Journal of Nuclear Materials 511 (2018) 220–230

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

A hardening model for the cross-sectional nanoindentation of ion-irradiated materials

Xiazi Xiao [a, *], Long Yu [b]

[a] Department of Mechanics, School of Civil Engineering, Central South University, Changsha, 410075, PR China
[b] State Key Laboratory for Turbulence and Complex System, Department of Mechanics and Engineering Science, College of Engineering, Peking University, Beijing, 100871, PR China

HIGHLIGHTS

- A hardening model is raised for cross-sectional indentation of ion-irradiated metals.
- An average defect density within the plasticity affected region is considered.
- Results of the proposed model match well with corresponding experimental data.

ARTICLE INFO

Article history:
Received 16 July 2018
Received in revised form
10 September 2018
Accepted 11 September 2018
Available online 17 September 2018

Keywords:
Hardness
Irradiation effect
Theoretical model
Cross-sectional indentation

ABSTRACT

In this paper, a hardening model is developed for the cross-sectional nanoindentation of ion-irradiated materials. The model is based on the derivation of an average defect density within the formed plasticity affected region, which depends on the distribution of defect density in the irradiated region, indentation depth and distance from the irradiated sample surface. A succinct parameter calibration process is proposed by comparing the theoretical results with experimental data at a given indentation depth. A good agreement with experimental data can be achieved for both the fitted relationship between irradiation hardening and indentation distance from the irradiated sample surface under cross-sectional nanoindentation, and predicted hardness as a function of indentation depth under surface nanoindentation. Therefore, the rationality and accuracy of the proposed model are effectively verified. Based on the analysis of this proposed model, it is available to characterize the property of plasticity affected region and irradiation depth of ion-irradiated materials based on the experimental data measured through cross-sectional nanoindentation.

1. Introduction

Ion irradiation, combining with the technology of nanoindentation, has now become the most widely applied method to study the mechanical behavior of nuclear materials with irradiation effect [1–7]. Comparing with neutron irradiation, the application of ion irradiation experiments is not only time-saving and cheap [8], but also convenient to analyze the synergetic effect of multifarious irradiation particles [9,10]. Other obvious features of ion irradiation include that the irradiation depth of ion-irradiated materials is quite limited (up to tens of micrometers), and the distribution of defects within the irradiated layer is usually non-uniform [11], which make it appropriate to apply small scale mechanical testing for ion-irradiated materials [12]. Thereinto, nanoindentation, with the advantage of easy sample preparation and large amounts of experimental data offered within a limited time, provides a convenient way to characterize the localized mechanical properties of ion-irradiated materials near the sample surface.

Nanoindentation of ion-irradiated materials can be categorized into surface nanoindentation (or say regular nanoindentation) and cross-sectional nanoindentation [12–14]. Thereinto, surface nanoindentation has been widely adopted to characterize the hardening behavior of different kinds of ion-irradiated materials [3–6]. For instance, three different oxide-dispersion-strengthened ferritic

---

* Corresponding author.
E-mail address: xxz2017@csu.edu.cn (X. Xiao).

https://doi.org/10.1016/j.jnucmat.2018.09.019
0022-3115/

steels were irradiated by high-energy Ni ions with the energy ranging from 13 MeV to 358 MeV, and the maximum irradiation depth reached 24 μm [1]. For all three kinds of steels, irradiation hardening was obviously indicated according to the measured hardness-depth relationship by surface nanoindentation. In order to analyze the synergistic effect of cascade damage and ion accumulation on the mechanical behavior of nuclear materials, Wei et al. [9] compared the hardening behavior of China low active martensitic steel respectively irradiated by single (He^{+}) and dual (He^{+} plus Fe^{11+} subsequently) ions. The corresponding experimental data indicated that no obvious increase of irradiation hardening was observed for the dual ions irradiated samples when compared with the single ion irradiated ones due to the decreasing of defect density during the subsequent irradiation process of Fe^{11+} ions.

In order to obtain a sophisticated comprehension of the irradiation hardening behavior of ion-irradiated materials, several theoretical models have been proposed for the case of surface nanoindentation [8,15–19]. The most widely adopted theoretical model is the Nix-Gao model [20], which was original proposed to explain the indentation size effect of unirradiated materials by introducing the concept of geometrically necessary dislocations. The application of Nix-Gao model for the hardness-depth relationship with ion irradiation effect should be careful as the original model contains no information for the non-uniformly distributed defects within the limited irradiation region. Recently, a hardening model was developed for the depth-dependent hardness of ion-irradiated materials through surface nanoindentation [8,19]. The model takes into account the influence of non-uniformly distributed defects, unirradiated soft substrate and geometrically necessary dislocations, which can effectively characterize the mechanism induced by damage gradient effect, soft substrate effect and indentation size effect. With the application to ion-irradiated stainless steels, the rationality and accuracy of the model are verified according to the good match with experimental data.

Comparing with cross-sectional nanoindentation, surface nanoindentation is easier to apply as surface polish of ion-irradiated samples is not necessary. Whereas, the influence of surface roughness and contamination after irradiation makes it nearly invalid to analyze the hardness data near the irradiated sample surface [14]. As an alternative method, cross-sectional nanoindentation can effectively avoid these malpractices, and offer more reliable experimental data [21]. Moreover, one can directly compare the hardness respectively obtained in the irradiated region and unirradiated region at the same indentation depth through moving the indenter tip along the irradiation direction [12]. Nowadays, the performance of cross-sectional nanoindentation on ion-irradiated materials has drawn increasing attention, and several sets of experiments have been reported in previous literature concerning single crystal copper [14] and 304 stainless steels [22,23]. It is noticed that irradiation hardening is obvious when the indenter tip is within the irradiated region, and the hardness profile closely depends on the irradiation dose. When the indenter tip moves across the boundary of irradiation region, the measured hardness dramatically decreases to the value of unirradiated substrate [14,22]. Moreover, with the increase of testing temperature, the extent of irradiation hardening for ion-irradiated 304 stainless steels gradually weakens [23], which is similar to the case of neutron-irradiated copper [24].

It is not trivial to obtain a fundamental understanding of the irradiation hardening behavior of ion-irradiated metals through cross-sectional nanoindentation as the hardening behavior is closely related to the average defect density in the formed plasticity affected region, which can be a function of the defect density distribution within the irradiated region, indentation depth, and the distance between the indenter tip and irradiated sample surface. Therefore, both irradiation conditions and the location of indenter tip could affect the measured hardness. Unfortunately, to the authors' knowledge, there is no such related theoretical model proposed for the analysis of irradiation hardening of ion-irradiated samples by cross-sectional nanoindentation.

In this work, a hardening model is developed for the cross-sectional nanoindentation of ion-irradiated materials, which is mainly based on the derivation of an average defect density that is a function of defect density distribution in the irradiated layer, indentation depth, and the distance between the indenter tip and irradiated sample surface. The proposed model could help understand how the distribution of irradiation-induced defects and indenter position affect the irradiation hardening behavior, and assist the analysis and verification of obtained experimental data. In Section 2, a detailed derivation of the hardening model is described for cross-sectional nanoindentation. Following, the parameterization process of the proposed model is presented in Section 3. In Section 4, numerical results obtained from the parameterized model are compared with corresponding experimental data. To close up, some conclusions are summarized in Section 5.

2. Hardening model for cross-sectional nanoindentation

In order to obtain the hardness expression for ion-irradiated materials with cross-sectional nanoindentation, both the hardening contribution of dislocations and defects should be taken into account, i.e.

$$H_{\mathrm{irr}}^{\mathrm{cro}}(x,h)=H_{\mathrm{uni}}+\Delta H_{\mathrm{d}}^{\mathrm{ion}}(x,h), \tag{1}$$

where x and h are respectively the distance from the indenter tip to irradiated sample surface and indentation depth. The first term on the right-hand side of Eq. (1) is the hardening contribution of dislocations within the plasticity affected region. Following the theoretical model proposed by Nix and Gao [20], i.e. $H_{\mathrm{uni}}=H_0\sqrt{1+\overline{h}^{*}/h}$ is the indentation depth dependent hardness of unirradiated materials that can effectively characterize the indentation size effect induced by geometrically necessary dislocations. Here, H_0 and $\overline{h}^{*}$ are respectively the hardness of bulk materials and characteristic length.

As a comparison, the hardening model from Kasada et al. [25] also utilizes the Nix-Gao approach [20], but adds the irradiation-induced defects to the pre-existing defects, and the distribution of defects in the ion-irradiated region is taken to be homogeneous. It is, of course, easy to apply Kasada's model to high dose irradiated materials. Unfortunately, this model is difficulty to apply on the materials containing a defect gradient in the irradiated region despite being physically correct and in agreement with Nix-Gao's model [20]. In this work, we introduce a different approach deviating from the Nix-Gao's physical meaning of H_0, but allowing to provide an analytical solution to the problem with a good match with experimental data. The deviation of H_0 is found to be appropriate due to the fact that one simply cannot obtain a representative hardness number at the infinite depth for ion-irradiated materials with uniformly distributed defects in the irradiated region.

With irradiation effect, irradiation-induced defects could lead to the additional increase of hardness, i.e. $\Delta H_{\mathrm{d}}^{\mathrm{ion}}$. Following the framework of Orowan theory [26], irradiation hardening comes from the impediment of sliding dislocations by sessile irradiation-