



The effect of injected interstitials on void formation in self-ion irradiated nickel containing concentrated solid solution alloys



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HIGHLIGHTS

- The effect of injected interstitials on Ni containing solid solution alloys varies.
- A refined defect cluster migration mechanism is proposed for NiCo.
- The dose-dependent swelling is studied for two ion fluences.

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ABSTRACT

Pure nickel and three nickel containing single-phase concentrated solid solution alloys (SP-CSAs) have been irradiated using 3 MeV Ni²⁺ ions at 500 °C to fluences of 1.5×10^{16} and $5.0 \times 10^{16} \text{ cm}^{-2}$. The radiation-induced voids were characterized using cross sectional transmission electron microscopy that distributions of voids and dislocation loops were presented as a function of depth. A various degree of void suppression was observed on the tested samples and a defect clusters migration mechanism was proposed for NiCo. In order to sufficiently understand the defect dynamics in these SP-CSAs, the injected interstitial effect has been taken into account along with the 1-dimensional (1-D) and 3-dimensional (3-D) interstitial movement mechanisms.

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1. Introduction

Nickel and various single-phase nickel alloys have been used in studying the alloying effects in irradiation induced defect evolution [1–4]. Heavy-ions and neutrons are commonly used as the irradiation source to investigate the effect of alloying elements on defect cluster formation, such as swelling and radiation-induced segregation [1–4]. In some cases, minor elements in nickel alloys existed in the form of ordered solutes [1,4–7] and their distributions were not completely random. Recently, a family of single-phase concentrated solid solution alloys (SP-CSAs) with two to five

equiatomic principle elements (Ni, Co, Fe, Cr, Mn) has been developed. These compositionally complex alloys were fabricated in the form of single-phase face-centered cubic (fcc) crystal structure with completely random atomic distributions. The alloys and single crystal nickel were then irradiated to various conditions to study the irradiation induced defect dynamics due to composition changes [8–12].

Previous studies have shown the changes in irradiation-induced defect behavior and mechanical properties for different SP-CSAs through experimental and simulation results. A variation in defect migration range and migration energy has been demonstrated in a series of recent studies [10,13–18]. In a study conducted at room temperature, three sets of nickel, NiCo and NiFe SP-CSAs were irradiated with 3 MeV Au²⁺ ions to fluences of 2×10^{13} , 1×10^{14} and $5 \times 10^{15} \text{ cm}^{-2}$. The research found that as the fluence

Abbreviations: SP-CSAs, single-phase concentrated solid solution alloys.

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increased, the defect migration range increased dramatically; this result was supported by cross-sectional transmission electron microscopy (TEM) images and Rutherford backscattering spectrometry (RBS-C) channeling spectra [13]. The extended defect migration range, which is deeper than predicted by SRIM simulation, was explained by enhanced defect migration. The highly mobile defects such as interstitials and small vacancy clusters were found to migrate further than the predicted range, with the assistance of mechanical stress from implanted ions. They also determined that the deeper migration range found in nickel than in NiCo and NiFe for all testing conditions was due to a higher defect migration rate in nickel; this conclusion was supported by a simulation study conducted by Béland et al. [19] using atomistic modeling.

To better study the dynamics of irradiation induced defects, high temperature irradiation was conducted at 500 °C (~0.45 T_m , melting point of Ni). Defect mobility and migration in nickel and nickel-containing SP-CSAs (NiCo, NiFe, NiCoFe, NiCoFeCr and NiCoFeCrMn) were studied by Lu et al. [20] with 1.5 and/or 3 MeV Ni^{2+} ions irradiated to 3×10^{15} and $5 \times 10^{16} cm^{-2}$, respectively. In that study, the authors found that void formation was suppressed in multicomponent single phase alloys and categorized the materials into two groups to explain the defect migration mechanism. The criteria were based on the relative depth distribution of voids and dislocation loops along with the average void size presented in the samples. The group containing nickel and NiCo showed large voids in the irradiated region closer to the sample surface while interstitial type dislocation loops were separated from voids and existed in a deeper region. The other group containing NiFe, NiCoFe, NiCoFeCr and NiCoFeCrMn showed much smaller voids distributed beyond the end of the predicted ion range, while dislocation loops existed closer to the surface. Increasing the irradiated ion energy or fluence did not alter the observed distribution of various types of defect clusters. The two different types of defect clusters were attributed to the two different modes for the interstitial cluster motion in alloys; from a long range one-dimensional (1-D) mode in nickel to a short range three-dimensional (3-D) mode in NiFe, revealed by molecular dynamic simulations. It was also determined that the void swelling resistance found in the SP-CSAs is correlated to the segregation between interstitial and vacancy type defect clusters. The self-organization of irradiation induced defects was affected by the different atomic sizes of principal alloying elements in these SP-CSAs.

The study of mechanical properties for nickel containing SP-CSAs was performed by Jin et al. [21]. The materials used in their study included single crystal nickel, NiCo, NiFe, NiCoCr, and NiCoFeCr, as well as polycrystalline NiCoFeCrMn with millimeter grain size. The irradiation at 500 °C was conducted using 3 MeV Ni^{2+} ions to a peak damage dose about 53 dpa. The swelling induced by irradiation was measured by a step-height method using optical profilometer. The results showed that nickel had the highest swelling, followed by NiCo, NiCoCr, NiFe and NiCoFeCr then NiCoFe and NiCoFeCrMn. The correlation between irradiation induced hardening and step-height based swelling was previously studied [21], atomic level insights on dislocation loop movements are highly desirable to reveal the correlation between the two measurements.

Even though the defect cluster movement and the swelling behavior at elevated temperature in Ni-containing SP-CSAs have been reported, the depth dependence of swelling has not been studied in detail. For example, in the study performed by Jin et al. [21] only the step-height based overall swelling is calculated, which did not reveal the commonly considered depth-dependent or peak swelling information. In ion irradiated materials, the damage and injected ion concentration vary with depth, and their interaction

can result in complicated swelling behavior.

Depth dependent information can be achieved through preparing TEM samples in a cross-sectional method. A full understanding of the swelling behavior in the Ni-containing SP-CSAs requires a detailed analysis of voids as a function of depth over the entire ion range and even beyond. The objective of this study is to explore the depth dependent swelling behavior of the irradiated material and to further explore its relationship to the 1-D and 3-D defect migration mechanism based on quantitative microstructure characterization.

2. Experiments

2.1. Ion irradiation experiments

Single crystal nickel and equiatomic nickel-containing SP-CSAs NiCo, NiFe and NiCoFeCr were investigated in this study. The single crystal materials were prepared using pure elemental metal Ni, Co, Fe and Cr (>99.9% pure) by arc melting. The single crystals were then grown from the polycrystalline ingots using a high temperature optical floating-zone directional solidification method. The actual composition of Ni (50.5 at%) and Fe (49.5 at%) determined by atom probe tomography (APT) [22] were close to the nominal composition, and no impurities other than trace O, N, and Ga, which were induced from APT sample preparation, were detected in all the samples. The nominal composition is, therefore, used for all the materials unless otherwise stated. Details of materials preparation and single crystal growth can be referred to reference [23]. Two sets of materials were irradiated with 3 MeV Ni^{2+} ions at 500 °C to fluences of 1.5×10^{16} and $5.0 \times 10^{16} cm^{-2}$, respectively. The ion flux was controlled at $2.8 \times 10^{12} cm^{-2} / s^{-1}$. The irradiation used a rastered beam with scanning frequencies of 517 and 64 Hz for the horizontal and vertical direction, respectively [24]. The irradiation induced damage profile and injected ion concentration were calculated using SRIM-2013 in Quick Kinchin Pease Mode with a displacement threshold energy of 40 eV. Since atoms were displaced in a completely random form in nickel-containing SP-CSAs, nickel was chosen to be the target material in SRIM simulation. The simulation was also performed for the rest of tested materials, but no significant difference was observed. The calculated peak damage dose is about 17 and 53 dpa for the two ion fluences, respectively. The results of the calculations are plotted in Fig. 1 as a function of depth.

2.2. Microstructure analysis

Cross-sectional TEM samples were prepared by FIB lift-out technique on an FEI Helios Nanolab workstation at the Michigan Center for Material Characterization at the University of Michigan (MC²). In order to remove defects induced by FIB preparation and focus on studying defects from the primary ion irradiation, flash polishing was performed [13]. The void distribution was characterized before flash polishing using JEM 3011TEM with under-focused condition in bright field. TEM samples were tilted away from the zone axis to enhance the contrast for voids. The dislocation loops were imaged near $g = [200]$ two beam condition after flash polishing with a small degree of tilting and defocus to enhance void contrast. Sample thickness was measured using the electron energy loss spectroscopy (EELS) method using a JEOL 2100F aberration-corrected TEM.

The calculation method for void swelling is described as follows. ImageJ was used as the image processing software for counting voids. The voids were profiled in 100 nm increments in depth, starting from the surface, except in the case of pure nickel irradiated to $5.0 \times 10^{16} cm^{-2}$. Since the average void diameter exceeded

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