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To cite this article: G. Velişa, E. Wendler, S. Zhao, K. Jin, H. Bei, W. J. Weber & Y. Zhang (2018) Delayed damage accumulation by athermal suppression of defect production in concentrated solid solution alloys, Materials Research Letters, 6:2, 136-141, DOI: [10.1080/21663831.2017.1410863](https://doi.org/10.1080/21663831.2017.1410863)

To link to this article: <https://doi.org/10.1080/21663831.2017.1410863>



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ORIGINAL REPORT



Delayed damage accumulation by athermal suppression of defect production in concentrated solid solution alloys

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ABSTRACT

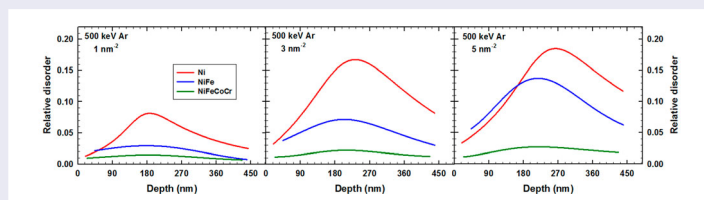
A combined experimental and computational evaluation of damage accumulation in ion-irradiated Ni, NiFe, and NiFeCoCr is presented. A suppressed damage accumulation, at early stages (low-fluence irradiations), is revealed in NiFeCoCr, with a linear dependence as a function of ion fluence, in sharp contrast to Ni and NiFe. This effect, observed at 16 K, is attributed to the complex energy landscape in these alloys that limits defect mobility and therefore enhances defect interaction and recombination. These results, together with previous room-temperature and high-temperature investigations, suggest ‘self-healing’ as an intrinsic property of complex alloys that is not a thermally activated process.

ARTICLE HISTORY

Received 24 October 2017

KEYWORDS

SP-CSAs; ion irradiation; RBS/C; *ab initio* calculations



IMPACT STATEMENT

A combined experimental and computational evaluation reveals a remarkable delayed damage accumulation due to significant athermal suppression of defect production in ion-irradiated concentrated solid solution alloys at 16 K.

1. Introduction

Interest in single-phase concentrated solid solution alloys (SP-CSAs) has rapidly expanded in recent years [1–13], fueled by the atomic-level tunability that results from altering principal alloying elements and modifying their concentrations. SP-CSAs contain multiple elemental species in equiatomic or high concentrations with different elements randomly arranged on a simple crystal structure (e.g. face-centered cubic, fcc). On the other hand, the random arrangement of multiple species in a simple crystalline structure results in chemical disorder (e.g. distortion of the electronic and lattice structures) and complex energy landscapes that allow tailoring physical properties of SP-CSAs [2,5]. Excellent mechanical properties [4,12,13], good corrosion resistance [7], and

high radiation resistance [2,5] are already discovered in some SP-CSAs. In extreme environments (e.g. nuclear reactor applications), a combination of these improvements is highly desirable, and exploiting the extreme chemical disorder of concentrated alloys may provide a new paradigm for designing structural materials for next-generation nuclear power systems.

Over the past few years, pioneering efforts have been devoted to the fundamental understanding of radiation defects in SP-CSAs, mimicking the radiation produced in nuclear reactors using external irradiations with various types of heavy ions at room temperature (RT) [14–19] and high temperatures (HT) [20–26]. The results of these studies have demonstrated that increasing chemical complexity strongly enhances the radiation resistance

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Supplemental data for this article can be accessed here. <https://doi.org/10.1080/21663831.2017.1410863>

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of these alloys, which has been attributed to sluggish diffusion [27–29] and enhanced dynamic annealing of simple defects at early stages during irradiations at RT and HT [2,15,23]. Expanding the scientific understanding of the temperature-dependent response of SP-CSAs under ion irradiation to cryogenic temperatures (e.g. 16 K) should clarify if the enhanced defect recombination is an intrinsic property of chemically disordered CSAs or if the observed radiation resistance under RT and HT irradiations is a thermally activated process.

Rutherford backscattering spectrometry in channeling conditions (RBS/C) is frequently employed to quantitatively determine depth profiles of disorder in ion-irradiated single crystals. Recently, we have utilized RBS/C to profile irradiation-induced damage evolution in SP-CSAs at RT [14–19]. The detection limit of the disorder is expected to be enhanced at cryogenic temperatures due to the reduction of thermal vibrations [30,31]. The resulting minimum channeled yield ($\chi_{\min}$) and the rate of dechanneling can be much reduced. Reduced background noise will decrease the measurement uncertainty in determining disorder in ion-irradiated single crystals [30,31]. It is thus desirable to perform RBS/C studies at temperatures as low as possible. More importantly, key knowledge on the primary damage process under ion irradiation in SP-CSAs can be gained at very low temperatures, where radiation damage may be frozen in the host matrixes [32].

2. Experimental details

To study cryogenic irradiation response, three fcc single crystals of elemental Ni, binary NiFe, and quaternary NiFeCoCr, all at equiatomic ratios, are chosen as model alloys for investigation. This set of crystals is ideal to understand the impact of chemical disorder and compositional complexity effects on the primary damage production under ion irradiations at low temperature. Crystals were irradiated at 16 K with 500 keV Ar^{2+} ions to fluences from 0.35 to 60 ions nm^{-2} . The upper ion fluence is chosen depending on the disorder saturation level identified using RBS technique (for details, see Table 1 and discussion below).

Argon ion irradiation and *in situ* RBS/C analyses were both performed at 16 K in a special target chamber at the Institut für Festkörperphysik in Jena, Germany. The experimental apparatus allows both ion irradiation and

subsequent ion beam analysis to be carried out without changing either the target temperature or the environment of the samples [33]. The mean projected ion range, R_p , of Ar ions, based on Stopping and Range of Ions in Matter (SRIM) calculations [34], is ~ 200 nm with a range straggling $\Delta R_p \sim 60$ nm. The corresponding displacements per atom (dpa) profile was predicted by SRIM using the Kinchin–Pease model under an assumed threshold displacement energy of 40 eV for all elements [14]. The conversion factor at the SRIM-predicted damage peak (~ 155 nm) from ion fluence (1 nm^{-2}) to dpa is 0.152. *In situ* RBS/C measurements, employing 1.4 He ions, were performed to obtain the damage profiles for each ion fluence.

To understand different defect behavior in Ni and two alloys, defect energetics were calculated from *ab initio* method based on density-functional theory using the VASP code [35]. The exchange–correlation interactions were described by the Perdew–Burke–Ernzerhof (PBE) functional [36]. Electron-ion interactions were modeled with the projector-augmented-wave (PAW) method [37]. The energy cutoff was set to be 270 eV and the energy convergence was set to be 10^{-4} eV. All calculations were performed with spin-polarization to allow full relaxation of the magnetic moments. A 256-atom supercell was used and a Gamma-point-based $2 \times 2 \times 2$ k-point mesh was employed. The alloys were modeled using special quasirandom structures [38,39] constructed by minimizing the short-range order parameter. The migration barriers of interstitials and vacancies were calculated by directly optimizing the saddle-point configuration [40].

3. Results

In order to evaluate qualitatively the evolution of the relative disorder resulting from the Ar-ion irradiation, disorder profiles were extracted from the analysis of RBS/C spectra (these spectra are plotted in Figure S1 of the supplementary material) using an iterative procedure [41], which has been previously used to analyze ion-irradiated SP-CSAs at RT [18,19] and validated in some recent molecular dynamics simulations [42,43]. The obtained disorder profiles for Ar-irradiated Ni (red circles) are illustrated in Figure 1 and compared with those for Ar-irradiated NiFe (blue triangles) and NiFeCoCr (green squares) single crystals. For clarity, only three irradiation fluences are presented: (a) 1, (b) 3, and (c) 5 nm^{-2} . The data points of these three fluences are representative of measurement statistics of the whole fluence range investigated. The solid lines are curve fits to the data to guide the eye. The results in Figure 1 demonstrate that the disorder in NiFe and NiFeCoCr are lower compared

Table 1. Ion fluence range for 500 keV Ar^{2+} irradiation at 16 K on Ni, NiFe, and NiFeCoCr.

	Ni	NiFe	NiFeCoCr
Fluence range (nm^{-2})	0.35–5	0.35–20	0.35–60

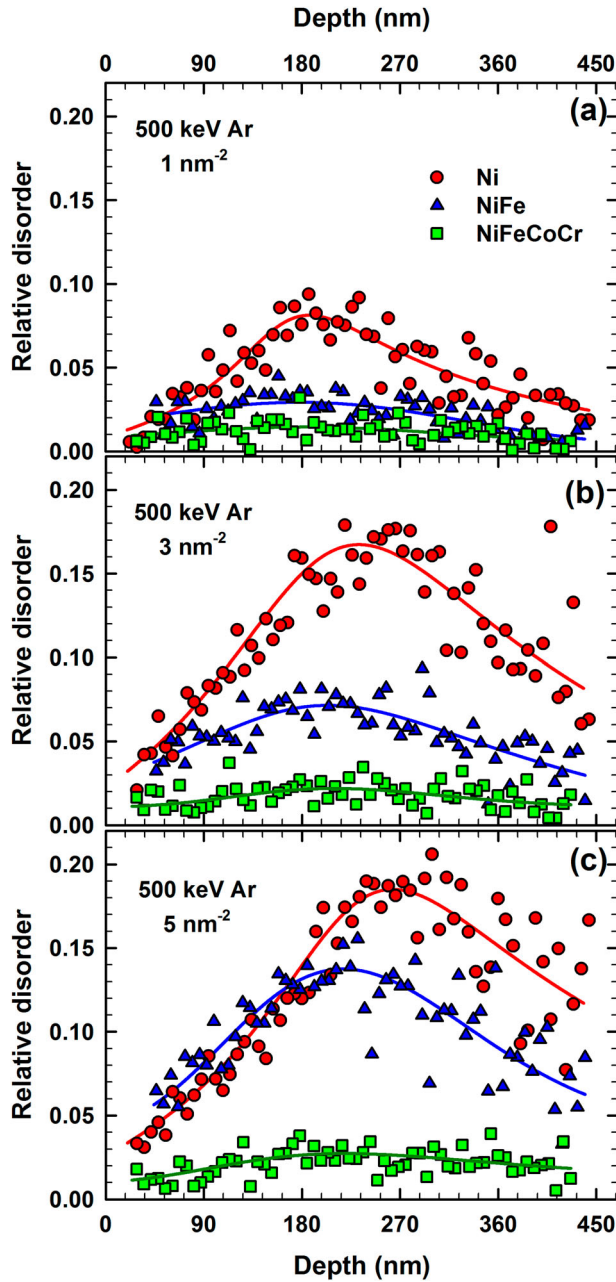


Figure 1. Disorder profiles, extracted using an iterative procedure [41] from the corresponding channeling spectra for Ni, NiFe, and NiFeCoCr single crystals irradiated at 16 K with 500 keV Ar ions at indicated fluences: (a) 1, (b) 3, and (c) 5 nm^{-2} , respectively. The solid lines are curve fits to guide the eye.

with Ni, indicating less damage in the alloys. Between the two alloys, the damage level in NiFeCoCr is lower than in NiFe.

Figure 2(a) depicts for all the irradiated samples the evolution of the disorder measured at 180 nm as a function of both the Ar-ion fluence (bottom axis) and the dpa value (top axis) at the SRIM-predicted dpa peak. At low ion fluences (i.e. up to 1 nm^{-2}), the maximum of the damage peak is located around 180 nm (see Figure 1(a)), i.e. very close to the maximum of the SRIM-predicted

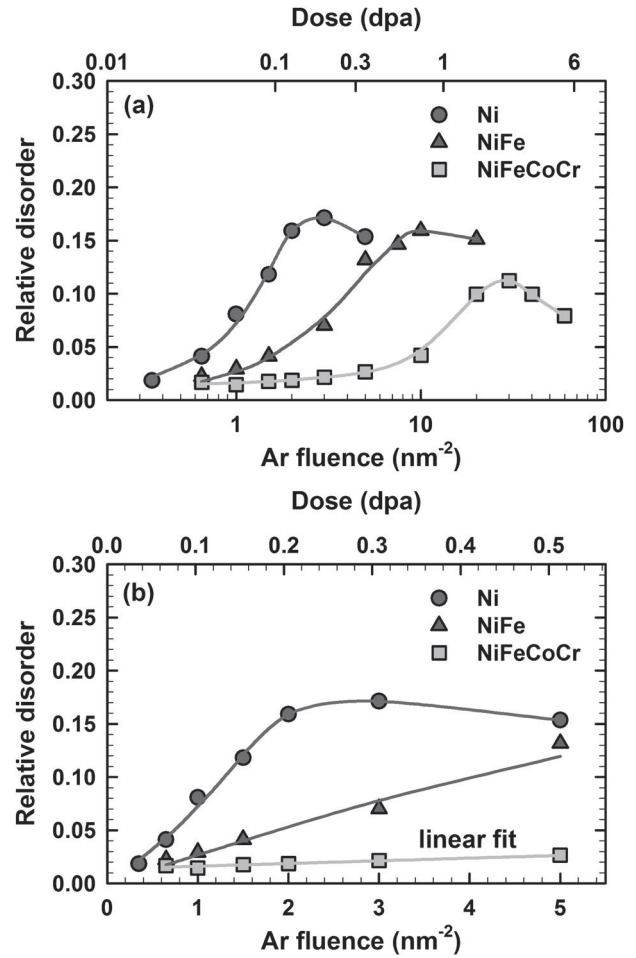


Figure 2. Disorder at the damage peak ($\sim 180 \text{ nm}$) as a function of both the Ar-ion fluence (bottom axis) and the dpa value (top axis) for Ni, NiFe, and NiFeCoCr irradiated with 500 keV Ar ions at 16 K. For convenience, Ar fluence is converted in displacements per atom (dpa). The dashed lines are curve fits to guide the eye. For better visibility of all data, part (a) has a logarithmic and (b) a linear scale.

dpa peak ($\sim 155 \text{ nm}$). While at higher ion fluences, the depth of maximum damage shifts deeper into the material due to long-range effects (for a detailed description of the effects, see e.g. Ref. [17,22]); this is the reason why the damage evolution is compared at 180 nm. The results in Figure 2 clearly show that, with continuously increasing ion fluence, the damage reaches saturation in Ni between fluences of 3 and 5 nm^{-2} , while the damage build-up continues in NiFe and NiFeCoCr until fluences approaching 10 and 30 nm^{-2} , respectively. These results reveal the existence of a material-dependent threshold value for the ion fluence where disorder level, determined using RBS, reaches saturation. Moreover, the linear dependence of damage accumulation in NiFeCoCr (see linear fit in Figure 2(b)) from 0.35 to 5 nm^{-2} indicates small residual irradiation damage after each ion strike, which is not the case in Ni and NiFe. This result reveals suppressed defect

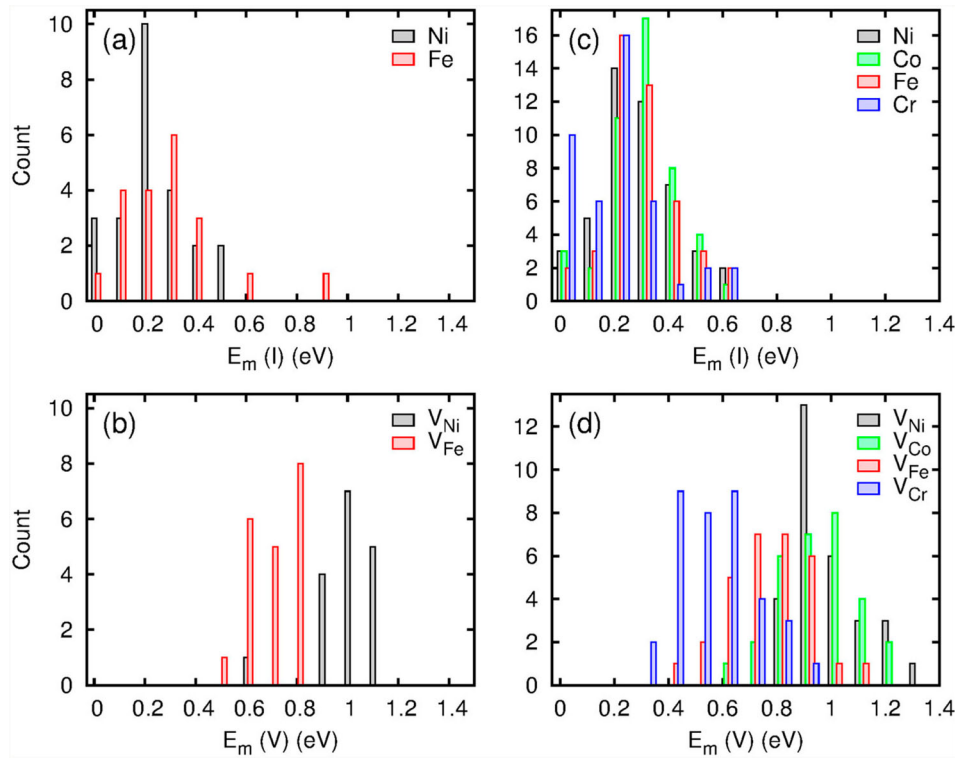


Figure 3. Migration barrier (E_m) distribution of interstitials (I) and vacancies (V) in NiFe (left column) and NiFeCoCr (right column).

production at low fluences in NiFeCoCr, which delays damage accumulation in NiFeCoCr compared to Ni and NiFe.

4. Discussion

In the SP-CSAs used in this study, the constituent elements are distributed randomly on an fcc lattice, which means there is a variation in the nearest neighbors from lattice site to lattice site. This variation in both local composition and in the size of constituent atoms creates unique site-to-site lattice distortion and locally disordered chemical environments [2,5,14]. It has been reported that increasing chemical disorder can lead to a substantial reduction in electron mean free paths, a decrease in electrical and thermal conductivity, and complex energy landscapes, all of which affect defect production and damage evolution [2,5]. Recently, systematic physical property measurements on a variety of SP-CSAs compositions have revealed that NiFeCoCr alloy has the highest electrical resistivity and lowest thermal conductivity among the model crystals chosen in this study [2,5,14,44]. The decrease of thermal conductivity may enhance the recombination of irradiation-induced point defects due to a prolonged thermal spike under irradiation [45,46]. This is consistent with recent results from two-temperature MD (2T-MD) simulations that indicate that energy exchange between the atomic and electronic

subsystems clearly affects defect production and evolution [45,46]. The electronic energy loss returned to the lattice via e-ph interactions enhances defect recombination and results in fewer defects. In other words, more localized heat in complex alloys (higher thermal ‘spike’ temperature for a longer time) results in less isolated defects, and smaller clusters. The 2T-MD results are consistent with the slow damage accumulation observed in NiFeCoCr alloy (see Figure 2), and provide underlying insights.

In this study, the energy transferred to SP-CSAs from 500 keV Ar is high enough to knock out lattice atoms, creating lots of interstitials and vacancies. Therefore, their evolution after creation plays a dominant role in governing the observed distinct disorder profiles. To understand the difference, the migration barriers in the considered SP-CSAs are characterized and provided in Figure 3. In Ni, the migration barriers are 0.15 and 1.04 eV for interstitials and vacancies, respectively. Due to chemical disorder, migration barriers exhibit distributions in SP-CSAs.

Figure 3 shows that most migration barriers of interstitials in SP-CSAs are higher than those in Ni, whereas those of vacancies in SP-CSAs are lower than those in Ni. The results confirm sluggish diffusion of interstitials in SP-CSAs [28,29,47]. In addition, the increased barriers for interstitials and lowered barriers for vacancies show a significant overlapping region in their migration barrier

distributions, which tends to enhance the interactions between interstitials and vacancies to further facilitate defect recombination [28,29,47]. Larger overlap region is observed in NiCoFeCr compared to NiFe, which may explain the high defect recombination efficiency found in NiCoFeCr, as shown in Figure 2.

Experimental evidence has shown that in Ni [48] energetic displacement cascades collapse in vacancy dislocation loops even at irradiation temperatures as low as 30 K, where long-range thermal migration of point defects is not possible, and thus indicates that collapse is athermal. This phenomenon requires that vacancies and interstitials do not interact, so that a large proportion survives recombination, and a direct formation of extended defect from cascades interacting with accumulated defects [48]. This interpretation could hold for the present results, since under high fluence irradiation, the formation and growth of stable extended defects are expected [2,5].

5. Conclusion

In conclusion, the defect evolution in Ar-irradiated Ni, NiFe, and NiFeCoCr single crystals was studied at 16 K using *in situ* RBS/C measurements. Our results show that the defect evolution is delayed with increasing compositional complexity from Ni to NiFe, and to NiFeCoCr alloy. These results are in good agreement with previous RT and HT investigations that have showed suppressed damage accumulation with increasing chemical disorder in SP-CSAs under ion irradiation, which is attributed to sluggish diffusion and dynamic annealing of simple defects at early stages during irradiations. The results presented demonstrate that the enhanced radiation resistance is an intrinsic property of complex concentrated solid solution alloys that is not a thermally activated process.

Acknowledgements

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Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported as part of the Energy Dissipation to Defect Evolution (EDDE), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Basic Energy Sciences.

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