BNL--36287

DE85 010235

BNL 36287

CONF-850169--7

9-C 2146

5936000

REDUCTION OF THE TRAPPING OF POSITRONS IN DISLOCATED SINGLE CRYSTALS OF IRON WHEN CHARGED WITH HYDROGEN

Yong-Ki Park*, James T. Waber*
C. Lewis Snead, Jr.**

* Department of Materials Science and Engineering, Northwestern University, Evanston, Il. 60201, U.S.A.

** Metallurgy and Materials Science Division, Brookhaven National Lab., Upton, N.Y. 11973, U.S.A.

The positron annihilation measurement was carried out with the pure iron single crystals deformed in various ways before and after hydrogen permeation. The positron trapping intensity was reduced more in the screw dislocation than in the edge dislocation by hydrogen charging. The trap occupancy by hydrogen was very close to the fraction of the reduction in positron trapping intensity.

INTRODUCTION

It has been known that a positron is trapped by dislocations in the deformed metals.[1,2] Recently it was found that the number of edge and screw dislocations can be determined separately by measuring the positron lifetime spectrum in the deformed iron single crystals.[3]

One of the applications of this non-destructive microstructure characterizing method is the study of hydrogen trapping phenomena in metals. This study is important to understand and control the hydrogen embrittlement in metals.

Hydrogen permeation method is one of the most frequently used techniques in the study of hydrogen trapping. The major diffuculty in this method is that there is no conclusive identification of trapping site.

Positron annihilation method is one of the complementary techniques to solve the above difficulty in the hydrogen permeation method. Hydrogen in metals is known as a screened proton[4]. Since it has the same charge as the positron, a hydrogen which is trapped in the defect will repel a positron and will reduce the probability of a positron trapping in the same type of defects.

In the present research, low concentration of hydrogen was charged in the pure iron single crystals. The positron lifetime and Doppler broadening was measured in the hydrogen-charged specimens in order to identify the trap site and determine the trap density.

EXPERIMENT

Pure iron single-crystal specimens were prepared and purified by circulating ZrH_x purification system. They were deformed by various ways such as bending, cold rolling at room temperature, and elongating at 200 K.

Hydrogen permeation measurement was carried out using the Devanathan and Stachurski type electrochemical cell.[5] Experimental details are decribed in elsewhere.[6]

Hydrogen - charged specimens were quenched in the liquid nitrogen. Positron annihilation lifetime and Doppler broadening line-shape were measured concomitantly with the specimens before and after hydrogen charging. In order to keep the hydrogen in the specimen, the positron annihilation measurement was carried out with the specimens in the liquid nitrogen Dewar.

RESULTS AND DISCUSSION

Figure 1 shows the total positron trapping intensity of trap site in hydrogen-charged and uncharged iron crystals which were deformed in tension at 200 K. The total positron trapping intensity was decreased about 20% by small amounts of hydrogen (ca. 0.3 atomic ppm).

It was found that the screw and edge dislocations could be determined

DISCLAIMER

separately by the measurement of positron lifetime.[5] When hydrogen was charged in low temperature deformed iron the positron trapping rate decreased both in screw and edge dislocations. Figure 2 shows the positron trapping rate in screw and edge dislocations before and after hydrogen charging.

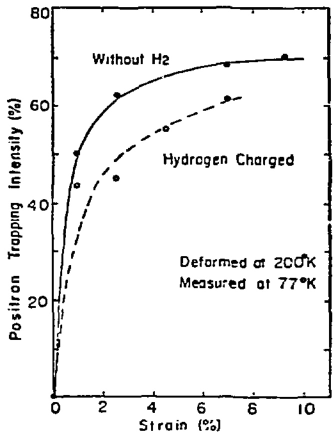

Figure 1. Total positron trapping intensity at trap site in hydrogen charged and uncharged iron single crystals which were deformed in tension at 200 K.

The hydrogen effect was larger in a screw dislocation than in a edge dislocation. This is probably because the binding of positrons is weaker in a screw dislocation than in an edge dislocation since the dilatatonal field around a screw dislocation is smaller than that around an edge dislocation.[7] Hence, the modification of the local strain field and the electron density profile by the trapped hydrogen could be more effective in reducing the positron trapping in the screw dislocation than in the edge dislocation.

The hydrogen trap density was determined from the hydrogen permeation experiments using several different analyzing models.[8-10] This results were compared with the dislocation density determined by positron annihilation and by transmission electron microscopy.

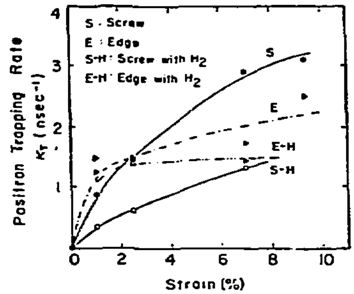

Figure 2. Positron trapping rate at screw and edge dislocations in hydrogen-charged and uncharged iron single crystals which were deformed in tension at 200 K.

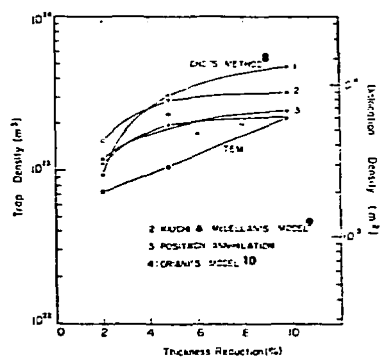

Figure 3. Hydrogen trap density in room-temperature cold-rolled iron single crystals.

The dislocation density and hydrogen trap density was correlated by assuming that there is one trapping site per unit cell length along the dislocation line.[8] The agreement between three independent measurement was good as shown in Figure 3.

The reduction of the positron trapping intensity of the trap in the hydrogen-charged iron could be anticipated because when a hydrogen atom which has the same charge as a positron occupies the trapping site it will repel a positron.

Table 1. Comparison of the trap occupancy by hydrogen determined by hydrogen permeation method and positron annihilation technique in iron single crystals deformed in tension at 200 K.

True strain (%)	Trap density N_T, m^{-3}	Average Hydrogen concentration C_{av}, H_{atoms}/m^3	C_{av}/N_T	ΔI $=(I_D-I_{DH})/I_D$
1	8.5×10^{22}	1.44×10^{22}	0.17	0.14
2.5	1.64×10^{23}	2.4[illegible] $\times 10^{22}$	0.15	0.27
4.4	2.78×10^{23}	2.66×10^{22}	0.10	0.14
6.9	4.14×10^{23}	1.89×10^{22}	0.05	0.11

*I_D: positron trapping intensity in deformed specimen (without hydrogen)

I_{DH}: positron trapping intensity in deformed and hydrogen-charged specimen

In Table 1 the quantitative comparison between the trap occupancy by hydrogen atoms and the reduction of the positron trapping intensity was made. In this table the trap occupancy by hydrogen was defined by the ratio between the average hydrogen concentration and hydrogen trap density which were determined from hydrogen permeation experiment. The equivalent parameter in terms of positron trapping intensity can be defined by

$$\Delta I = (I_D - I_{DH})/I_D$$

where I_D and I_{DH} is the positron trapping intensity in a specimen deformed but not charged and that in a specimen deformed and charged, respectively. This parameter depends on the hydrogen occupancy in the defect and also on the intensity of repulsion of positron by a trapped hydrogen atom.

According to the theoretical calculation by Jena *et al.*[11] the electron charge pileup around the proton is localized. Therefore it is anticipated that hydrogen trapped in the defect will reduce the positron trapping intensity by about the same fraction as the hydrogen occupancy.

As seen in Table 1, hydrogen occupancy determined by hydrogen permeation method is actually very close to the ΔI as anticipated above. Further, the hydrogen occupancy along the dislocation obtained in the present research is also in good agreement with the reported value of Gibala[12], namely about 0.25. This value was obtained from his internal – friction measurements.

ACKNOWLEDGEMENTS

This research was supported in part by the U.S. Army Research Office. DAAG 29 81-K-0153 and DOE Div. of Mat. Sci., Office of Basic Energy Sciences, under contract DE-AC 02-76-CH 00016.

REFERENCES

1. M. Doyama and R.M.J. Cotterill, *Proc. 5th Intl. Conf. Positron Annihilation*, Japan, 1979, p.89.
2. S. C. Sharma, R. M. Johnson, and L.M. Diana, *"Novel NDE Methods for Materials"*, ed. Bhakta B. Rath, Met. Soc. of AIME, 1983, p.45.
3. Yong-Ki Park, James T. Waber, C. Lewis Snead, Jr., M. Meshii and C. G. Park, *submitted to Phys. Rev.*
4. R. A. Oriani, *loc. cit.*, p.32.
5. M. A. V. Devanathan and Z. Stachurski, *Proc. Roy. Soc.*, 270, 90(1962).
6. Yong-Ki Park, Ph.D. thesis, Northwestern University, 1985.
7. Y. T. Chou, *Acta Met.*, 13, 251(1965).
8. J.Y. Choi, *Met. Trans.* 1, 911(1970).
9. K. Kiuch and R. B. McLellan, *Acta Met.*, 31, 961(1983).
10. R. A. Oriani, *Acta Met.*, 18, 147(1970).
11. P. Jena, M. J. Ponnambalam, and M. Manninen, *Phys. Rev.*, B, 24, 2884 (1981).
12. R. Gibala, *Trans. Met. Soc. AIME*, 239, 1574(1967).