Journal of Nuclear Materials 482 (2016) 93–98

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Effect of dislocations on helium retention in deformed pure iron

Y.H. Gong [a], X.Z. Cao [a,*], S.X. Jin [a], E.Y. Lu [a], Y.C. Hu [a], T. Zhu [a], P. Kuang [a], Q. Xu [b], B.Y. Wang [a]

[a] Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100049, China
[b] Research Reactor Institute, Kyoto University, Osaka 590-0494, Japan

HIGHLIGHTS

- Helium diffusion and desorption behavior was researched by DBS and TDS measurements.
- Helium atoms combining with dislocation change the distribution of electron density.
- Helium atoms combined with jogs were more stable than dislocations.
- Dislocations promote helium desorption from He_nV_m clusters (n/m = 1.8).

ARTICLE INFO

Article history:
Received 24 June 2016
Received in revised form
30 September 2016
Accepted 6 October 2016
Available online 8 October 2016

Keywords:
Helium diffusion
Dislocation
Thermal desorption
Positron annihilation

ABSTRACT

The effects of dislocations created by deformation on helium retention in pure iron, including the helium atoms diffusion along the dislocation line and desorption from dislocation trapping sites, were investigated. The dislocation defect was introduced in specimens by cold-rolling, and then 5 keV helium ions were implanted into the deformed specimens. Slow positron beam technology and thermal desorption spectroscopy were used to investigate the evolution of dislocation defects and the desorption behavior of helium atoms under influence of dislocation. The behaviors of S-E, W-E and S-W plots indicate clearly that lots of helium atoms remain in the deformed specimen and helium atoms combining with dislocation change the distribution of electron density. The helium desorption plot indicates that dislocation accelerates helium desorption at 293 K-600 K and facilitates helium dissociation from He_nV_m ($n/m = 1.8$) cluster.

1. Introduction

Irradiation hardening, swelling and helium embrittlement are the main problems in first-wall materials of nuclear fusion reactor. These problems have relations with helium atoms in the material, and helium atoms were generated almost due to the (n, α) nuclear reaction [1,2]. It is well known that the helium atoms have low migration energy and low solubility in iron-based alloy, and have strong interaction with the micro-defects, such as vacancies, dislocations, grain boundaries and vacancy clusters. As a result, the helium atoms were easily trapped by the micro-defects and aggregated to form He_nV_m clusters nearby the micro-defects, which increase the ductile brittle transition temperature of materials. Eventually, helium embrittlement is considered as one of the menaces to the reactor safety. Many researchers have been committed to focus on helium embrittlement, helium bubbles formation and materials swelling phenomenon [3–6], and also paid attention to effects of micro-defect on helium atoms migration. Noteworthy, helium atoms, as the impurity atoms, can easily diffuse along the dislocation line direction [7–9]. Xu et al. reported that the dislocation-trapped helium could affect the mechanical properties of pure iron, and the deformation dislocations can enhance the helium diffusion along dislocation line [10]. The interaction between dislocations and helium atoms has attracting researchers, which is benefit for understanding helium aggregation, nucleation and growth mechanism nearby the dislocation.

The reduced activation ferritic/martensitic steels are one of the candidate structural materials for fusion reactor. The nuclear structural materials including Eurofer 97, CLAM, F82H and Fe9Cr2WVTa, are iron based alloy. In order to investigate the interaction between dislocation and helium, pure iron which has similar lattice structure to aforesaid nuclear structural materials

* Corresponding author.
E-mail address: caoxzh@ihep.ac.cn (X.Z. Cao).

http://dx.doi.org/10.1016/j.jnucmat.2016.10.014

was used in present study.

Positron annihilation Doppler broadening spectroscopy (DBS) is usually used to characterize micro-defect evolution because the positrons were easily trapped in micro-defects and annihilated with electron around micro-defects. The results of DBS can be used to analyze the change information of defects [11]. Thermal desorption spectrum (TDS) is an effective experimental methods to observe the helium desorption behavior in materials. Helium desorption activation energy with the temporal temperature can be estimated from the relationship between the helium desorption rate and heating time. In this paper, the interaction between dislocation and helium atoms was analyzed by DBS and TDS.

2. Experimental procedure

Polycrystalline iron was used and the iron (purity: 99.99%) was cut to 10 × 10 mm with a thickness of 0.3 mm. All specimens were annealed at 1023 K for 2 h in vacuum (~10^{-5} Pa). In order to introduce dislocation defects, the well-annealed specimens were rolled 10% thickness reduction by cold rolling machine. And then, the specimens were annealed at 673 K for 1 h in vacuum (~10^{-5} Pa) to eliminate the vacancy type defects [12], and reserve the dislocation type defects in the matrix, the transmission electron microscope (TEM) images of the well-annealed specimen, before and after 673 K annealed of deformed specimen were shown in Fig. 1. Before the helium implantation, all specimens were chemically polished to clean the surface. Helium implantation was carried out using 5 keV He^+ with a flux of 5.0 × 10^{17} He^+/m^2s at room temperature, and the total irradiation dose was 1 × 10^{20} He^+/m^2.

DBS of slow positron beam was carried out to characterize the defect depth distribution. Positrons are generated by 45 mCi ^{22}Na (2014) radiation source and moderated by solid Ne moderator. The single HPGe detector was used in DBS measurement to detect the emitted γ rays by positron annihilation. The monoenergetic positrons were implanted into the specimens with the energy varied from 0.1 keV to 20 keV. S and W parameters were usually used to describe the momentum distribution of annihilated electron. S parameter is defined as the ratios of the central area counts for low Doppler-shift (510.2 keV-511.8 keV) and W parameter is defined as the two flanks regions counts for high Doppler-shift (504.2 keV-508.4 keV and 513.6 keV-517.8 keV) in the DB spectra to the total counts. The total counts of the DB spectrum is accumulated to 2.0 × 10^6 to reduce the statistical error.

TDS was widely used for studying helium desorption processes as a usual experimental tool [13,14]. In this experiment, the temperature is ramping with 1 K/s from 293 K to 1400 K by infrared radiation heater. During heating, helium was monitored by a quadrupole mass analyzer in a vacuum of 10^{-6} Pa.

3. Results

The microstructure of dislocations were shown in Fig. 1. The well-annealed specimen has little dislocation and the deformed specimen has lots of dislocation in the dark area. After 673 K annealed for deformed specimen, there are still lots of dislocation. In the positron annihilation lifetime test, the positron lifetime of well-annealed specimen was 105.9 ps? The long lifetime τ_2 was changed from 150 ps (deformed) to 117.3 ps(673 K annealed). It demonstrates that deformed specimen annealed at 673 K which can eliminate the vacancy type defects and reserve the dislocation type defects in the matrix.

The distribution of implanted helium and produced vacancies were simulated by SRIM, as shown in Fig. 2. The displacement energy of Fe was set to 40 eV [15]. SRIM calculation demonstrates that the approximate helium maximum penetration depth is only 84 nm. The vacancy peak and helium distribution peak locates at ~12 nm and ~23 nm, respectively.

S and W parameters as a function of incident positron energy are shown in Fig. 3, and the positron implantation depth is estimated using the following experimental equation [16]. The calculated results are also shown on the upper horizontal scale in Fig. 3.

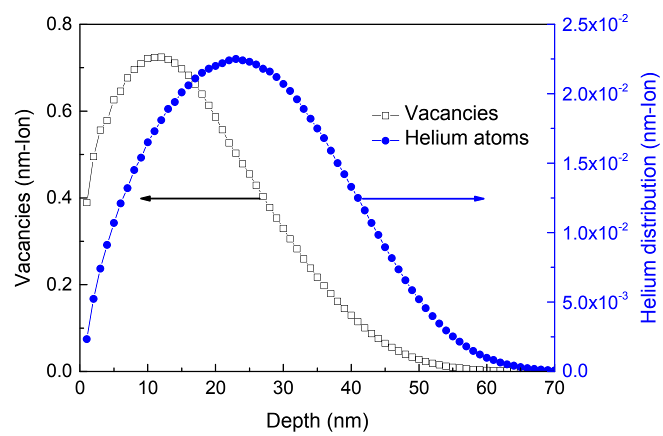

Fig. 2. Profiles of vacancies and helium atoms distribution in iron implanted with 5 keV He^+ calculated with SRIM2013.

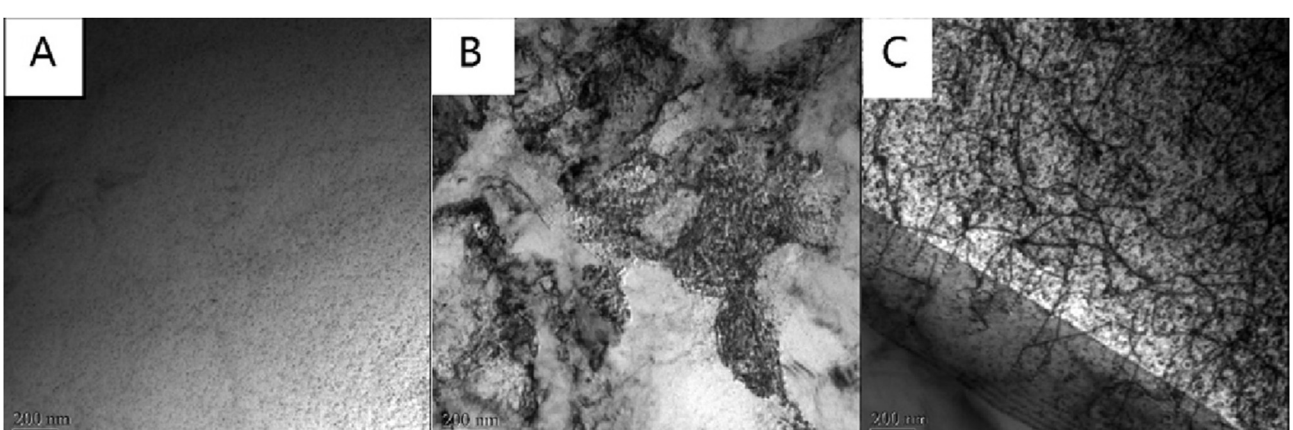

Fig. 1. TEM result for microstructure of dislocation. (a): well-annealed specimen, (b): deformed specimen, (c): deformed specimen and annealed at 673 K for 1 h.