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SIZE EFFECT OF CORROSION FATIGUE PROPERTIES IN MICRO-SIZED 304 AUSTENITIC STAINLESS STEEL SPECIMENS

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ABSTRACT

Corrosion fatigue properties of micro-sized materials are extremely important to design MEMS devices and micro-machines used in corrosive environments. However, there have been few studies that investigate corrosion fatigue properties of micro-sized materials. Thus, it is necessary to develop a corrosion fatigue test method for micro-sized materials. However, there are several difficulties in corrosion fatigue tests on micro-sized specimens. It is also necessary to clarify the problems for the method and to find their solutions. Corrosion fatigue tests for micro-sized 304 austenitic stainless steel specimens have been carried out in air and a 0.9% NaCl solution and the size effects on corrosion fatigue properties have been discussed. Specimens of cantilever-beam-type with dimensions of 10 x 10 x 50 μm^3 were prepared from a 304 austenitic stainless steel thin sheet by focused ion beam machining. The fatigue life of the specimen tested in air was more than 270000 cycles, while that tested in the corrosive environment was 9900 cycles. Distinct environmental effects on fatigue properties were observed. Several problems and solutions for the testing method were also described.

KEYWORDS

Micro-sized specimens, 304 austenitic stainless steel, Corrosion fatigue, Fatigue life, NaCl solution

INTRODUCTION

MEMS(Micro electromechanical systems) devices are expected to be used in human body as Bio-MEMS (diagnosis and treatment devices) and in corrosive environments as inspection devices. The size of components used in such MEMS devices are considered to be in the order of microns. This size is smaller than the grain diameter in conventional metals and alloys. Mechanical properties of such micro-sized materials are considered to be different from those of bulk materials. In addition, the effect of corrosion is considered to be more prominent, because the specific surface area of micro-sized specimens is larger compared with that of bulk specimens. Crevices, the size of which is also in the order of microns, may exist in MEMS devices and thus crevice corrosion is also important. Therefore, corrosion fatigue properties of micro-sized materials are extremely important to design such devices. However, there have been few studies to investigate corrosion fatigue properties of micro-sized materials. This may be due to difficulties

in corrosion fatigue tests on micro-sized specimens. In our previous study [1], we have developed a corrosion fatigue testing machine for micro-sized specimens and have succeeded to carry out corrosion fatigue tests.
In this investigation, corrosion fatigue tests for micro-sized austenitic stainless steel specimens have been carried out in a 0.9% NaCl solution and the size effect on corrosion fatigue properties has been discussed.

EXPERIMENTAL PROCEDURE

The material used in this study was a 304-type austenitic stainless steel thin sheet. At present, austenitic stainless steels and titanium alloys are widely used for medical applications. The 304 austenitic stainless steel thin film is commercially available, and has a fine grain (about 7.6 μm in diameter) structure.

The thickness of the as-received sheet of 304 austenitic stainless steel was 10 μm. A disc with a diameter of 3mm was cut by a punch press. Then, a cantilever beam type specimen was prepared by focused ion beam machining as shown in Figure 1. The thickness and the width of the specimen were 10 μm. Two grooves with a radius of 1 μm were introduced at the both sides of the specimen as stress concentration sites as shown in Figure 1. The stress concentration sites were 10 μm away from the fixed end of the specimen. Thus, the distance from the loading point to the stress concentration sites, L, was 30 μm.

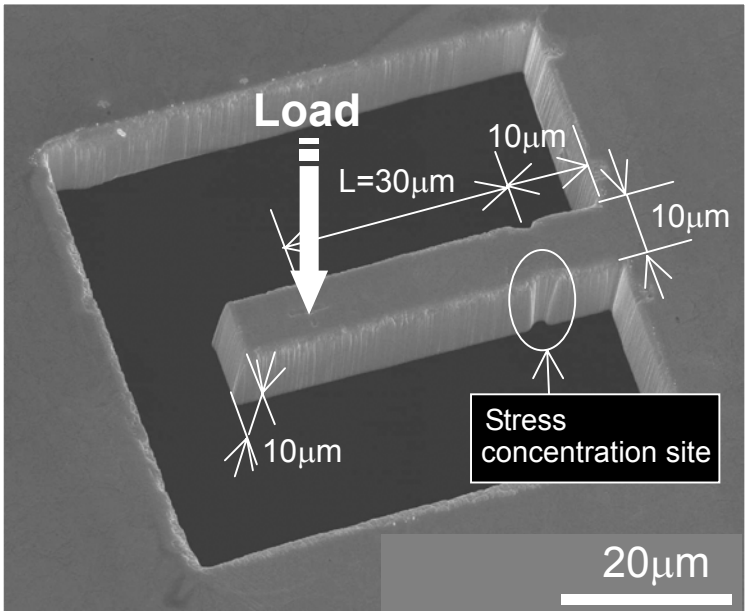
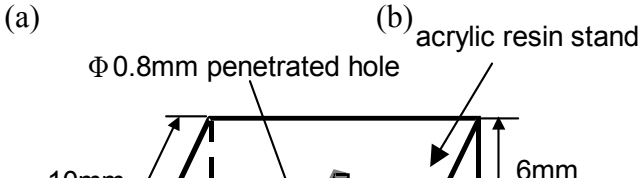


Figure 1: Micro-cantilever beam specimen of the 304 austenitic stainless steel



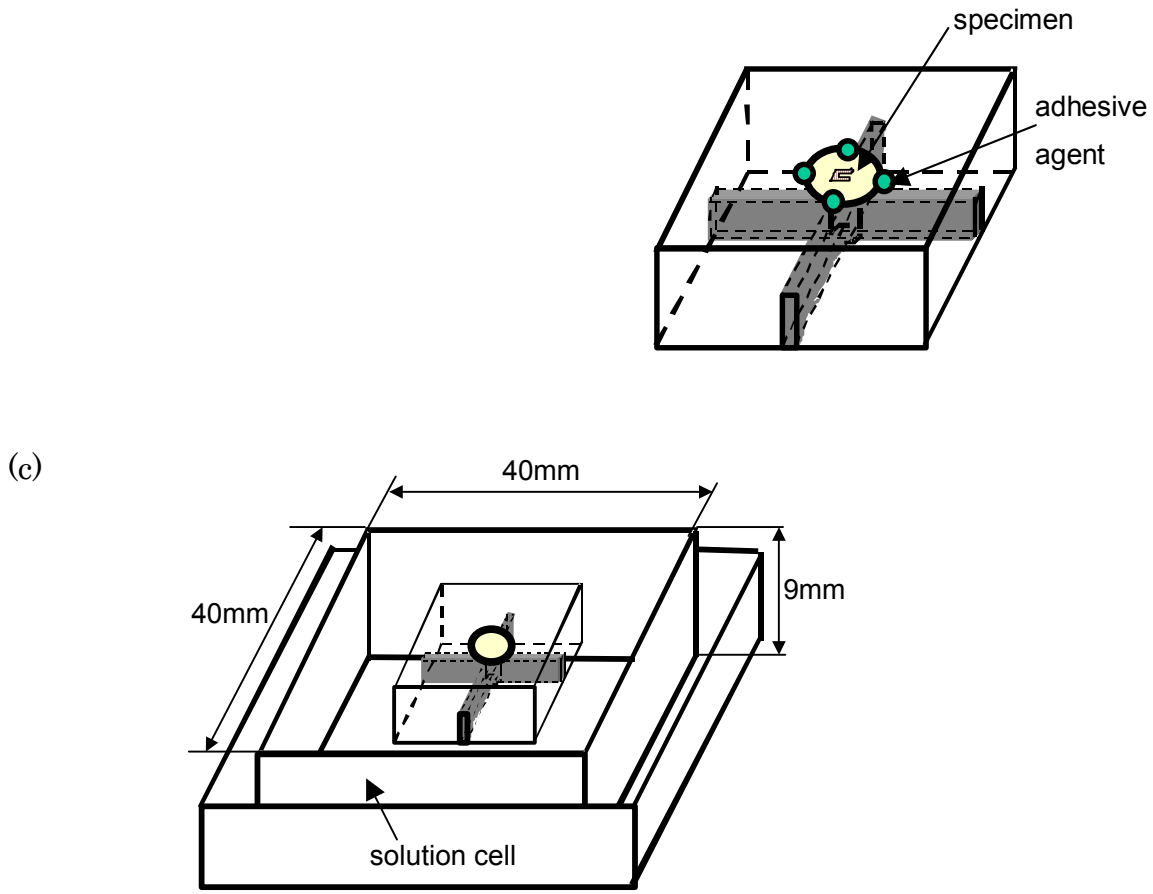


Figure 2: (a) Specimen holder made of acrylic resin. A specimen is mounted on the holder using an adhesive as shown in (b) and the specimen with the holder is immersed in solution as shown in (c).

As shown in Figure 2, a specimen was mounted using an adhesive at the 0.8 mm hole penetrated at the intersection of two 1x3 mm^2 ditches in a stage made of acrylic resin. The specimen with the stage was immersed in the solution cell with dimensions of 40 mm square and 9 mm depth. 0.9% NaCl solution, which simulates human body fluid, was poured into the solution cell. The cell can be positioned with an accuracy of 0.1μm using a precise X-Y stage of the mechanical testing machine for micro-sized materials. A diamond tip (5 μm in radius) was attached to a pushrod. A load cell was connected between the pushrod and the actuator. The load resolution of the load cell used was 10 μN, and the displacement resolution of this testing machine was 0.005 μm. The details of this testing machine, which has been developed in our previous investigation, are described in our previous papers [2-6].

Static bending tests were performed prior to the fatigue tests. The position of the diamond tip connected to the actuator was placed at the loading point, which was 40 μm from the fixed end of the specimen. The specimen was then gradually deflected by descending the diamond tip with displacement steps of 0.3 μm/s. The load applied to the specimen was recorded with the corresponding displacement.

Fatigue life tests were performed at a frequency of 10Hz in air and of 1Hz in the NaCl solution and at a stress ratio (the ratio of minimum to maximum load applied over fatigue cycle) of 0.5. The maximum load (P_{max}) over the fatigue cycle was controlled to 0.7 P_B, where P_B is the bending strength measured in the static bending tests. Specimens were observed after the tests using a field emission gun scanning electron microscope (FE-SEM). This testing machine is set up in a clean room with constant temperature and humidity to eliminate dust and the effect of temperature change during the measurement. The testing machine is also placed in a wind screen box to shield from the slight wind stream in the room.

RESULTS AND DISCUSSION

In order to develop a corrosion fatigue test method for micro-sized materials, three problems had to be solved. The first problem was how to hold the specimen in the aqueous solution. The specimen holder should not elute into the aqueous solution to avoid any contamination and should not be made of metals to avoid galvanic corrosion. Thus, a specimen stage was made of acrylic resin and a specimen was fixed with an adhesive.

The second problem was how to cover the specimen with aqueous solution. Aqueous solution does not enter thin holes or ditches due to water repellency and surface tension. Thus, we poured the aqueous solution into the cell in a vacuum chamber and the coverage of the specimen was confirmed using a CCD (charge coupled device) camera.

The third problem was how to eliminate error in load measurements. In micro-sized testing, the measuring load would be so small that the buoyancy of the pushrod might not be negligible. In addition to this, the viscosity and surface tension of the aqueous solution would cause error in load measurements. For example, surface tension would be added to and reduced from the actual load applied to the specimen due to the fluctuation of the meniscus during corrosion fatigue tests, as shown in Figure 3. During this situation, the buoyancy of the pushrod also fluctuates because the deflection of the specimen results in a change in the depth of the pushrod immersed under the aqueous solution. The evaporation of the aqueous solution also causes a change in the buoyancy during the tests. Thus, it is quite difficult to correct the error completely. However, no detectable difference in phase was observed between applied displacement and detected load signals, when the loading frequency was reduced to 1Hz. Thus, the error caused by the viscosity of the aqueous solution was negligible at this loading frequency. (Note that the inclusion of the force caused by viscosity leads to a change in phase because the viscosity resistance of Newtonian fluid is in proportion to the velocity.)

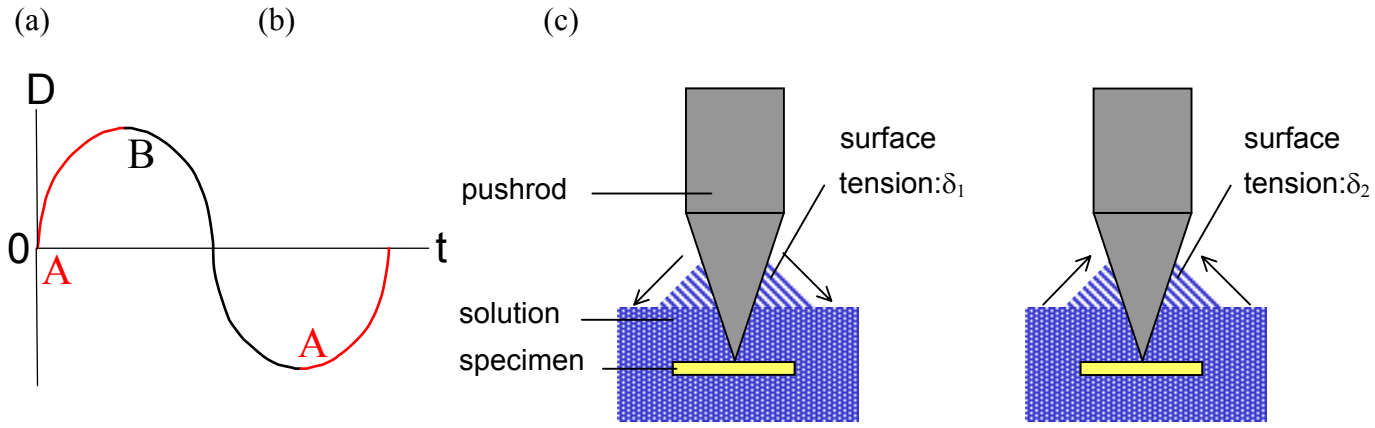


Figure 3: (a) Schematic diagram of displacement as a function of time, and schematic illustrations around the pushrod showing that surface tension (b) added at A and (c) subtracted at B from the actual loads.

(a) (b)

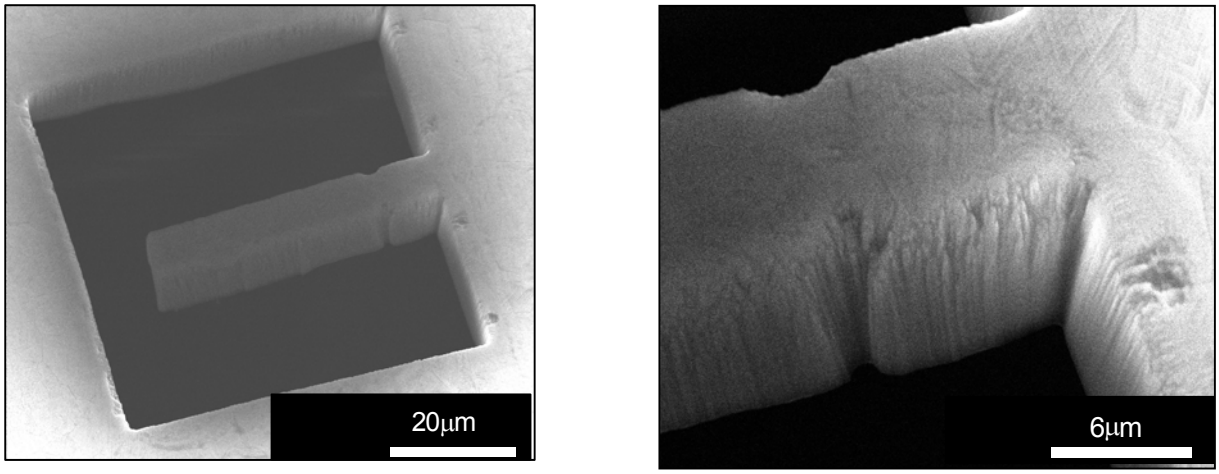


Figure 4 : Stainless steel specimen after a fatigue test in air, showing that (a) the specimen did not fail, and (b) no crack was found at the stress concentration sites.

(a) (b)

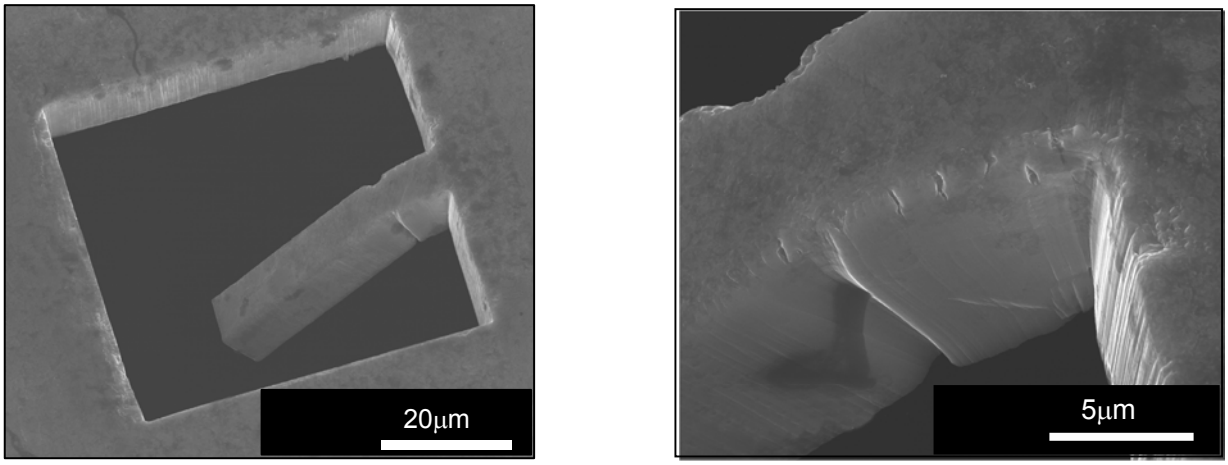


Figure 5: Stainless steel specimen after a corroison fatigue test, showing that (a) the specimen failed, and (b) cracks were found at the stress concentration sites and also the root.

The stainless steel specimens did not fail after 2.7 x 10^5 cycles in air, on the other hand, failed after only 9.9 x 10^3 cycles in the NaCl solution. Figure 4 shows SEM-images of the stainless steel specimen after fatigue test in air. No crack was found on the stainless steel specimen fatigue tested in air. Figure 5 shows SEM-images of the stainless steel specimen after fatigue test in the corrosive environment. Some cracks were found at the fixed end of the specimen and at the stress concentration sites of the specimen tested in the corrosive environment. The fatigue life of the micro-sized specimen tested in the corrosive environment was one order of magnitude lower than that tested in air, which corresponded with data of ordinary-sized specimens.

The fatigue life in the corrosive environment was much lower than that in air. No crack was found on the specimen fatigue tested in air, but some cracks were found at the fixed end and at the stress concentration sites on the specimen fatigue tested in the corrosive environment. Therefore, environmental effects on fatigue properties of micro-sized specimens were clearly observed.

CONCLUSIONS

The fatigue life of the specimen tested in air was more than 270000 cycles, while that tested in the corrosive environment was 9900 cycles. In other words, the fatigue life of the micro-sized specimen tested in the corrosive environment was one order of magnitude lower than that tested in air, which corresponded with data of ordinary-sized specimen. Distinct environmental effects on fatigue properties were observed. Several problems and solutions for the testing method were also clarified

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