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Evaluation of stainless steel under tensile stress using positron microbeam

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Abstract. Defect evolution near the crack tip in stainless steel (SUS 304) under tensile stress has been observed through *in-situ* positron microbeam measurements. It was found that the S parameter of the Doppler broadening of annihilation radiation spectrum increases near the crack tip. Upon the corrosion treatment, the crack progress occurred preferentially in the high S parameter region. The further increase of S parameter due to the complementary influences of tensile stress and corrosion environment was observed at the region away from the progressed crack. These results suggest excess that vacancy type defects play an important role in the stress corrosion cracking of stainless steel.

1. Introduction

Stainless steel is widely used as materials for nuclear power plants because of its excellent corrosion resistance. The corrosion resistance depends on toughness of chromium oxidization film formed on the surface. However, by inappropriate heat treatment, the oxidization film is degraded because of chromium precipitation. When tensile stress and corrosion environment exist under such condition, stress corrosion cracking (SCC) steel happens. Although the SCC is an important issue for the safety of nuclear power plants, its mechanism has not yet fully been clarified. To elucidate the SCC mechanism, it is important to understand structural change at crack tips in an atomic scale. Recently, fine cracks (tight cracks) of a few nano-meter are found at crack tips using an analytical transmission electron microscope (ATEM) [1]. The introduction of such tight cracks cannot be explained by the conventional theory [2, 3]. In the tight crack model, vacancy-type defects are thought to play an important role in the crack progress. Actually previous positron microbeam observation implies the introduction of vacancy-type defects around crack tips [4]. Li reports that the crack progress is affected by dislocations introduced by the plastic deformation from the *in-situ* TEM observations of SCC on the thin stainless film [5, 6].

In this work, we performed the positron microbeam observation of stainless film under the tensile stress during continuous corrosion. We will discuss the possibility of contribution of vacancy type defects to the crack progress.

2. Experimental

Figure 1 schematically shows the apparatus of our positron microbeam. Sodium-22 of 55 MBq was deposited into a 2 mm-diameter hole and sealed by a cap with a Ti window of 5 μm thick. The source is cooled down to 4 K and a solid neon film is grown on the source window as a moderator [7]. A slow positron beam with energy of 20 keV is generated by an electrostatic way [8]. The positron beam is focused onto sample surface using the commercial scanning electron microscope optics. The positron beam is laterally scanned by moving the sample. The Doppler-broadening of annihilation radiation (DBAR) is measured using a Ge detector. The obtained DBAR spectra are characterized by so-called S parameter, which is defined as the peak intensity of the spectrum.

The sample used in this study was SUS304 stainless steel of 20 μm thick. After the solution treatment at 1150 °C for 2 hours, for the sensitisation the sample was heated at 650 °C for 24 hours and finally quenched into water. The specimen was pre-cracked in the air at room temperature. The sample was fixed by a special holder made of stainless steel as shown in figure 2. Using the tension nuts, tensile stress of ~ 300 MPa was loaded to the sample. This stress is enough to introduce the SCC failure. The corrosion environment, treatments were carried out using boiled $MgCl_2$ solution along with the Japanese Industrial Standards method (JIS G0576). After the corrosion under the tensile stress, optical microscope observations and positron microbeam observations were alternatively conducted.

3. Results and discussions

First we show the results of the observations without the corrosion treatment. Figures 3(a), (b) and (c) shows the optical image around the pre-crack, the distribution of S parameter around the same crack

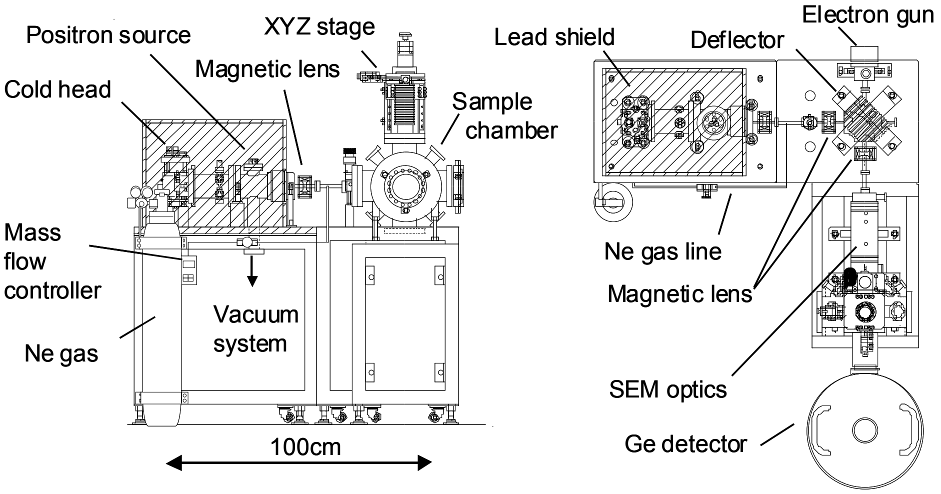

Figure 1. Schematics of the positron microbeam in JAEA

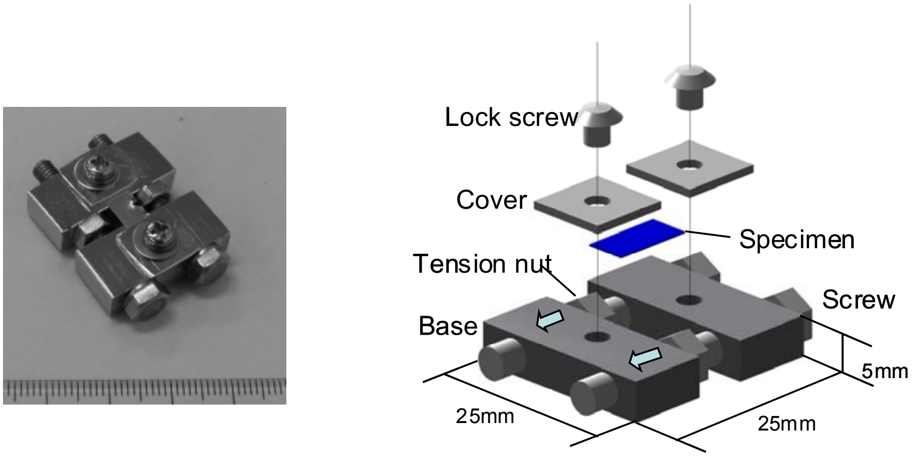

Figure 2. Schematics of the sample holder

tip and their superimposed image, respectively. Although no morphological change is observed by the optical microscope, S parameter increases at the region of ~200 µm away from the crack tip. The relative increase of S parameter to the bulk is ~1 %. Such a small increase of S parameter may be attributed to the vacancy-type defects, dislocations and/or volume expansion.

Figures 4(a), (b) and (c) show the similar images to figures 3(a), (b) and (c) obtained after exposuring the sample to the $MgCl_2$ solution for 24 hours. From the optical image, the crack progress is observed from the pre-cracked position denoted by the dotted line on figure 4(a). The crack seems to

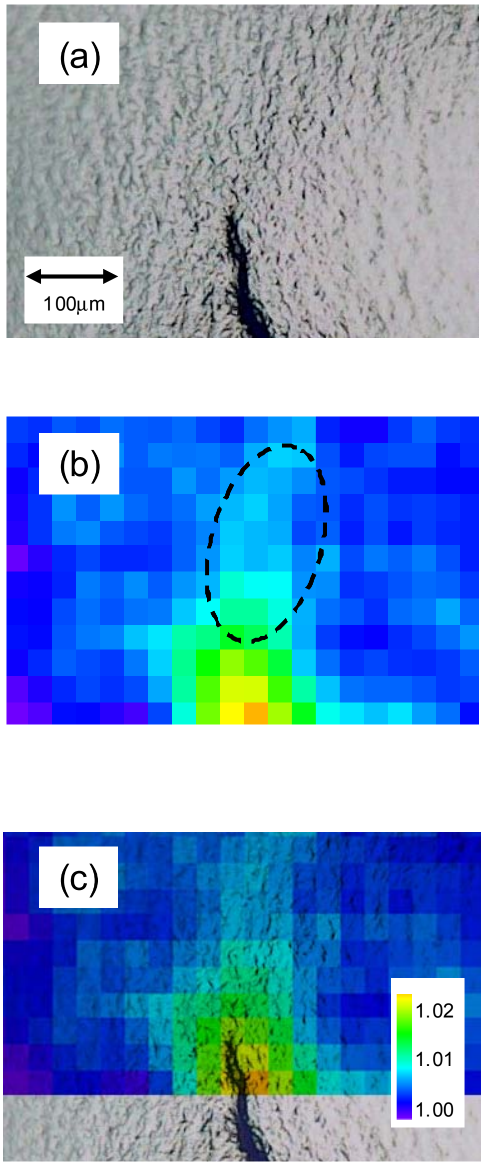

Figure 3. (a) Optical image of the pre-crack before the corrosion, (b) positron image around the crack tip and (c) superimposed image of (a) and (b).

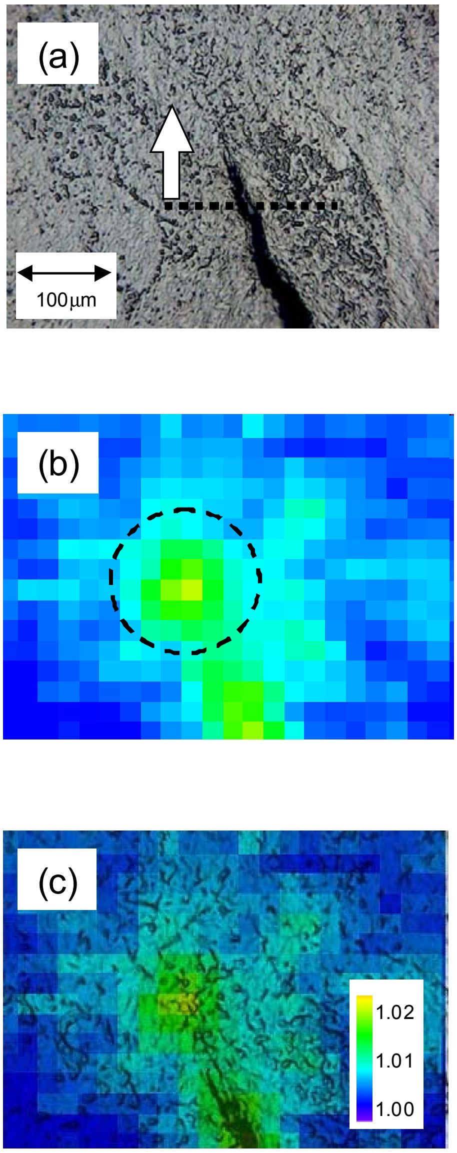

Figure 4. (a) Optical image of the SCC crack after the corrosion for 24 hours in boiled $MgCl_2$ solution, (b) distribution of S parameter around the crack tip and (c) superimposed image of (a) and (b). Dotted line on (a) shows the position of crack tip before corrosion.

progress preferentially to the direction where S parameter increased in figure 3(b). The further increase of S parameter (~2 %) is observed at the region away from the progressed crack tip. Such increase of S parameter is hardly attributed to the dislocations and/or volume expansion. This suggests that excess vacancy type defects are introduced due to the additional corrosion treatment. However, S parameter does not increase near the pre-crack region that is free from the tensile stress even after the corrosion treatment. From the above results, S parameter increases as the consequence of complementary influences due to tensile stress and corrosion environment.

In the tight crack model, some physical and chemical reasons, for example, preferential dissolutions at elementally atoms of stainless steel, dislocation movements, surface oxidation, stress at crack tip singularity and so on, are proposed as candidates for the source of vacancies. Our results seem to be consistent with the tight crack model that requires the introduction of vacancy defects.

4. Summary

We have carried out positron microbeam observations of stainless steel before and after the corrosion treatment. Applying the tensile stress, the increase of S parameter was observed near the crack tip. This suggests that vacancy-type defects are introduced by the stress concentration. The crack progress by the corrosion occurs preferentially in the region where vacancy type defects are introduced. Again, vacancy type defects are introduced near the progressed crack tip. By the further studies focusing on the vacancy type defects by the positron microbeam, the SCC mechanism will be clarified in detail.

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