

論文の内容の要旨

論文題目 Role of Si addition in determining fine-scale defect formation
condition and mechanism in reactor structural material
(炉内構造材の照射損傷機構に対するケイ素添加の影響)

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Stainless steel is used in the core of light water reactors as structural material. Due to the high neutron fluence, high temperature, stress load and corrosive water chemistry in reactors, the stainless steel components may crack during their service time in reactors. Therefore, the ageing management of stainless steel components in light water reactors is an important issue to ensure reactor safety. Currently, to evaluate the condition of stainless steel components in service, the Japan and U.S. ageing management standards mainly rely on mechanical databases of cracking. Threshold curves of temperature and cumulative dose are drawn based on the cracking databases, and the stainless steel components that are working in conditions exceeding these thresholds are supposed to be susceptible to cracking.

However, different stainless steel components have different working conditions, which the cracking databases cannot fully cover. Also, since the setting up of cracking databases are limited to the current understanding of cracking mechanisms, the uncertainties brought up by possible unknown factors or mechanisms could be large. Therefore, besides the cracking databases, continuous efforts should also be made to trace back to the microstructure evolution process in irradiated stainless steel, which is the origin of the mechanical degradation.

The main objective of this research is to investigate the formation mechanisms of black dots, dislocation loops and γ' precipitates. The interactions between these defects are completed during microstructure evolution process. Therefore, irradiation condition is controlled so that the formation of black dots and Frank loops could be isolated, and their formation mechanisms could be discussed respectively by tuning the Si content of stainless steel. At higher temperatures, γ' precipitates are introduced. The formation

mechanisms of γ' precipitates are analyzed by combining near-atomic scale three dimensional atom maps with first principle calculation. The possible relationship between the formation of precipitates and dislocation loops are discussed. Additionally, attempts are made to quantitatively correlate these microscopic defects with macroscopic hardening in heavy ion irradiation by utilizing the heterogeneous defect depth distribution observed.

High purity solution-annealed 316L stainless steel model alloys are prepared, and are irradiated by heavy ions at 290~450°C in this study. The major post irradiation analysis include nano-indentation, transmission microscopy and atom probe tomography. The principal results obtained are:

- 1) The formation of black dot is not much influenced by Si at 290°C. However, Frank loops are distinctively suppressed by Si addition at 400°C in both density and size, especially in the near-surface region. This could be explained by Si's role in enhancing the effective diffusivity of vacancies and thus promoting recombination. It could also be explained if the Si addition can promote the trapping of interstitials by surface sink. For low Si samples, the unfauling of Frank loops is not evident until the irradiation temperature is raised to 450°C.
- 2) Ni-Si precipitates are formed in both base Si (0.42wt.%) and high Si (0.95wt.%) samples irradiated at 450°C to 5dpa. In well-developed Ni-Si precipitates, Ni/Si atom ratio is found to be smaller than 3 while maintaining Ni+Si \approx 96at.% by atom probe tomography. It could be explained by VASP calculation that when one Ni atom is replaced by Si, the configuration is still preferable as its defect formation energy is very close to zero. Mo and Mn are fully depleted at an early stage of precipitate formation.
- 3) Some Ni-Si precipitates are found to be of ring shape. And base on the shape, size and orientation, they should have formed on dislocation loops. Si addition retards loop unfauling, possibly via suppressing Frank loop size or stabilizing Frank loops by precipitation.
- 4) The irradiation hardening tested by nano-indentation matches the microstructure observed in this work. The Orowan model can also be applied in heavy ion irradiation by averaging the inhomogeneous loop density and size in a semi-spherical plastic zone. A hardening coefficient of around 0.30 is obtained for all the three samples irradiated at 400°C to 3dpa by assuming the maximum depth of

the plastic zone to be five times of the indentation depth. This hardening coefficient value meets the lower limit of previous literature data.

The present study is a fundamental research on stainless steel degradation under irradiation. It contributes to ageing management and nuclear safety by improving the knowledge base of degradation behavior. It found the complexity in the stoichiometry of Ni-Si precipitates, and confirmed the interactions between dislocation loops and precipitates, which both emphasize the importance of further studies on Ni-Si precipitates. It provides references for future designs of stainless steels by further clarified the effects of alloying element Si in irradiation. It improves the reliability of using the heavy ion irradiation tool to emulate neutron damage.

Further work on Ni-Si precipitates is suggested based on the results obtained in this work. The nature of Ni-Si precipitates and their role during stainless steel deformation needs to be reconsidered. Their interactions with dislocation loops need to be further analyzed by modeling.