Austenitic stainless steels are widely used in many industries utilizing high temperature components such as heat exchangers and chemical reactors because of their good mechanical properties at elevated temperatures and their excellent corrosion resistance. However, when an austenitic stainless steel is welded, its heat-affected zone (HAZ) is often sensitised by formation of intergranular Cr-rich carbides, which deteriorates the corrosion properties of the welded joint. Since the formation of the Cr carbides is caused by exposure to the temperature range of 773 to 1 073 K, rapid cooling in the welding cycle is preferable for prevention of sensitisation. Friction stir welding (FSW) suppressed sensitisation in the HAZ, which could be explained by short duration at sensitisation temperatures during welding. On the other hand, many grain boundaries were deeply corroded in the AS, where the corrosion resistance was significantly degraded. The microstructural observation revealed that sigma phase was formed in the AS during FSW. Sigma formation produced the wide and deep Cr depletion zone with the minimum Cr content less than 12 wt% in the vicinity of the grain boundary in the AS, which severely deteriorated the corrosion resistance in the AS.

1 INTRODUCTION

Austenitic stainless steels are widely used in many industries utilizing high temperature components such as heat exchangers and chemical reactors because of their good mechanical properties at elevated temperatures and their excellent corrosion resistance. However, when an austenitic stainless steel is welded, its heat-affected zone (HAZ) is often sensitised by formation of intergranular Cr-rich carbides, which deteriorates the corrosion properties of the welded joint. Since the formation of the Cr carbides is caused by exposure to the temperature range of 773 to 1 073 K, rapid cooling in the welding cycle is preferable for prevention of sensitisation. Friction stir welding (FSW), which is a relatively new solid state joining process and has been the focus of constant attention in joining low and high temperature materials [1-25], holds promise as an effective method of suppressing development of sensitisation in HAZ because it is a low heat input welding process.

In a previous study [26], Park et al. examined the microstructural evolution in a friction stir welded (FSWed) 304 stainless steel and reported that small sigma phases are rapidly formed along the grain boundaries in the advancing side of the stir zone having a dynamically recrystallised grain structure. Generally, sigma formation in austenitic stainless steels deteriorates the corrosion resistance [27], but the effect of the local small sigma formation on corrosion properties in the FSWed 304 stainless steel has not yet been clarified.

The objective of the present study was to evaluate the corrosion properties of an FSWed 304 stainless steel, especially the advancing side of the stir zone and the HAZ. Corrosion properties of these regions were investigated using the ferric sulfate-sulfuric acid test and the double-loop electrochemical potentiokinetic reactivation (DL-EPR) test, and corrosion properties of the HAZ were compared with those in a gas tungsten arc (GTA) weld.
and 0.003 S. A bead-on-plate friction stir (FS) weld was produced on the base material plate at a travel speed of 1.33 mm/s and a rotational speed of 550 rpm using a polycrystalline cubic boron nitride (PCBN) tool with a length of 4.75 mm [28]. The welding processes used for the present study, including the welding equipment and the other FSW parameters, were the same as those for a previous study [26].

Corrosion resistance in the as-welded specimen containing both the base material and the stir zone was qualitatively examined by a ferric sulfate-sulfuric acid test for 72 h. Since the specimen for this test had dimensions of $10^w \times 40^l \times 6^t$ mm, two cross sections perpendicular to the welding direction, the top surface and the bottom surface of the weld were in contact with the test solution during the test. The tested specimens were observed by optical microscopy (OM) and scanning electron microscopy (SEM). The corrosion resistance in the HAZ of the FS weld was compared to that in the HAZ of the GTA weld by the DL-EPR test. The bead-on-plate GTA weld was made on a base material plate in an Ar atmosphere at a welding current of 300 A and a travel speed of 0.67 mm/s. Depth of the fusion zone was roughly the same as that of the stir zone in the FS weld.

Detailed microstructures were observed by transmission electron microscopy (TEM). Thin disks for TEM with a diameter of 3 mm were cut from various locations of the weld using an electrical-discharge machine and were electrolytically polished in a 10 % perchloric acid + 90 % ethanol solution. The thin foils were observed at 200 kV using a JEOL-2000EXII transmission electron microscope (TEM) and a Philips CM200FEG TEM equipped with an energy-dispersive X-ray spectroscopy (EDS) analysis system, using a 0.6 nm electron probe with a spatial resolution of 1.2 nm. The grain boundary plane was aligned as parallel as possible with both the incident electron beam and the direction of the X-ray detector.

### 3 RESULTS AND DISCUSSION

#### 3.1 Corrosion resistance in FSWed 304 stainless steel

Figure 1 (a) shows the cross section perpendicular to the welding direction in an FSWed 304 stainless steel after the ferric sulfate-sulfuric acid test. The weld was classified into four characteristic regions, i.e., base material (BM), HAZ, stir zone (SZ) and advancing side of the stir zone (AS), which are indicated as BM, HAZ, SZ and AS, respectively, in Figure 1 (a). The sensitised region of the HAZ can be faintly observed in the cross section.

The AS is remarkably corroded and has the worst corrosion resistance in the weld. A cross section perpendicular to the welding direction of the 304 GTA weld after the ferric sulfate-sulfuric acid test is also shown in Figure 1 (b). The sensitised region of the HAZ is distinctly located further outside the fusion zone (FZ), and the width of the sensitised region is much larger than that in the FS weld.

![Figure 1 – Cross sections perpendicular to the welding direction after application of ferric sulfate-sulfuric acid in the FS weld (a) and GTA weld (b)](image-url)