Materials Science Forum Vol. 607 (2009) pp 266-268
online at http://www.scientific.net
© (2009) Trans Tech Publications, Switzerland

Application of positron microprobe for nuclear materials

M. Maekawa[1], A. Kawasuso[1], T. Hirade[2] and Y. Miwa[2]

[1]Advanced Science Research Center, Japan Atomic Energy Agency
Watanuki 1233, Takasaki, Gunma Japan
[2]Nuclear Science and Engineering Directorate, Japan Atomic Energy Agency
Tokai-mura 2-4, Naka-gun, Ibaraki, Japan
maekawa.masaki@jaea.go.jp

Keywords: Positron microbeam, Stress corrosion cracking, stainless steel, vacancy defects

Abstract. We have developed a positron microbeam using magnetic lenses based on the commercial scanning electron microscope. The minimum beam diameter was 1.9 μm on target. Two-dimensional image of S parameter was successfully obtained. Using this apparatus, S parameter distribution around the crack tip introduced by a stress corrosion cracking of a stainless steel was obtained. S parameter increases at the further region of the tip of the crack. This shows that vacancy type defects may be generated as crack precursor.

Introduction

Stainless steel is an excellent material in corrosion resistance and widely used as nuclear materials. Corrosion resistance of stainless steel is dependent on a tough oxidization film which is formed by chromium distributed in stainless steel. However, by the inappropriate heat treatment, chromium deposit and oxidization film is not formed. When the stress is loaded in the corrosion environment to such a material, stress corrosion cracking (SCC) will be started. Although this is critical issue to secure nuclear power plants, it has been not fully clarified due to its complicated reaction process. Especially, an understanding of crack progress is important.

Employing a positron microbeam, spatial distribution of positron annihilation near subsurface region can be obtained [1,2]. For instance, it is possible to investigate the relation between vacancy-type defects and crack progress. In this work, we developed a positron microbeam by fabricating a small source, a solid neon moderator, and magnetic lenses. Using this, we attempted to evaluate of the crack tip introduced by SCC of stainless steel for the nuclear materials.

Positron microbeam in JAEA

Figure 1 schematically shows the apparatus. To reduce the initial beam emittance, we newly developed a sealed source having the small active area. Sodium-22 of 55 MBq was deposited into the 2 mm-diameter hole and sealed by a capsule with a Ti window of 5 μm thick. The source is mounted on the top of the cold head and cooled down to 4 K. Solid neon film grown on the source window is used as a moderator [3]. Slow positron beam is generated by the electrostatic lenses [4] and accelerated by the electrostatic potential difference between the source and the anode. The beam energy used in this work is 20 keV. The positron beam is transported by magnetic lenses and 90° direction deflector. The positron beam is finally focused by the optics of a commercial scanning electron microscope. The beam is scanned in two-dimensionally by moving the sample. The Doppler-broadening of annihilation radiation is measured using a Ge detector and characterized by S parameters, which are defined as the peak intensities.

By scanning the beam across a sharp metal knife-edge on a silicon wafer, the beam diameter was measured. The result is shown in Fig.2. The minimum diameter was determined to 1.9 μm. Spatial resolution was evaluated by scanning the test pattern. Figure 3(a) shows the optical image of the test pattern. Three deferent sizes of pattern are scanned. The line widths of each character are 12, 25 and

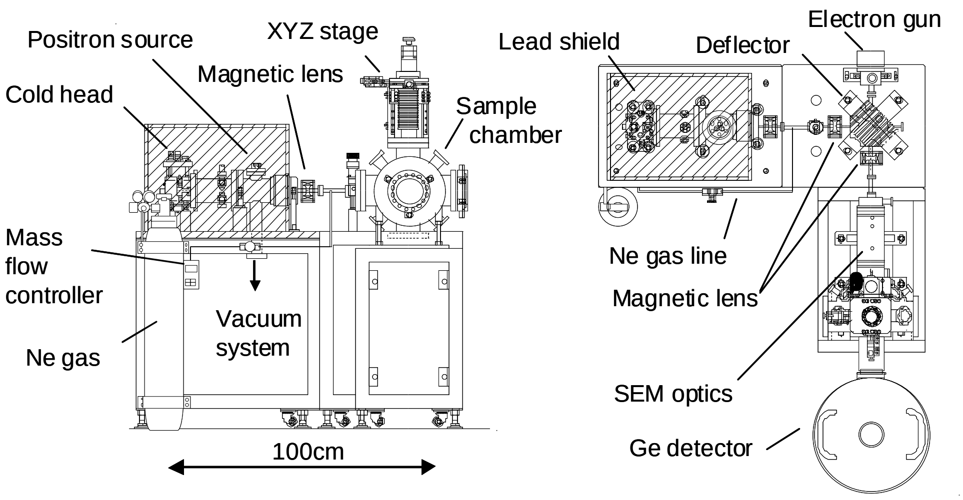

Fig. 1. Schematic drawing of the positron microbeam apparatus. The left picture is side view and the right picture is top view.

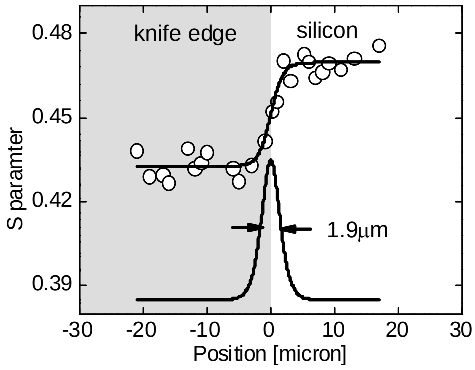

Fig. 2. The result of the measurement of the beam diameter of the positron microbeam by the scanning of the knife edge.

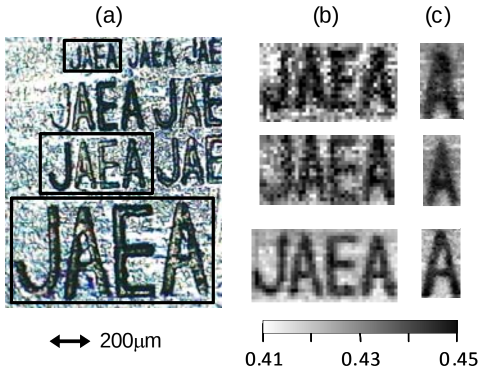

Fig. 3. Optical image of the test pattern and S parameter distribution.

50μm. Fig.3 (b) is the S parameter distribution of the rectangular area on (a). Fig.3(c) is the result of the high resolution scan (double density) of the character 'A'. Even if it is the minimum character size, the clear contrast of S parameter is appeared. This spatial resolution is enough high to investigate defect properties in small region, such as tip of cracks.

Application for the nuclear materials

The evaluation of a crack tip of stress corrosion cracking (SCC) on the stainless steel was carried out. This specimen was fabricated from heat-treated (650 ^{o}C×15 h + water-cooling) sensitized SUS304 stainless steel. Prior to SCC test, the specimen was pre-cracked in the air at room temperature by an electric discharge machine and subsequently fatigue. After that, the specimen was put into simulated boiling water reactors (BWR) water with 8 ppm dissolved oxygen concentration at a temperature of 288 ^{o}C. External load was applied to the specimen for crack growth [5]. Figure 4(a) shows the optical image of this sample at the tip of a crack. The small line with Y shape is the crack generated by the SCC process. On this region, two-dimensional scan of positron microbeam was performed to obtain the distribution of the S parameter. Scan area is 2700×1900 μm and scan step is 100 μm. The result is shown in Fig. 4(b). The optical image is superimposed at the same region. S parameter increases at the further region of the tip of the crack. It seems that the vacancy type defects are introduced before crack grows. This result is in agreement with the destruction mechanism usually understood [6]. Vacancy type defects may be generated as crack precursor. This defect might not be a vacancy cluster because of the small changes of S parameter. The electron momentum distribution of the high S parameter region (inside the circle in Fig.4 (b)) is measured and compared with the defect model calculation by the first principal calculation [7]. The result is shown in Fig. 5. These momentum distribution are expressed as a ratio curve divided by the spectrum of the bulk. The calculation result of the monovacancy (V1) reproduces an experimental result. This result shows that size of defects introduced in advance of crack progress is small with the size of a monovacancy. These defects are undetectable in the observation by the optical microscope and the electron microscope. Using positron microbeam, it is considered that the positron measurements give information which cannot be found by conventional measurements.

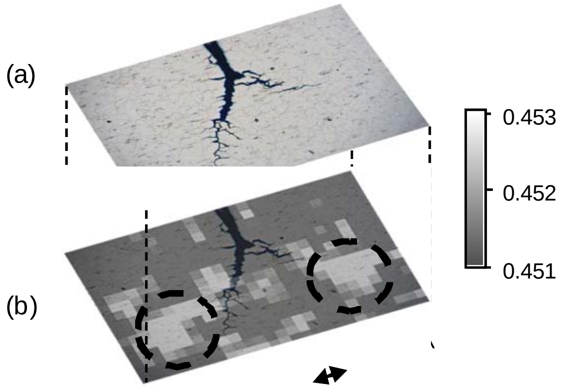

Fig. 4. (a) Optical image of the SCC on the stainless steel. (b) The spatial distribution of S parameter.

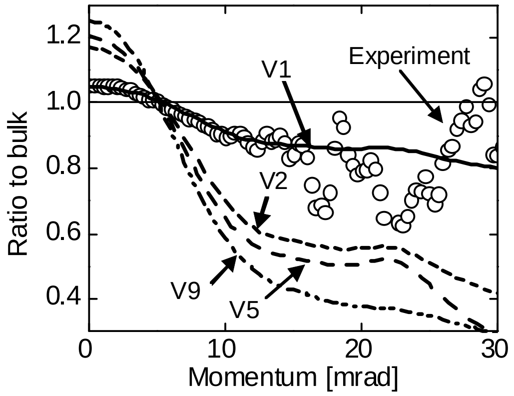

Fig. 5. Momentum distribution for the defective area of Fig.4(b) and calculated ratio curves for the monovacancy (V1), divacancy (V2), and vacancy clusters (V5 and V9).

Summary

We developed a new positron microbeam. We succeeded to focus the positron beam to 1.9 μm. The S parameter spatial distribution around crack tip introduced by the SCC in stainless steel was obtained. Vacancy type defects may be generated as crack precursor. The positron microbeam can supply the detail information about vacancy type defects which cannot be found by conventional measurements.

Acknowledgements

Present study is the result of "Innovative Nuclear Research and Development Program" entrusted to "Japan Atomic Energy Agency (JAEA)" by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

References

[1] A. David, G. Kögel, P. Sperr and W. Triftshäuser, Phys. Rev. Lett. 87, 067402 (2001).
[2] U. Männig, K. Bennewitz, H. Bihr, M. Haaks, W. Sigle,k C. Zamponi and K. Maier, Appl. Surf. Sci. 149, 227 (1999).
[3] A. P. Mills, Jr. and E. M. Gullikson, Appl. Phys. Lett. 49, 1121 (1986).
[4] A. Kawasuso, T. Ishimoto, M. Maekawa, Y. Fukaya, K. Hayashi and A. Ichimiya, Rev. Sci. Inst. 75, 4585 (2004).
[5] R. S. Yu, M. Maekawa, Y. Miwa, T. Hirade, A. Nishimura, and A. Kawasuso, Phys. Stat. Sol. (c) 4, 3577 (2007).
[6] G. Kögel, W. Egger, S. Rödling and H. -J. Gudladt, Mater. Sci. Forum445-446, 126 (2004).
[7] A. Kawasuso, M. Yoshikawa, H. Itoh, T. Chiba, T. Higuchi, K. Betsuyaku, F. Redmann, and R. Krause-Rehberg, Phys. Rev. B 72, 045204 (2005).

Positron and Positronium Chemistry
doi:10.4028/0-87849-348-4
Application of Positron Microprobe for Nuclear Materials
doi:10.4028/0-87849-348-4.266