Development and application of positron microprobe

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We have developed a positron microbeam using magnetic lenses based on the commercial scanning electron microscope (SEM). A slow positron beam was generated using a handmade ^{22}Na source and a solid neon moderator. The minimum beam diameter was 3.9 μm on target. Two-dimensional image of S parameter was successfully obtained. By introducing a beam pulsing section, positron lifetime measurement is also available.

Key words: Positron microbeam, Scanning positron microscope, Solid neon moderator, Beam pulsing

1. INTRODUCTION

Positron annihilation spectroscopy (PAS) is a powerful tool to detect open volume defects in solids. Using a slow positron beam, a depth profiling of defects with typical depth of several microns is possible. In addition, employing a positron microbeam spatial distribution of positron annihilation near subsurface region can be obtained. For instance, it is possible to investigate the relation between vacancy-type defects and crack progress of nuclear materials. Several positron microbeams have already been developed [1-5]. Munich group and Bonn group successfully formed a positron microbeam with diameters of 2 μm and 20 μm. We also attempted to form the positron microbeam using a scanning electron microscope (SEM) optics [6-8]. However, the obtained beam diameter was 80 μm due to the large initial beam diameter (4 mm). In this work, we developed a positron microbeam by fabricating a small source, a solid neon moderator, magnetic lenses and a SEM optics. In addition, a beam pulsing system was also developed which allows the positron lifetime spectroscopy measurement.

2. CONSTRUCTION OF POSITRON MICROEAM

2.1 Slow positron generation system

Figure 1 schematically shows the apparatus. To make microbeam effectively, the initial beam emittance should be reduced. This is realized by reducing positron generating area. The active diameter of commercial source is typically 4 mm. This is not suitable for reduction of the emittance. We newly developed a sealed source as shown in Fig. 2 (a). Sodium-22 of 55 MBq was deposited into the 2 mm-diameter hole on a tungsten-copper block and sealed by a capsule with a Ti

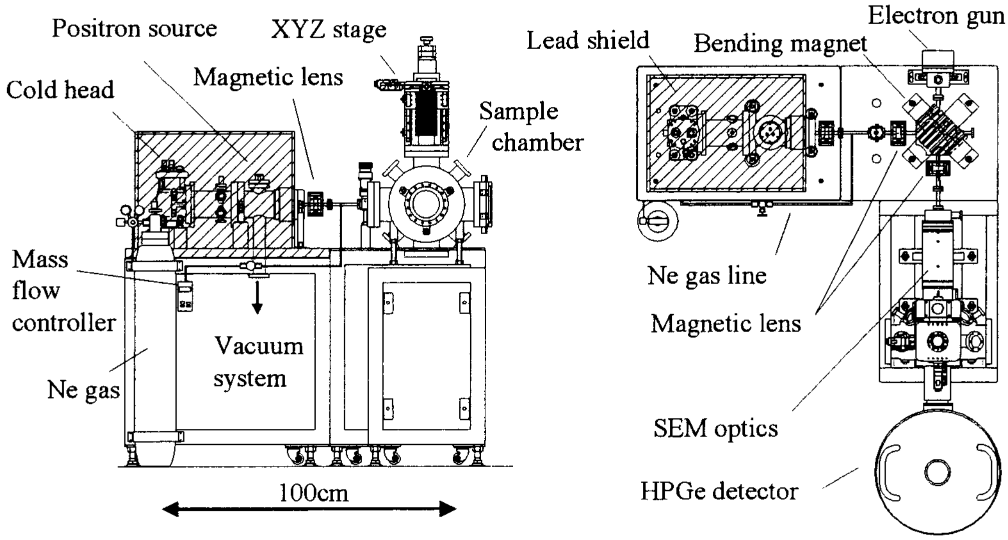

Fig. 1. Schematic drawing of the positron microbeam apparatus. The left picture is side view and the right picture is top view.

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window of 5 μm thick. Figure 2(b) shows the schematic drawing of the positron gun. The source is mounted on the top of the cold head with the radiation shield, and cooled down to 4 K. As a moderator, solid neon film is grown on the source window by introducing neon gas [9]. The flow rate is controlled by a mass flow controller. The base vacuum pressure is typically 6×10^{-6} Pa , and Ne gas deposited at a pressure of 4×10^{-4} Pa for about 80 minutes. Slow positron beam is generated by the immersion lens system composed of an extractor, a wehnelt, a Soa and an anode [6]. The slow positron beam is further accelerated by the electrostatic potential difference between the source (moderator) and the anode. The maximum beam energy is 30keV.

2.2 Transportation and focusing systems
The positron beam is transported by magnetic lenses and deflected to the 90° direction with a magnetic prism to avoid fast positrons and gamma-ray background. The positron beam is finally focused by the SEM optics (Topcon SM-300). An adjustable collimator (0.5 mm diameter) can be installed in front of the objective lens. Electron beam which is generated by a electron gun equipped on a straight port of the magnetic prism can be also focused. This allows normal SEM operation.

2.3 Scanning system
By moving the sample to the X-Y direction, the beam is scanned in two-dimensionally. The Doppler-broadening of annihilation radiation from the sample is measured using a Ge detector. The detector is placed behind the specimen to enhance the counting rate. The obtained Doppler broadening spectra are characterized by *S* parameters, which are defined as the peak intensities. *S* parameter tends to increase at open-volume type defects because of the narrowing of the Doppler spectrum due to the reduction of core electron annihilation rate which gives rise to a greater Doppler shift.

2.4 Pulsing system
Positron lifetime measurement can be realized by utilizing a pulsed positron beam. For this purpose, a

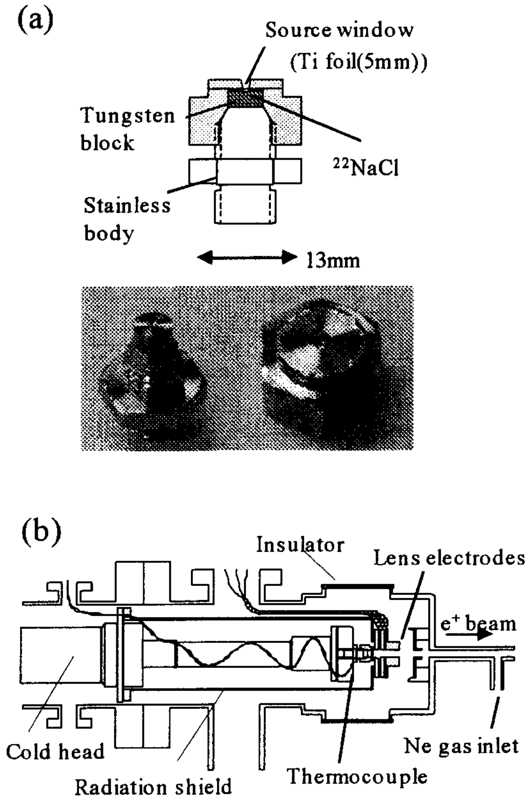

Fig. 2. Schematic drawings of (a) the positron source capsule and (b)the positron gun. The source is installed on the top of the cold head and cooled down to 4 K.

bunching unit [10, 11] is installed between the positron gun and the magnetic prism. The schematic of this pulsing system is shown in Fig. 3. In this mode, the positron beam with energy of 10 eV is generated and transported to the bunching unit in the continuous guiding magnetic field. This DC positron beam is first

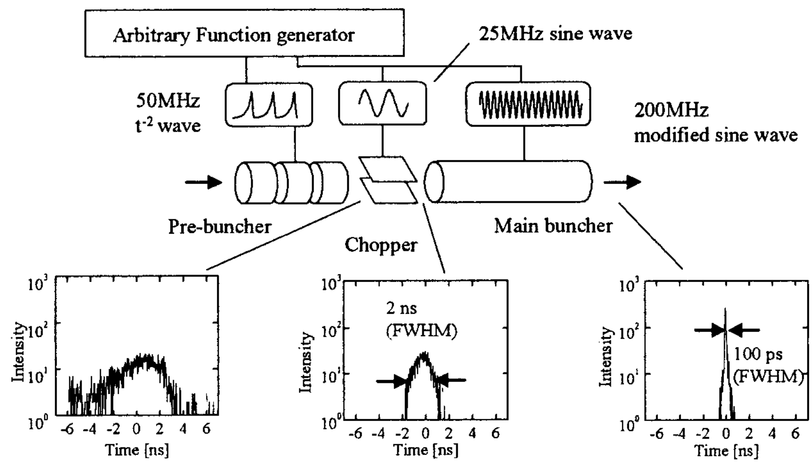

Fig. 3 The schematics of pulsing unit and results of numerical calculations of the behavior of pulsed beam. The pulsed beam of 100 ps (FWHM) is finally obtained.

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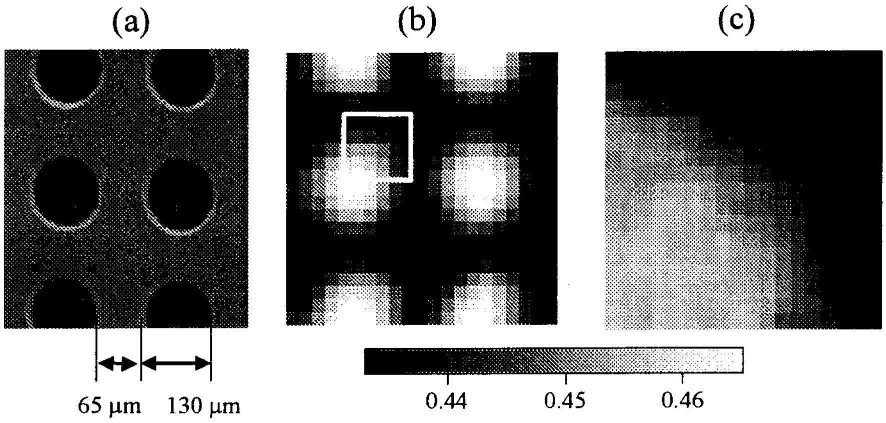

Fig. 4. (a) SEM image of the stainless mesh. (b) The spatial distribution of S parameter with the scan area of 400 × 400 μm. (c) The spatial distribution of S parameter with the scan area of 80 × 80 μm which corresponds to the square in (b).

bunched to several nanoseconds at the pre-buncher driven by a 50 MHz t^{-2}-wave. The beam in the unbunched phase is removed by the parallel-electronode type chopper driven by a 25 MHz sine wave. Final beam bunching is done by the resonator-type main buncher driven by the superimposed wave with a 200 MHz sign and its 3rd harmonics. Figure 3(b) shows the result of beam pulsing obtained by numerical calculation. The pulse width of 100 ps (full with at half maximum: FWHM) having high peak-to-background ratio is feasible. The pulsed beam is extracted from the guiding magnetic field by accelerating up to several keV and focusing by a magnetic lens. Subsequently, the pulsed beam is injected to the SEM optics [8].

3 PERFORMANCE TESTS

By scanning the beam across a sharp metal knife-edge on a silicon wafer, the beam diameter was measured. The minimum diameter was 12.5 μm without the collimator. When the collimator was used, the beam diameter was reduced to 3.9 μm, though the counting rate reduced to one fifth. Figure 4 shows the results of the two-dimensional scan of a stainless steel mesh. Fig.4 (a) is the SEM image of this mesh. The hole diameter is 130 μm and the interval is 65 μm. Fig.4 (b) shows the S parameter distribution of this mesh. The scan area is 400 × 400 μm and scan step is 20μm. Total measurement time was approximately 36 hours. The image of S parameter with high contrast was obtained. Fig. 4(c) shows the zoom image of the part of a hole indicated by the square in Fig. 4(b). The scan area is 80 × 80 μm and scan step is 5 μm. The mesh edge is appeared clearly. These results show that our positron microbeam is capable to study of small structure of the same dimension as the above mesh.

Figure 5 shows the time structure of the bunched positron beam at the end of buncher unit. The pulse width is determined to be 140 ps in FWHM. This is sufficient pulse width for the positron lifetime measurement.

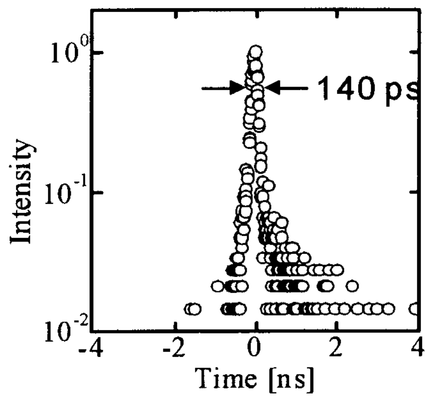

Fig. 5. The time structure of the bunched positron beam.

4 APPLICATION

As an application, the evaluation of a crack tip of stress corrosion cracking (SCC) on the stainless steel was carried out[12]. This specimen was fabricated from heat-treated (650 °C×15 h + water-cooling) SUS304 stainless steel. Prior to SCC test, the sensitized SUS304 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 °C. External load was applied to the specimen for crack growth. Figure 6 shows the optical image of this sample and the electron microscope image at the tip of a crack. The wide groove is formed by the electric discharge machine and the narrow groove is pre-crack by the fatigue. The small line with Y shape is the crack generated by the SCC process. On this SCC region, two-dimensional scan of positron microbeam was performed to obtain the distribution of the S parameter.

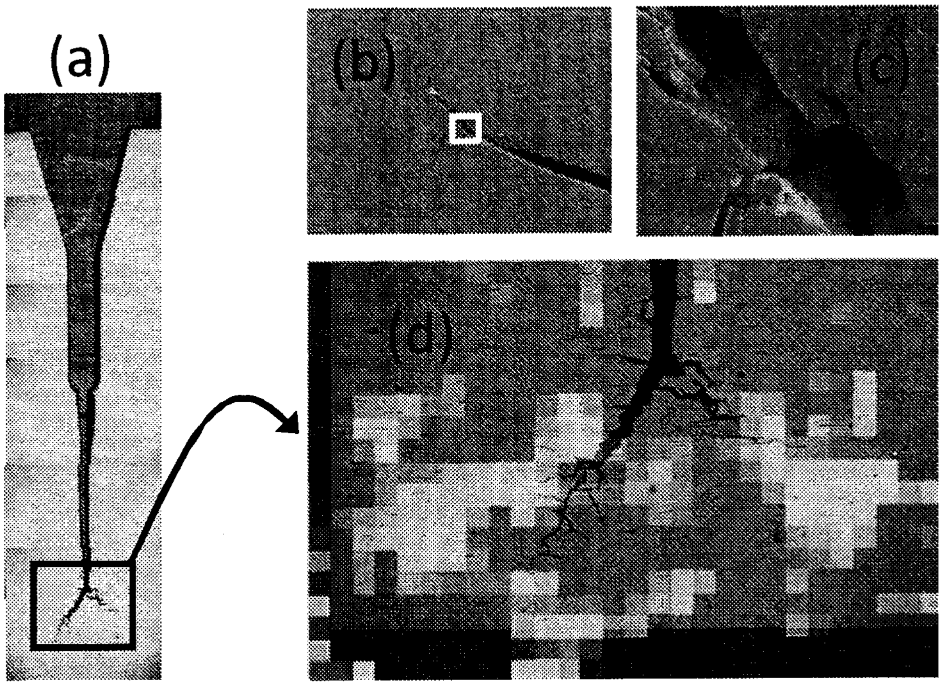

Fig. 6. (a) Optical image of the SCC on the stainless steel. (b) SEM image of the tip of crack. (c) SEM image of crack corresponding to the square in (b). (d) The spatial distribution of S parameter with the scan area of 2700 × 1900 μm which corresponds to the square in (a).

Scan area is 2700×1900 μm and scan step is 100 μm. Approximately 36000 counts are accumulated for each point. The result is shown in Fig. 6(d). The optical image is superimposed at the same region. S parameter does not increase on the crack, but 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 [13]. Vacancy type defects may be generated as crack precursor. It is considered that the positron measurements give information which cannot be found in optical measurement.

5 CONCLUSION

We developed a new positron microbeam. We succeeded to focus the positron beam to 3.9 μm. We also demonstrated the S parameter spatial mappings using a test mesh and SCC sample. The beam pulsing system, which can form the pulsed beam of 140 ps, was also developed.

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