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Nanocrystallization of Surface Layer in a 316 Austenitic Stainless Steel by Ultrasonic Shot Peening

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Nanocrystallization of material's surface will construct a smart structure in a material as this structure will suppress the crack initiation at the surface of material and may improve the fatigue property of material. In this study, we have developed a nanocrystallization method for surface layer in a type-316 austenitic stainless steel using an ultrasonic shot peening (USSP) technique. Heavy plastic deformation was applied to the specimen surface by USSP and the specimen was then heat-treated up to 973 K. Transmission electron microscopy observations in the surface layer (5 μm in depth from the surface) were performed. Grains of austenite near the surface layer were entirely transformed into strain-induced α'-martensites with a grain size of 100 to 400 nm after USSP and high-density dislocations were observed in the martensites. Electron diffraction patterns indicate that grains in the as-shot peened specimens are randomly oriented. Fine grains of austenite with a grain size of 100 to 400 nm were observed in the surface layer of the specimen annealed at 973 K after USSP. It was confirmed that the USSP technique is useful for nanocrystallization of surface layer in austenitic stainless steels.

Key words: USSP, nanocrystallization of surface layer, austenitic stainless steel, martensitic transformation

1. INTRODUCTION

The nanocrystallization of metallic materials is one of the effective methods to improve mechanical or functional properties of materials. Fabrication methods of nanocrystalline materials (materials with a grain size under 100 nm) have been extensively investigated in recent years. A variety of preparation techniques, such as inert gas condensation and compaction [1], severe plastic deformation [2] and crystallization of amorphous solids [3], have been developed for the production of nanocrystalline materials. It has been reported that nanocrystalline materials generally exhibit superior mechanical properties [1].

It is known that austenitic stainless steels are transformed into α'-martensite by plastic deformation [4]. Austenitic stainless steel with fine grain is often produced by reverse transformation of deformation-induced α'-martensite [5-6]. It is necessary to form fine α'-martensites by severe plastic deformation in order to obtain nanocrystalline austenitic stainless steel by this method because grain size of the transformed austenite is almost determined by the grain size of the deformation-induced α'-martensite.

Whole region of the material is generally nanocrystallized in these techniques. However fatigue properties depend on the microstructure of material surface because stress caused by bending becomes maximum near the material surface and fatigue-crack is generally initiated on the material surface [7]. Therefore nanocrystallization of material surface is considered to be effective for the improvement of fatigue properties of material.

An ultrasonic shot peening (USSP) technique is considered to be one of the effective methods to apply severe plastic deformation on material surfaces [8-9]. Many balls hit against the specimen and severe plastic deformation is applied only near the material's surface in this technique. One of the benefits in this method is that texture is not formed in the produced material because the material is loaded from random directions. Additionally, materials in various shapes can be treated in the USSP technique even after welding of components in structures.

In this study, we have developed a method to nanocrystallize only the sub-surface layer in a type-316 stainless steel by USSP technique.

2. EXPERIMENTAL

2.1 Ultrasonic shot-peening machine

Figure 1 shows a schematic illustration of USSP equipment used in this study. An atmosphere can be controlled in a chamber and shot peening is usually performed in vacuum. Steel balls are placed in a plate that is attached to an ultrasonic transducer. The diameter of steel ball was 1.5 mm and the distance between the specimen and steel balls was set to be 0.1mm in this study. The balls jumping from the vibrated plate hit against the specimen and severe plastic deformation is applied on the material surface.

2.2 Material and specimen preparation

The material used in this study is a commercially available type-316 austenitic stainless steel rod with a cross sectional diameter of 15 mm. Grain size of the received material was 30 ~ 50 μm. The chemical composition of the material is shown in Table I. Disks with a thickness of 1mm were cut from this rod using a wheel cutter. Electro polishing was performed on the disks using a solution of 10 % perchloric acid and 90 % acetic acid and electro etching was performed using an

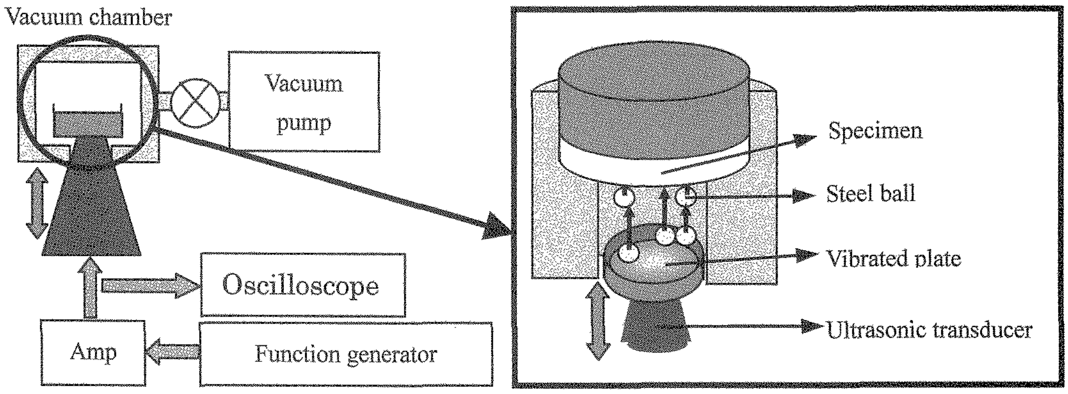

Fig. 1 Schematic illustration of the USSP equipment. The balls jumping from the vibrated plate hit against the specimen and severe plastic deformation is applied on the material surface.

Table I Chemical composition of a type-316 austenitic stainless steel.

C	Si	Mn	P	S	Ni	Cr	Mo
0.05	0.34	1.35	0.031	0.027	10.1	16.86	2.08

(mass%)

oxalic acid solution. Then, the prepared disks were used as the specimens for USSP. USSP treatment was performed on the specimens at room temperature for 60, 600 or 6000 s.

Cross-section of the shot peened specimen was observed by a scanning laser microscope after electro-etching. Disks of 3 mm in diameter were cut from the shot-peened area. Thin foil specimens were prepared from the 3 mm-disks using a twin-jet polishing technique. Sub-surface region which located at 5 μm in depth from the shot-peened surface was in the thinner area of the thin foils. Transmission electron microscopy (TEM) observations were carried out using a Philips CM200 operated at 200kV.

Differential Scanning Calorimetry analysis (DSC) was carried out in the shot peened specimen in order to measure reverse transformation temperature.

The shot peened specimens were annealed at temperatures up to 973 K for 600 s in vacuum and then quenched in water. Microstructures (5 μm in depth from the surface) were observed by TEM.

3. RESULTS AND DISCUSSION

3.1 Ultrasonic shot peening

Observation of the specimen shot peened for 60 s by laser microscopy showed only slip bands near the surface in the cross-sectional area of the specimen. Many slip bands were observed at about 10 μm in depth from the surface in the specimen shot peened for 600 s. Austenite is partially transformed into α'-martensite in each grain from the result of TEM observation. It is considered that the amount of applied plastic deformation was insufficient to transform whole the austenite grains.

The cross-sectional morphology (cross-section of the specimen) of the sample shot peened for 6000 s observed by optical microscopy is shown in Fig. 2. Many slip bands were observed at about 30 μm deep from the surface. It is expected that sufficient plastic deformation is applied in the sub-surface layer.

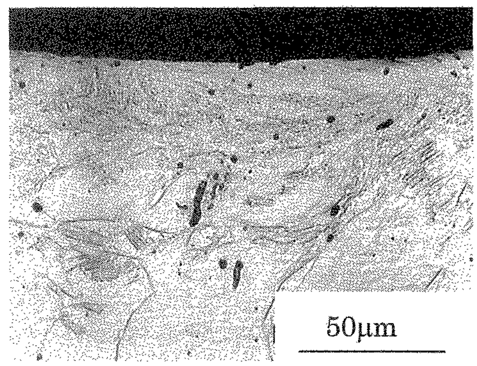

Fig. 2 The cross-section of the specimen shot peened for 6000 s. Many slip bands were observed at about 30 μm deep from the surface.

Figure 3 shows a TEM bright field image of the specimen shot peened for 6000 s. Fine grains with a grain size of 100 ~ 400 nm were observed in Figs. 3(a) and (b). Debye ring was observed on the selected area diffraction (SAD) of this area. This indicates that each fine grain was randomly oriented. It is considered that the fine grains consist of α'-martensite, judging from the radius of the Debye rings.

α'-Martensite holds Kurdjumov-Sachs (KS) orientation relationship [10] with respect to the parent austenite in this alloy system [4]. An austenitic grain is divided into several martensitic grains having deferent crystal orientations by martensitic transformation because α'-martensite possesses 24 KS variants. In addition, grain rotation of martensites is considered to be induced during sever plastic deformation. Therefore α'-martensites having randomly crystalline orientations were considered to be formed by USSP treatment in this study.

Thus, α'-martensites with a grain size of 100 to 400 nm, which were randomly oriented each other, were produced on the surface by USSP. It was confirmed that austenite grains are almost entirely transformed into

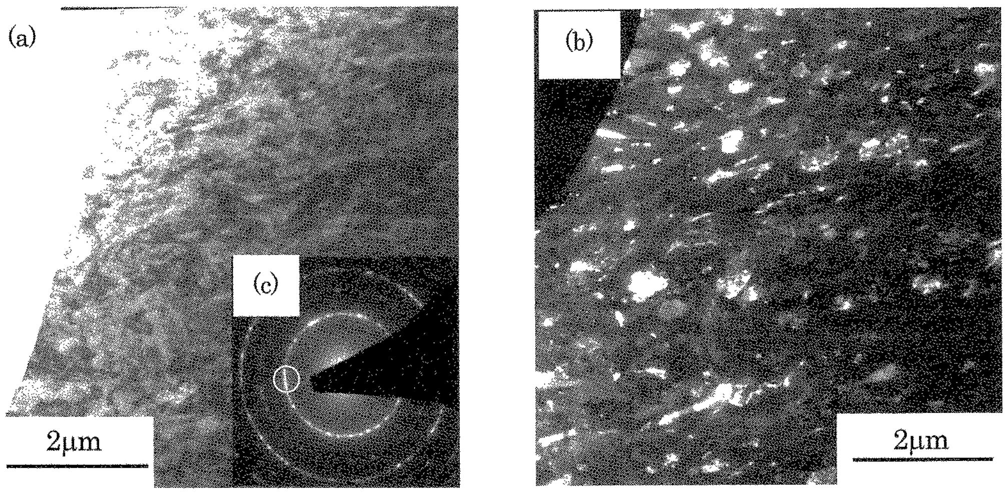

Fig. 3 TEM image observed in the specimen shot peened for 6000 s (a) Bright field image, (b) Dark field image and (c) SAD pattern. α'-martensites with a grain size of 100 to 400 nm were produced on the surface by USSP.

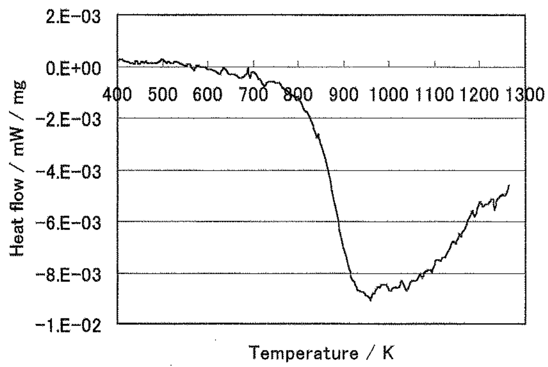

Fig. 4 DSC analysis of the specimen shot penned for 6000 s.

α'-martensite by USSP treatment of 6000 s in this study. According to the results presented above, fine grained austenite was expected to be obtained by a suitable heat-treatment to induce reverse transformation of α'-martensite.

3.2 DSC analysis

DSC analysis was conducted on the specimen USSP-treated for 6000 s to determine reverse transformation temperature. DSC was measured from temperatures of 293 K to 1273 K and heating speed was 10 K/min. From the result of DSC analysis (Fig. 4), it is confirmed that reverse transformation starts at about 820 K. The specimen has to be heat-treated at the temperature as low as possible in order to suppress grain growth after reverse transformation. The shot peened specimen was then annealed at the temperature up to 973 K in this study.

3.3 Microstructure of the annealed specimens

Specimen annealed at temperature range of 473 ~ 773 K almost entirely consisted of α'-martensite and microstructure seemed to be the same as observed in as-shot peened specimen. It is considered that reverse transformation did not occur in this temperature range. This agrees with the result of DSC measurement.

Microstructure of the specimen annealed at a temperature of 873 K was different from that of the as-shot peened specimen. Microstructure mixed austenite and martensite was observed in the specimen annealed at 873 K. It is considered that reverse transformation had partially occurred at this temperature.

Then, annealing at 973 K was performed on an as-shot peened specimen. It was expected that deformation-induced α'-martensites are fully transformed into austenite by the annealing at 973 K, according to the result of DSC measurement. Figure 5(a) shows a TEM bright field image of the specimen annealed at 973 K. Grains of few hundred nm in diameter were observed. Each grain seemed to have quite low density of dislocations, compared to that of as-shot peened specimens. SAD pattern was taken from an area in Fig. 5(a) using selected area aperture of 3.5 μm in diameter. Figure 5(b) shows obtained SAD pattern and Fig. 5(c) shows a schematic illustration of 5(b). The Debye ring of the smallest diameter in Fig. 5(b) was due to $(111)_\gamma$ reflections from austenite (about 2% smaller in diameter, than that of $(110)_\alpha$ type Debye ring of α'-martensite). In addition, $(200)_\gamma$ type Debye ring was clearly observed. According to the results of selected area diffractions, as-shot peened specimen was found to be transformed into fine grained austenite with almost random orientation, by the annealing at 973 K in this study. Because the grain size of α'-martensite in the as-shot peened specimen was 100 to 400 nm, it is considered that this fine grained austenite with a grain size of 100 to 400 nm was formed by reverse transformation. Figure 5(d) shows a dark field image taken by the encircled reflections in Fig. 5(b). Grains which contrasted in white were located randomly and did not adjoin each other in Fig. 5(d). It is suggested that tilt angle of grain boundaries were not so small.

From the above result, nanocrystallization only near the surface layer was achieved in a type-316 austenitic stainless steel. Randomly oriented nanocrystallized

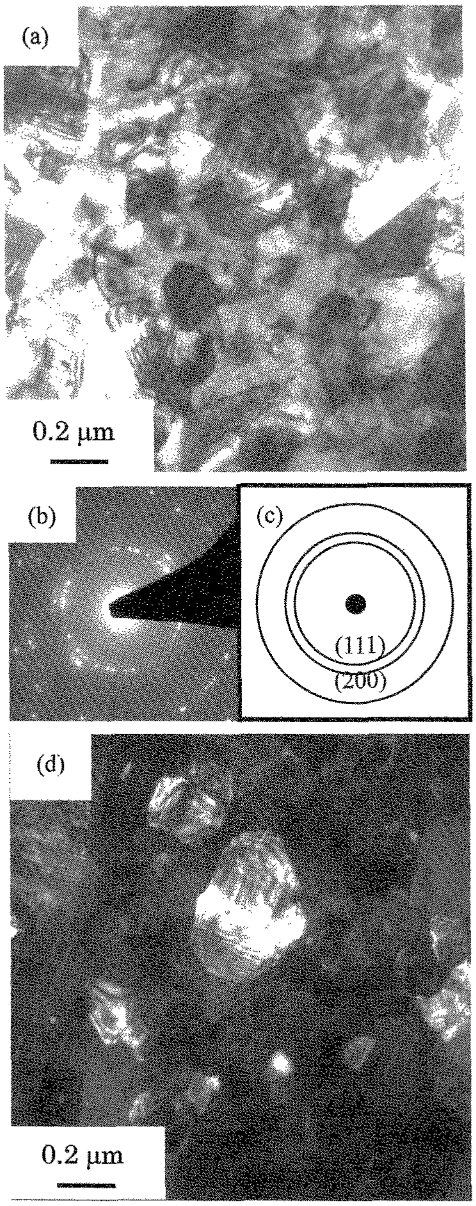

Fig. 5 TEM micrograph in the specimen annealed at 973 K after USSP. (a) Bright field image, (b) SAD pattern, (c) Schematic illustration of SAD and (d) Dark field image. Fine grained austenite with a grain size of 100 to 400 nm was formed by reverse transformation.

structure can be obtained without formation of voids or pores in this method. USSP technique is effective to nanocrystallize only the sub-surface layer of the austenitic stainless steel.

4. CONCLUSION

Deformation-induced α'-martensite with a grain size of 100 to 400 nm was produced by USSP for 6000 s in a type-316 austenitic stainless steel. Reverse transformation of α'-martensite occurred by annealing this specimen at a temperature of 973 K. Fine grained austenite with a grain size of 100 to 400 nm was formed in the sub-surface layer of the specimen annealed at 973 K.

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