Irradiation-Tolerant Refractory High-Entropy Alloys

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Along with the globalization of environmental problems and the rapid development of the field of nuclear technologies, the severe irradiation damage of materials has become a big issue, restricting the development of advanced nuclear reactor systems. Refractory high-entropy alloys (RHEAs) have the characteristics of a complex composition, a short-range order, and lattice distortion and possess a high phase stability, outstanding mechanical properties, and excellent irradiation resistance at elevated temperatures; thus, they are expected to be promising candidates for advanced nuclear reactors.

Keywords: refractory high-entropy alloys ; irradiation damage ; nuclear power

1. Introduction

The environmental problems caused by traditional fossil fuels are increasingly prominent, and the development and utilization of clean energy have become a global issue. Nuclear energy has many advantages, such as a high energy density, great power generation efficiency, low comprehensive cost, continuous power supply, and small regional climate impact, but the inherent safety of nuclear reactors is a key issue limiting its development [1]. The properties of structural/cladding materials are fundamental in the inherent safety; however, they are affected by long-term irradiation of high-temperature and high-energy neutrons and fission products, which usually causes thermal stress expansion and cracks, in addition to radiation hardening and embrittlement [2][3][4][5], radiation swelling [6], phase transformations [7][8], helium effect behavior [9], etc.

Due to the exposure of materials to high-energy radiation, changes at the atomic level in the materials cause embrittlement. For instance, radiation-induced changes in the crystal structure create additional obstacles to dislocation movement, making the material more brittle. They may also lead to the nucleation and growth of precipitate phases within the material, which serve as initiation points for cracks and significantly affect the ductility and strength of the material. Elements like helium, produced by radiation reactions, tend to accumulate preferentially at these grain boundaries, leading to intergranular cracking and thereby increasing the brittleness of the material. Helium also forms bubbles within the lattice, causing swelling and further embrittlement. Temperature has a significant impact on materials as well. At lower temperatures, defect mobility is restricted, resulting in higher concentrations of point defects and dislocation loops. As the temperature increases, some defects may annihilate each other. However, higher temperatures can also accelerate diffusion-driven processes, which can lead to precipitation and grain boundary segregation.

High-entropy alloys (HEAs) have attracted increasing attention [10] due to their high entropy effect, lattice distortion effect, sluggish diffusion, and cocktail effect [11]. These effects make it difficult for HEAs to accumulate defects in irradiation environments and provide new possibilities for advanced nuclear reactors. Radiation-tolerant 3D transition group HEA systems with a face-centered cubic (FCC) structure have been widely studied in recent years. To compare the effects of different elements and components on the irradiation performance, Lu and Jin et al. [12] started their research on pure Ni in an FCC system and gradually added components from binary NiCo and NiFe. Later three- to five-element alloys include CoCrNi [13], FeCoNi, CoCrFeNi [14], FeMnCoNi, and CoCrFeMnNi [15]. As the composition complexity of the alloy increases, it is found that its irradiation resistance also improves because the high entropy leads to a change in the interstitial atom–vacancy migration energy barrier, resulting in less lattice damage. In particular, the five-element alloy NiCoFeCrMn has excellent mechanical properties and its swelling resistance is 40 times higher than nickel, but it might not be ideal for nuclear applications due to the presence of Co, which can become radioactive 60Co upon neutron absorption. Wang et al. [16] compared the approximate component alloys (Ni, NiFe, NiCo, NiFeCr, NiFeCo) and found that with the increase in the number of elements from one to three, the size of helium bubbles tended to decrease, except for Cr ternary alloys. It was found that Cr can exchange with vacancies to cause vacancy accumulation and promote the growth of helium bubbles. However, research on helium embrittlement and the swelling resistance of FCC alloys containing nickel shows the opposite, because Ni has a high neutron activation and an adverse effect on the hydrogen absorption of materials, which make the alloy susceptible to transmutation under irradiation. To sum up, it was found in previous studies that many elements in the 3D transition group possess high neutron absorption and activation, which are not ideal for nuclear applications. In addition, HEAs with a body-centered cubic (BCC) structure have begun to be explored owing to their higher resistance to helium embrittlement and swelling [5][17][18][19]. Therefore, new HEA systems with a BCC structure have recently attracted attention.

2. Composition Design and Preparation of RHEAs

The composition of RHEAs is notably diverse, which consequently imparts a variety of effects on material properties. This diversity adds complexity to material design, as selecting suitable ratios and combinations from myriad possibilities is a key focus in current alloy design. Two primary challenges arise in the design of these alloys: the selection of proper elements and the appropriate proportions of these elements. Leveraging the cocktail effect, one of the four core effects of HEAs, certain elements can be chosen based on their intrinsic properties to achieve specific performance goals.

In terms of designing RHEAs with a high radiation resistance, neutron absorption and activation should be considered as a priority from the outset. A low neutron absorption cross-section is a crucial requirement for radiation-resistant alloys, as it enhances the neutron economy of nuclear reactors.

Following the selection of elements for RHEAs, it becomes imperative to determine the proportion of each element. This determination can be guided by empirical methods, computational simulations, or high-throughput experimental approaches. Thermodynamic considerations such as configurational entropy, mixing enthalpy, melting point, atomic size difference, and valence electron concentration offer a framework to establish a reasonable range for these proportions. The use of phase diagram calculations further elucidates the impact of temperature on phase composition, facilitating the assessment of alloy phase stability. Additionally, employing first principles calculations [20][21] from a thermodynamic perspective enables a deeper analysis of the alloy. This approach yields critical data, including entropy, enthalpy, and Gibbs free energy, which are essential for comprehending the thermodynamic properties and potential performance of the alloy. Such a comprehensive analysis is integral to optimizing the design of HEAs for specific applications, particularly in demanding environments where material stability and performance are crucial.

The fabrication of RHEAs can be accomplished through various advanced techniques, such as magnetron sputtering [8][22] and laser metal deposition (LMD) [23][24]. However, to produce bulk materials, vacuum arc melting [25] is the most prevalent method. This technique is widely favored due to its efficiency in producing high-quality, homogeneous alloys. A smaller proportion of RHEA production utilizes alternative methods like levitation melting [26] and powder metallurgy [27]. While a variety of techniques is available for the preparation of RHEAs, the choice of method largely depends on the specific requirements of the alloy being produced, including its composition, desired properties, and intended application.

3. Irradiation Resistance of RHEAs

3.1. Irradiation Hardening and Embrittlement

In nuclear applications, materials are often exposed to extreme radiation, which can lead to irradiation hardening: an increase in material hardness and brittleness due to the formation of defects like dislocation loops and voids in the crystal lattice. This phenomenon can severely impact the performance and safety of nuclear reactors [28].

Kareer et al. conducted ion irradiation experiments using 2 MeV V^+ ions on four different alloys at 500 °C, with an irradiation fluence of 2.26×10^{15} ions/cm^2. Observations were made on pure V and the high-entropy alloys TiVNbTa, TiVZrTa, and TiVCrTa. It was found that pure V exhibits the most significant change in hardness, approximately 37%, prior to and following irradiation. In contrast, the three RHEAs showed relatively minor changes in hardness, with TiVNbTa exhibiting a hardening rate of about 6%. Notably, the low-activation TiVZrTa and TiVCrTa RHEAs demonstrated negligible hardening rates.

Chang et al. [19] explored the irradiation resistance of the HfNbTaTiZr RHEAs. The experiment involved subjecting samples of the HfNbTaTiZr alloy to 300 keV Ni^+ ion bombardment at a fluence of 1.5×10^{16} cm^{-2} at 100 °C, with a damage level of over 30 dpa. The researchers discovered that irradiation-induced swelling is remarkably suppressed in the HfNbTaTiZr alloy. The nanoindentation hardness after irradiation is almost unchanged, indicating a high resistance to hardening due to irradiation. This suggests that the material experiences extensive lattice distortion and has altered diffusion kinetics under irradiation. The lattice distortion and diffusion kinetics of RHEAs like HfNbTaTiZr endow them with superior radiation tolerance. The results reveal that the swelling and hardening effects in HfNbTaTiZr RHEAs are significantly suppressed compared to those in conventional nuclear materials.

3.2. Irradiation-Induced Phase Transformations of RHEAs

The phenomenon of the segregation of alloy elements caused by irradiation was firstly postulated by L.E. Anthony in 1972 [29]. At Argonne National Laboratory, the segregation effects of principal and trace elements within the matrix of both binary and ternary alloy systems were studied using techniques like Auger electron spectroscopy to reveal the complex changes in materials exposed to radiation [30]. It was found that the segregation of elements can affect the mechanical properties of materials. Under irradiation, the movement of vacancies and interstitial atoms in the material promotes diffusion, leading to element segregation and precipitation, resulting in enrichment and depletion of elements. The movement of point defects promotes the movement of solute atoms. When the radius of body atoms is larger than that of solute atoms, solute are easily combine with collective atoms to form a positive binding energy; these atoms move to defect traps such as grain boundaries, so solute atoms precipitate at the grain boundaries under irradiation conditions. On the contrary, when the radius of the matrix atom is smaller than that of the solute atom, the solute atom easily combines with the collective atom to form a negative binding energy. The atoms move away from the boundary, so the phenomenon of solute atom dilution occurs at the grain boundary.

Pu et al. [8] discuss the mechanisms of irradiation-induced crystallization in amorphous TaTiWVCr RHEAs and W films when exposed to 60 keV He^+ ion irradiation. Both materials were irradiated using a 60 keV He ion beam platform, with fluences ranging from 1×10^{16} cm^{-2} to 2×10^{17} cm^{-2}. Defect accumulation increases the free energy and triggers crystallization when a critical concentration is reached. This process is propelled by the thermal-spike effect, where energy transfer from hot electrons to the cooler lattice causes short-range diffusion and a rise in the lattice temperature. Grain growth is further aided by interactions at the grain boundaries (GBs) between amorphous and nano-crystalline areas, reducing the GB's curvature. He^+ ion irradiation introduces vacancies and interstitial defects, enhancing atomic mobility and rearrangement and thus forming nanometer-sized grains.

The NbZrTi series of RHEAs has excellent phase stability and high mechanical properties at elevated temperatures [31], such as NbZrTiHf [32][33], NbZrTiTaHf [19][34][35], NbZrTiV [36], NbZrTiMoV [37], etc. Their lattice distortion and composition complexity have obvious inhibition effects on heavy ion irradiation [38].

3.3. Irradiation Effect of Helium

The nuclear reaction between neutrons and materials can produce hydrogen and helium, and the helium generated by transmutation is not easily diffused in the material because of its low solubility. Hydrogen is easily diffused out of the material under high-temperature conditions; hydrogen and helium in the alloy further accelerate the diffusion of atoms in the material under irradiation with the capture and diffusion among crystal defects, such as vacancy point defects, dislocation rings, and interstitial atomic clusters. This results in phase precipitation and element segregation in the material, making the material undergo hydrogen embrittlement or helium embrittlement [39]. The formation of defect groups through the combination and decomposition of defects leads to radiation swelling of the material. Defects such as dislocation rings lead to hardening of the material. When the irradiation temperature is greater than 0.5 Tm, helium ions generated by transmutation reactions in the nuclear material form helium bubbles or cavities under the action of stress, which causes swelling inside the material, resulting in further improvements in the brittleness of the material [40]. High-temperature helium embrittlement can be used as one of the reference standards to determine the highest temperature of structural materials in a neutron irradiation environment.

Cui et al. [18] studied the irradiation behaviors of pure W and W-rich RHEAs (RHEA-W). The RHEA-W alloys consist of ultrafine grains with a body-centered cubic (BCC) structure. They are composed of W-based and Ti-rich grains with an ultrafine grain size, which is significantly finer than that of pure W. These alloys exhibit excellent surface stability under He irradiation. With an irradiation fluence of 0.9×10^{25} $ions/m^2$, the surface of the RHEA-W alloys remains nearly flat. The impressive stability of these alloys under He^+ irradiation is attributed to their self-healing mechanism. The high-entropy matrix undergoes microstructural rearrangement to fill in damaged areas, atom diffusion to repair defects, phase transformations that mitigate damage, and potential chemical reactions.

4. Summary

The introduction of refractory elements into BCC-structured HEAs can further improve their high-temperature mechanical properties. Although stable BCC-structured HEAs are resistant to irradiation hardening, their phase stability under irradiation conditions remains a key issue. The second phase precipitated by phase instability can serve as the recombination core of annihilation, thus inhibiting the formation and growth of helium bubbles. An interface between the precipitated phase and the matrix, especially the substructure interface, can lead to the formation, accumulation, and nucleation of lattice defects, thus inducing hardening embrittlement and other ion-induced damage, and its effects need to be further studied. RHEAs have been considered for use in irradiation conditions due to their high melting points and potentially superior radiation damage resistance. However, several challenges arise when they are exposed to such harsh environments:

- Radiation-induced phase segregation and precipitation: RHEAs may experience changes in the microstructure due to radiation, including segregation of elements and precipitation of new phases, which can change their mechanical properties and lead to embrittlement or reduced strength.

- Swelling and void formation: exposure to irradiation can lead to the formation of voids and swelling in the material, which can damage the integrity of the material's structure and lead to premature failure of the alloy under mechanical stress.

- Helium embrittlement: Transmutation reactions within nuclear reactors can lead to the formation of helium or helium bubbles within the alloy. These helium bubbles create pressure within the material, leading to embrittlement and cracking.

- Long-term behavior: The long-term behavior of RHEAs under irradiation is not fully understood, making it difficult to predict their performance over the lifespan of a nuclear reactor. This uncertainty can be a significant barrier to their adoption in safety-critical applications.

- Irradiation-induced changes in other properties: Irradiation can lead to variation in the microstructure of RHEAs, thus resulting in changes in other properties, such as damping, armoring, corrosion and oxidation resistance, thermal expansion, etc. On the one hand, the study of the variation in these properties is a necessary part of the comprehensive evaluation of RHEAs under irradiation conditions; on the other hand, it can also broaden the range and possibility of their applications. However, research in these fields has rarely been reported.

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