

# Ion irradiation induced disappearance of dislocations in a nickel-based alloy



H.C. Chen, D.H. Li, R.D. Lui, H.F. Huang, J.J. Li, G.H. Lei, Q. Huang, L.M. Bao, L. Yan<sup>\*</sup>, X.T. Zhou<sup>\*</sup>, Z.Y. Zhu

Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China

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## ABSTRACT

Under Xe ion irradiation, the microstructural evolution of a nickel based alloy, Hastelloy N (US N10003), was studied. The intrinsic dislocations are decorated with irradiation induced interstitial loops and/or clusters. Moreover, the intrinsic dislocations density reduces as the irradiation damage increases. The disappearance of the intrinsic dislocations is ascribed to the dislocations climb to the free surface by the absorption of interstitials under the ion irradiation. Moreover, the in situ annealing experiment reveals that the small interstitial loops and/or clusters induced by the ion irradiation are stable below 600 °C.

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## 1. Introduction

Dislocations are ubiquitous defects and have great effects on the properties of crystalline materials, such as mechanical, chemical and physical properties [1–4]. In metallic alloys, the mechanical properties are often governed by the generation and interaction of the defects and dislocations [5]. Therefore, the modification of dislocations is an important issue in metallic alloys [6,7].

Irradiation may introduce many defects, such as vacancies, interstitials, clusters, loops, voids and stacking-fault tetrahedra (SFT), which have dramatic effects on the microstructure and mechanical properties of the structural materials [8–11]. Thus, numerous studies are focused on the interaction of defects [12–15]. Moreover, under ion irradiation, the behavior of the intrinsic dislocations is also important.

In some neutron irradiated metals, the decoration of the intrinsic dislocations with interstitial clusters or interstitial-type loops has been reported [16–20]. Trinkaus et al. and Mukouda et al. ascribed the decoration to the trapping of the interstitial loops or clusters by the intrinsic dislocations [17,18]. However, this mechanism are not enough to explain the fact that the intrinsic dislocations density decreases with increasing the irradiation dose [18].

Molten Salt Reactors (MSR) is one of the six most promising Generation IV fission reactors [21]. Hastelloy N alloy (US

N10003) has been selected as structure materials for MSR, due to its excellent compatibility with fluoride and high-temperature mechanical properties [22,23]. Here, the disappearance of intrinsic dislocations under ion irradiation in a nickel-based Hastelloy N alloy (US N10003) was studied. The disappearance of the intrinsic dislocations arises from the climb of the intrinsic dislocations due to the absorption of interstitials.

## 2. Experimental procedure

The material used in the present study is a commercial Hastelloy N alloy (US N10003). The chemical composition of the alloy is shown in Table 1. The samples cut from the Hastelloy N alloy (US N10003) plate were mechanically polished to a thickness of approximately 100 μm and punched to discs with a diameter of 3 mm. Then, the discs were electro-polished with a solution of 5% HClO<sub>4</sub> and 95% CH<sub>3</sub>CH<sub>2</sub>OH below −30 °C.

The as-prepared thin foils were irradiated with 1 MeV Xe<sup>20+</sup> ion beams at room temperature on the terminal of 320 kV High-Voltage Experimental platform equipped with an electron cyclotron resonance ion source in Institute of Modern Physics, Lanzhou, China. The ion doses were  $3.07 \times 10^{13}$  ions · cm<sup>−2</sup> and  $7.68 \times 10^{13}$  ions · cm<sup>−2</sup>, respectively. The irradiation damage was estimated by SRIM-2008 with the “Quick” Kinchin-Pease option [24,25] and plotted in Fig. 1, wherein the displacement energy was 40 eV [25]. The peak irradiation damages was 0.13 dpa (for  $3.07 \times 10^{13}$  ions · cm<sup>−2</sup>) and 0.33 dpa (for  $7.68 \times 10^{13}$  ions · cm<sup>−2</sup>), respectively. The main

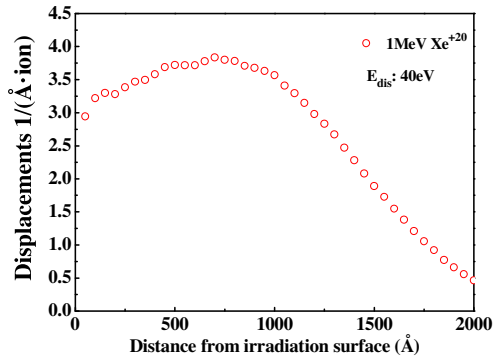
<sup>\*</sup> Corresponding authors at: Shanghai Institute of Applied Physics, Chinese Academy of Sciences (CAS), 2019 Jialuo Rd., Jiading, 201800 Shanghai, China.

E-mail addresses: [yanlong@sinap.ac.cn](mailto:yanlong@sinap.ac.cn) (L. Yan), [zhouxingtai@sinap.ac.cn](mailto:zhouxingtai@sinap.ac.cn) (X.T. Zhou).

**Table 1**

Chemical composition of Hastelloy N alloy (US N10003) (wt.%).

Ni	Mo	Cr	Fe	Mn	Si	Ti	Cu	Co	C
71.88	16.6	7.09	3.83	0.52	0.46	<0.002	<0.002	<0.002	0.04

**Fig. 1.** SRIM calculation of the damage profile produced by 1 MeV  $\text{Xe}^{20+}$  ion irradiation.

radiation region is distributed in the range of 0–100 nm which is fit for transmission electron microscopy (TEM) observations.

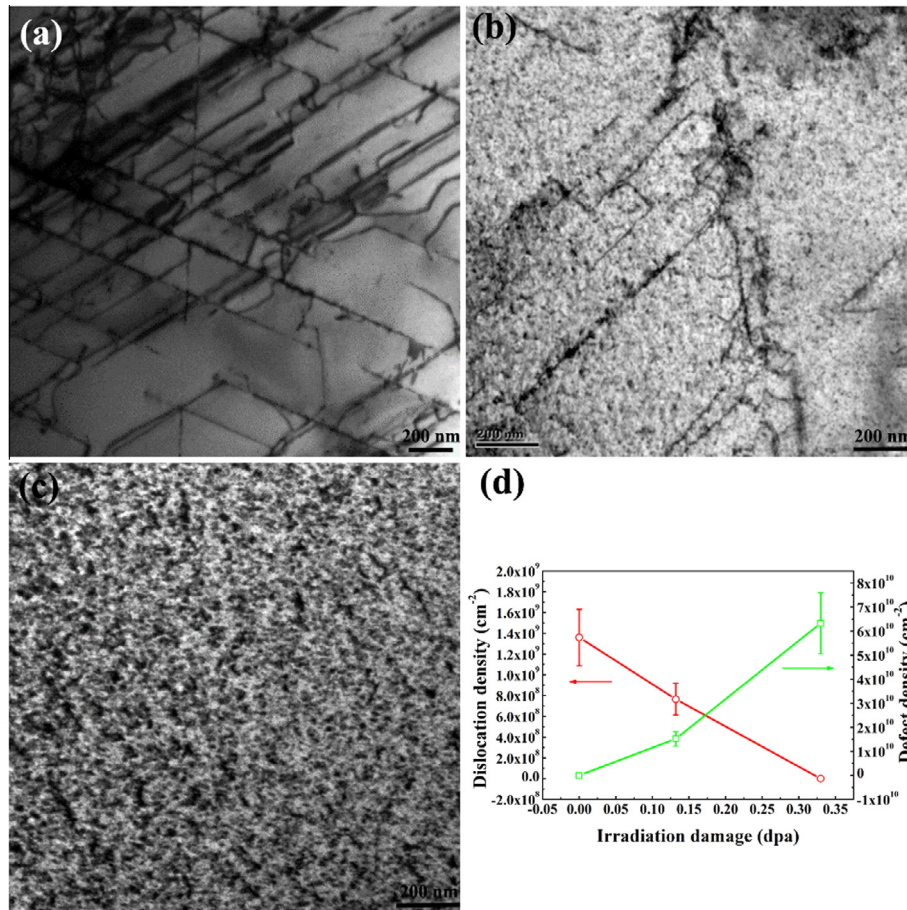
The microstructural analysis was performed at room temperature on a FEI Tecnai G<sup>2</sup> F20 TEM, operated at 200 kV. In situ isochronal annealing of the irradiated sample with an irradiation

damage of 0.33 dpa was carried out at the TEM employing with a Gatan 652 double-tilt heater, and the desired temperature was reached by a  $\sim 10^\circ\text{C}/\text{min}$  ramp rate. TEM micrographs were taken almost every  $100^\circ\text{C}$ . The statistics analysis of the intrinsic dislocations and the irradiation induced defect were carried out using Image J [26], and the TEM images for statistics analysis are all acquired at down-zone condition [27]. The thickness of TEM foils can affect the distribution of the defects induced under ion irradiation [28,29]. Here, the foil thickness was estimated by convergent-beam technique, and the TEM images were taken at the thickness of  $\sim 100$  nm.

### 3. Results and discussions

#### 3.1. Damage evolution

The typical TEM bright-field (TEM-BF) images of as prepared and irradiated thin foils are shown in Fig. 2. For the as-prepared thin foil sample, a lot of intrinsic dislocations were observed, and little other defects were presented (Fig. 2(a)). The intrinsic dislocations are edge dislocations dominantly due to the perpendicular between the Burgers vector and the direction of dislocations. After irradiation, the density of dislocations becomes lower, compared to the as prepared sample. Further, the dislocations density decreases with increasing the irradiation damage. Moreover, the black dot-defects include small dislocation loops and clusters, are accumulated in the form of a decoration along the intrinsic dislocations, which is corresponding with our previous work [30]. Similar

**Fig. 2.** TEM bright-field (TEM-BF) images of Hastelloy N alloy (US N10003) samples (a) before irradiation and after irradiation with a damage of (b) 0.132 dpa and (c) 0.33 dpa. All the images were taken at  $[110]$  zone axis. (d) The plots of the defect and dislocation densities vs the irradiation damage, the errors given are counted by statistics of different images.

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