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Size Effects of the Micro-Sized Polycrystalline SUS304 Tensile Specimen Fabricated by Electrolytic Polishing Technique

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The tensile specimens with diameters less than 100 μm were fabricated from a 0.5 mm diameter wire of polycrystalline austenitic stainless steel (SUS304) with average grain size of 166 μm using electrolytic polishing technique. Optical, laser and scanning electron microscope observations revealed a smooth and mirrored specimen surface. Tensile tests of these specimens were carried out by the testing machine for micro-sized materials. In this study, tensile properties of the specimens with diameters smaller than average grain size of the sample were investigated. Tensile tests showed the clear yield points and gradual work hardening behaviors on the stress-displacement curves. All specimens exhibited strengths like a single crystal. The yield and tensile strengths were about 100 and 180~240 MPa, respectively. These specimens exhibited anisotropic fracture surfaces after tensile tests. Scanning electron microscope observations revealed the deformations of these specimens mainly occurred at grains placed between grain boundaries. The specimen with diameter less than or equal to about maximum grain size of the sample exhibited strength like a single crystal.
Key words: size effects, micro-sized specimen, austenitic stainless steel, electrolytic polishing, grain boundary

1. INTRODUCTION

The ratio of surface area to volume increases as a material gets smaller. For this reason, the mechanical properties of a micro-sized material are strongly influenced by their surface conditions [1, 2]. Therefore, we need to test specimens with negligible work-affected layers to accurately study mechanical properties of micro-sized materials. However, conventional microfabrication methods are poorly suited for the manufacture of specimens that meet this condition. In order to overcome this problem, we focused attention on electrolytic polishing and developed a novel method for manufacturing micro-sized tensile specimens with negligible work-affected layer using a precision electrolytic polishing apparatus in our previous study [3, 4].

In this study, micro-sized tensile specimens with diameters smaller than average grain size of the sample fabricated from a polycrystalline austenitic stainless steel wire by electrolytic polishing, and tensile properties of these specimens were investigated.

2. EXPERIMENTAL

2.1 Precision Electrolytic Polishing Apparatus

Generalized Ohm's law is shown in the following equation.

$$i = \kappa \text{ grad } \varphi(x, y, z)$$

Where i is current density, κ is specific conductivity and φ is electric potential. This equation indicates that current density i, which is identical to the polishing rate, is controlled by the gradient of electric potential φ when specific conductivity κ is constant.

Fig.1 (a) shows electric potential distribution simulated on the conductive paper when the cathode of 0 V is arranged in one point to the long anode of 6V. Vertical and horizontal lines indicate a lattice drawn at intervals of 50 mm on the conductive paper. The curved lines are equipotential lines drawn at intervals of 0.2 V. The sample is locally polished in an electrolyte by achieving the electric potential distribution shown in Fig.1 (a). Therefore, we can fabricate a symmetric tensile specimen by performing electrolytic polishing with the arrangements shown in Fig.1 (b) in an electrolyte with a constant specific conductivity.

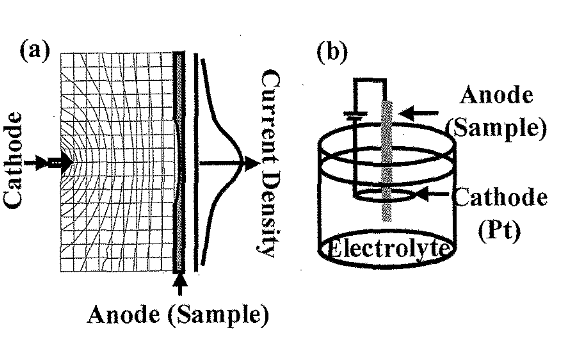

Fig.1 (a) Simulated electric potential distribution and (b) schematic illustration of the arrangements of electrodes in the electrolyte cell.

In a previous study, we developed a precision electrolytic polishing apparatus based on this idea. We successfully fabricated a micro-sized tensile specimen with a diameter of less than 100 μm from a stainless steel wire [3, 4]. The cathode of this apparatus is platinum ring of 0.3 mm in thickness and 4 mm in diameter. This apparatus can perform electrolytic polishing with keeping specific conductivity equal on the sample surface during electrolytic polishing by applying rectangular wave voltage to a sample in a flowing electrolytic solution.

2.2 Material and Specimen Preparation

A type-304 polycrystalline austenitic stainless steel wire with a diameter of 0.5 mm was selected as the material for study. This wire manufactured by TAKEUCHI METAL & FOIL CO., LTD. As-received curved wire was straightened by twisting it with applying tension. Solution heat treatment at 1,373 K for 1 h was performed with straightened sample. After heat treatment, average grain size of the sample was 166 μm. Sample rods were obtained from this wire. Micro-sized tensile specimens with circular cross-sections were fabricated from these rods using electrolytic polishing apparatus. 10 vol% percholoric acid and 90 vol% acetic acid solution was used as an electrolytic polishing. A sample rod was placed vertically at the center of a ring-shaped counter electrode and gripped by collet chuck, then a rectangular wave voltage with maximum voltage of 9 V, minimum voltage of 0 V, period of 6 s and duty cycle of 33 % was applied to a sample in an electrolytic solution flowing at a rate of 1.0 ml/s. After this polishing, final polishing was performed using rectangular wave voltage with maximum voltage of 9 V, minimum voltage of 0 V, period of 3 s, duty cycle of 33 % in a static solution for 10 min in order to remove striations remained along flow direction of electrolytic solution after polishing in a flowing solution. Polished specimens were alternately cleaned by distilled water and ethanol.

We can fabricate tensile specimens with various diameters by changing polishing time. In this study, specimens with diameters of 49, 59, 83 μm were fabricated as a specimen with diameter smaller than average grain size of the sample. For comparison, specimens with diameters of 200~400 μm were also fabricated as a specimen with diameter bigger than average grain size of the sample.

2.3 Tensile Tests of Micro-Sized Specimens

Tensile tests of the specimens with diameters smaller than average grain size of the sample were carried out using a mechanical testing machine for micro-sized specimens (MFT-2000), which we have developed in our precious investigation [5]. Tensile tests of the specimens with diameters bigger than average grain size of the sample were performed using a SHIMAZU tabletop type tester (EZTest). All tests were performed at the same crosshead speed of 2.0 mm/min and grip distance of 50 mm at room temperature.

2.4 Analytical Equipments

After the specimens had undergone electrolytic polishing and tensile testing, the shapes and surfaces were examined using an optical microscope (Nikon type102, Nikon Co.), a laser microscope (1LM21, Lasertec Co.) and two scanning electron microscopes (FE-SEM, S-4300 and S-4500, Hitachi High-Technologies Co.).

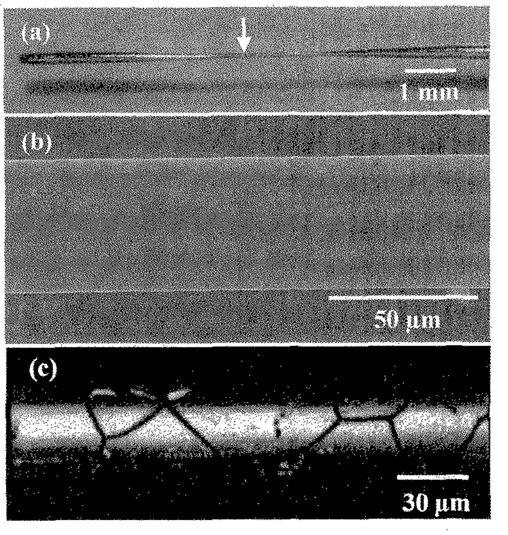

Fig.2 A micro-sized tensile specimen fabricated by electrolytic polishing. (a) Whole, (b) central point and (c) surface after 60 % HNO_3 etching images of the specimen. A white arrow in Fig.2 (a) indicates the central position of ring-shaped cathode.

3. RESULTS AND DISSCUSSION

3.1 Micro-Sized Tensile Specimen Fabricated by Electrolytic Polishing Technique

Fig. 2 shows a micro-sized tensile specimen fabricated using electrolytic polishing. Fig. 2 (a) shows the whole image of the specimen. A white arrow in this figure indicates the central position of ring-shaped cathode. Fig. 2 (b) shows the high magnified image of the center point of specimen. Examination with microscopes revealed a smooth and mirrored surface of the specimen. Fig. 2 (c) shows a laser micrograph of the specimen surface after 60 % HNO_3 etching at a voltage of 1 V for 1 min.

3.2 Tensile Properties of Micro-Sized Tensile Specimen with Diameter Smaller than Average Grain Size of Sample.

Fig. 3 shows a stress-displacement curve obtained by tensile test of the specimen with a diameter of 49 μm. The curve exhibited a clear yield point and gradual work hardening behavior after yielding. The yield and tensile strengths of the specimen were 100 and 240 MPa, respectively, or about the same as those of a single crystal. Fig. 4 shows a laser micrograph of the fracture specimen surface. Deformed grain in single slips as shown in Fig. 4 was observed in several parts of the specimen surface after tensile test. Fig. 5 shows scanning electron micrographs of the fracture surface of specimen with a diameter of 49 μm. Fig. 5 (a) and (b) represent front and side views of the fracture surface, respectively. The specimen exhibited anisotropic fracture surface necked in specific direction.

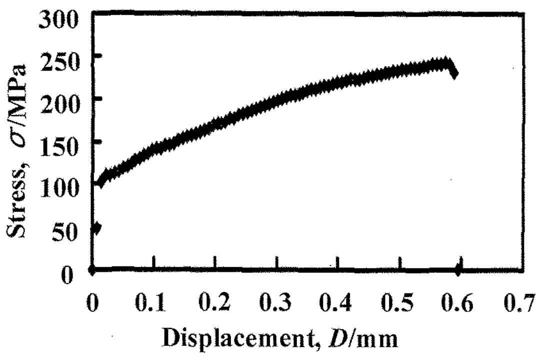

Fig.3 Stress-displacement curve obtained by tensile test of the micro-sized specimen with a diameter of 49 μm.

Necking deformations were also observed at regions away from fracture part. Fig. 6 shows one of necking deformation regions after 60 % HNO_3 etching at a voltage of 1 V for 20 s. A grain placed between grain boundaries existed in such a region. Dislocation activations in a grain like this are not prevented by grain boundaries because slip planes pass through a specimen. Therefore, dislocations can easily pass out from the specimen surface. It is considered that these grains are deformed like a single crystal. On earlier stage of deformation, these grains can be deformed by only primary slip system. As a result of deformation by limited slip system, anisotropic fracture surface as shown in Fig. 5 is considered to be formed after fracture. Similar results were obtained in all specimens with diameters smaller than average grain size of the sample.

From results obtained by present study, we proposed the following deformation behavior of a specimen with diameter smaller than average grain size of the sample. Firstly, a grain having largest Schmid factor in grains placed between grain boundaries is deformed in single slips. It is considered that the yielding of this grain corresponds to clear yield point observed on the stress-displacement curve. Next, crystal rotation in this grain occurs, followed by activation of secondary slip system. Interactions of dislocations lead to work hardening of this grain. Once work hardening in first slipped grain starts, single slip deformations of other grains mainly occur one after another. The specimen elongation led by deformations of these grains smoothed work hardening behavior of first slipped grain. Therefore, it is considered that gradual work hardening behavior is observed on the stress-displacement curve. Finally, the specimen fracture takes place in first slipped grain.

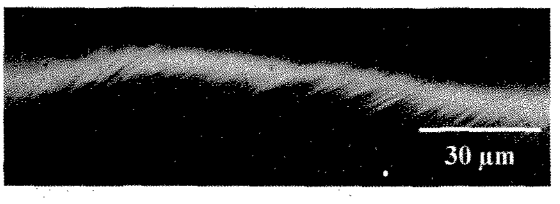

Fig.4 Laser micrograph of a deformed grain in single slips on the fracture specimen surface after tensile test.

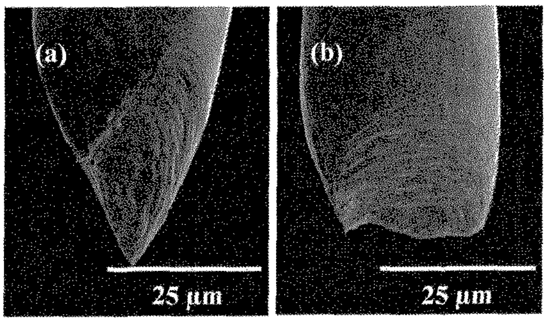

Fig.5 Scanning electron micrographs of fracture surface of the specimen with a diameter of 49 μm. (a) Front view and (b) side view of the fracture surface.

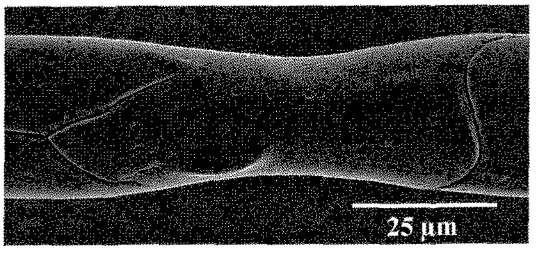

Fig.6 Scanning electron micrograph of necking deformation region away from fracture part after 60 % HNO_3 etching.

Fig. 7 shows the relationship between specimen diameter and yield & tensile strengths. Dotted line indicates the position of average grain size of the sample. Cross-hatched pattern region indicates range of maximum grain size of the sample. On this figure, the specimen with a diameter of 228 μm which is larger than average grain size also exhibits low strength. 228 μm is nearly maximum grain size of the sample used in present study. This result indicates that the specimen exhibits strength like a single crystal when a grain placed between grain boundaries exists in the specimen.

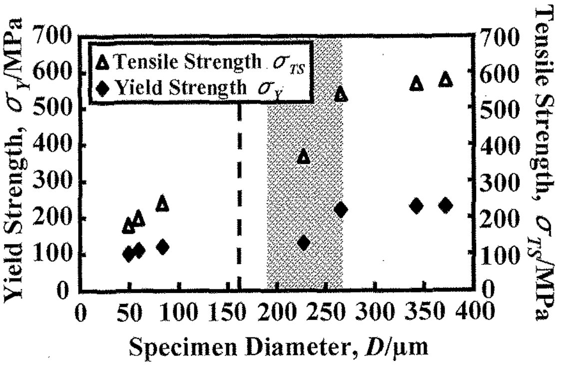

Fig.7 Relationship between specimen diameter and yield & tensile strengths. Dotted line indicates the position of average grain size of the sample. Cross-hatched pattern region indicates range of maximum grain size of the sample.

4. CONCLUSION

Micro-sized stainless steel tensile specimens with diameters smaller than the average grain size of the sample were fabricated using electrolytic polishing technique, and tensile tests of these specimens were carried out. The stress-displacement curves obtained by tensile tests of these specimens showed clear yield points and gradual work hardening behaviors after yielding. The yield and tensile strengths of the specimen were almost the same as those of single crystals. After tensile tests, deformed grains in single slips were observed on the fracture specimen surface. These specimens exhibited anisotropic fracture surfaces after fracture. The specimen deformation observed by scanning electron microscope mainly occurred at grains placed between grain boundaries. Present study represented that the specimen with diameter less than or equal to about maximum grain size of the sample exhibited strength like a single crystal.

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