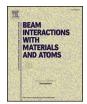
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## The irradiation hardening of Ni-Mo-Cr and Ni-W-Cr alloy under $\mathrm{Xe}^{26\,+}$ ion irradiation



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#### ABSTRACT

The irradiation hardening of Ni-Mo-Cr and Ni-W-Cr alloy was investigated.  $7 \, \text{MeV} \times \text{e}^{26\,^+}$  ion irradiation was performed at room temperature and  $650\,^{\circ}\text{C}$  with peak damage dose from 0.05 to 10 dpa. With the increase of damage dose, the hardness of Ni-Mo-Cr and Ni-W-Cr alloy increases, and reaches saturation at damage dose  $\geq 1$  dpa. Moreover, the damage dose dependence of hardness in both alloys can be described by the Makin and Minter's equation, where the effective critical volume of obstacles can be used to represent irradiation hardening resistance of the alloys. Our results also show that Ni-W-Cr alloy has better irradiation hardening resistance than Ni-Mo-Cr alloy. This is ascribed to the fact that the W, instead of Mo in the alloy, can suppress the formation of defects under ion irradiation.

#### 1. Introduction

Nickel-based alloy is attracting more and more attention for its perfect high temperature mechanical properties and good corrosion resistance, and considered as one of the candidate structure materials for Generation IV fission reactors, especially for molten salts reactor (MSR) [1-3]. The structural materials face serious engineering and technological challenges due to their extreme work environment such as high temperature, molten salts and neutron irradiation [2,4]. Thus, it is still being addressed to screen materials with excellent mechanical strengths and good irradiation resistance. To date, Ni-Mo-Cr alloy, which is a nickel-based solid solution strengthened alloy developed by Oak Ridge National Laboratory, is a candidate structure material for MSR [5]. In nickel-base alloys, the bond order of Ni-W d-d bond (1.730 eV) was larger than that of Ni-Mo d-d bond (1.611 eV), which reveals the strengthening effect of W is better than that of Mo [6]. Hence, the main effective solid solute hardener Mo was replaced by W in a new developed Ni-W-Cr superalloy [7,8]. Numerous studies have verified that both alloys have superior corrosion resistance and good mechanical properties [7,9–15]. However, there are few reports on the irradiation resistance of both alloys [16,17].

Irradiation hardening and embrittlement, which has been an important criterion to evaluate the irradiation resistance, is an essential

#### 2. Experiment procedure

The materials used in the study were Ni-Mo-Cr and Ni-W-Cr alloy. The cast ingots of Ni-Mo-Cr and Ni-W-Cr alloy were prepared by Vacuum induction melting (VIM) and then hot forged into bar with diameter of 40 mm. The nominal composition of the Ni-Mo-Cr and Ni-W-Cr alloy was shown in Table 1. Compared with Ni-Mo-Cr alloy, the

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degradation issue of nuclear structure materials. Generally, ion irradiation is an efficient method to study the irradiation effect of structural materials. Recently, nano-indenter technique has been used widely to characterize the irradiation hardening of materials because the bulk mechanical properties can be extracted from nano-indenter data by Nix-Gao model [16,18–21]. For instance, Zhang et al. evaluated the irradiation hardening of Fe-Cu alloy, ODS ferritic steels and V-Ti alloy using this model [18–20]. Takayama et al. analyzed the effect of nickel on the irradiation hardening of Fe-based alloy with Nix-Gao model [21]. Those works have verified that Nix-Gao model is an effective way to evaluate the irradiation hardening of materials. Here, the irradiation hardening of Ni-Mo-Cr and Ni-W-Cr alloy after Xe<sup>26+</sup> ion irradiation has been investigated and compared. The Makin and Minter's equation was used to describe the irradiation hardening of Ni-Mo-Cr and Ni-W-Cr alloy [22].

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Table 1
The component of the Ni-Mo-Cr and Ni-W-Cr alloy.

Component		Ni	Мо	Cr	Fe	Mn	Si	С	W
Ni-Mo-Cr	wt% at%	Bal. Bal.	17.34 11.15	7.01 8.32	3.92 4.33	0.60 0.67	0.45 0.99	0.06 0.28	-
Ni-W-Cr	wt% at%	Bal. Bal.	1.00 0.74	5.86 7.97	0.54 0.68	_	0.14 0.35	0.04 0.22	26.10 10.04

 Table 2

 The total dose and irradiation damage of the samples.

Samples	Temperature (°C)	Total dose (ions/cm²)	Irradiation damage (dpa) <sup>*</sup>
Ni-Mo-Cr, Ni- W-Cr	RT 650	$1.90 \times 10^{13}$ $3.80 \times 10^{13}$ $1.90 \times 10^{14}$ $3.80 \times 10^{14}$ $1.14 \times 10^{15}$ $3.80 \times 10^{15}$ $1.14 \times 10^{15}$	0.05 0.10 0.50 1.00 3.00 10.00 3.00

 $<sup>^{\</sup>star}$  The irradiation damage dose is calculated with peak value in Fig. 1.

main effective solid solute hardener Mo was replaced by W in Ni-W-Cr alloy. The samples with size of  $10\times10\times1~\text{mm}^3$  were cut from the bars. Prior to irradiation, the samples were heat treated at  $1250\,^{\circ}\text{C}$  for 1 h, and carefully prepared by SiC abrasive paper, diamond suspensions to obtain a smooth surface, and the vibration polishing was used to remove residual stress of the surface.

The ion irradiation of samples was carried out with  $Xe^{26+}$  ions at room temperature (RT) and 650 °C in a terminal of 320 kV High-Voltage Experimental platform equipped with an electron cyclotron resonance ion source in Institute of Modern Physics, Lanzhou, China. The energy of  $Xe^{26+}$  ion was fixed at 7 MeV, and the total doses of the samples were listed in Table 2. During ion irradiation, the incident flux was about  $2\times 10^{11}\, \rm ions/cm^2$ ·s, and the vacuum was in the range of  $1\times 10^{-5}$ – $1\times 10^{-4}\, \rm Pa$ . The irradiation damage was estimated by SRIM-2008 with the "Quick" Kinchin-Pease option and plotted in Fig. 1, wherein the displacement energy is 40 eV [23–25]. The peak value in Fig. 1 was used to calculate the irradiation damage dose.

Nano-indentation test was performed using Nano Indentor G200 (the Agilent Technologies, Inc.) with a Berkovich type indentation tip. The continuous stiffness measurement (CSM) was carried out to obtain

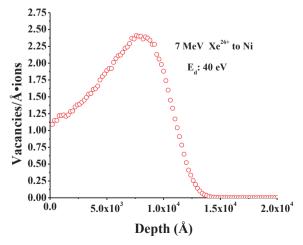
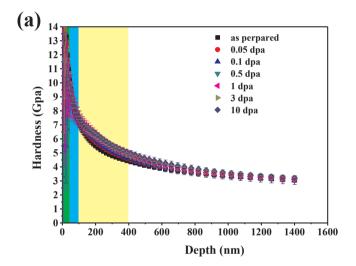


Fig. 1. The damage profile of 7 MeV Xe<sup>26+</sup> calculated by SRIM-2008.

depth (h)-profile of nano-hardness (H). Calibration of blunting of the indentation tip and calculation of hardness is based on the Oliver–Pharr method. 30 indentations were made on each sample with the maximum penetration depth of  $1.5\,\mu m$ , and average of the data was adopted by Analyst<sup>m</sup> software.

For microstructure investigation, the unirradiated TEM samples of Ni-Mo-Cr and Ni-W-Cr alloy were prepared by twin-jet electro-polishing in a solution of 5%  $\rm HClO_4$  and 95%  $\rm CH_3CH_2OH$ , and the cross-sectional TEM samples of Ni-Mo-Cr and Ni-W-Cr alloy with irradiation damage of 1 dpa were prepared by ion milling. Microstructural evolution was examined using a FEI Tecnai  $\rm G^2$  F20 TEM, operated at 200 kV. The image pro plus-software (IPP) was used to count and measure the size of defects for the statistics analysis based on the weak-beam dark field images [26]. The foil thickness was estimated by convergent-beam technique [27], and all the TEM images were taken at the thickness of  $\sim 100$  nm.



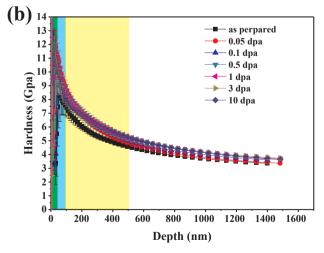


Fig. 2. Indentation-depth dependence of the nano-indentation hardness of (a) Ni-Mo-Cr, (b) Ni-W-Cr alloy.

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