studies of coolant–material interactions.

Irradiation experiments were successfully performed on Eurofer97 and 800H alloys using both the 6 MV Tandetron and the 500 kV ion implanter. The results demonstrated that the chosen strategies reliably achieved the required helium concentrations and damage distributions, with close agreement between designed and realized implantation profiles.

The irradiated specimens generated here will serve as the basis for detailed microstructural characterization, micromechanical testing, and environmental exposure studies, directly contributing to the ARTHEMIS project objectives of assessing transmutational helium effect on mechanical properties and synergistic effects relevant to advanced nuclear systems.

6. References

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