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A research on the microstructure evolution of austenite stainless steel by surface mechanical attrition treatment

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ABSTRACT

Severe plastic deformation (SPD) was performed on AISI304 stainless steel by surface mechanical attrition treatment (SMAT). The microstructure evolution of the specimens along the depth direction were examined by transmission electron microscope (TEM) and electron backscattered diffraction (EBSD) associated with field emission scanning electron microscope (FESEM). The results show that the specimens by SMAT can be divided into three regions. Region I is the untreated one, where a large number of annealing twins exist. Region II is the small plastic-deformed one, where the grains are deformed by dislocation slipping. Region III is the severe plastic-deformed one, where the grains are deformed by mechanical twinning. The hardness of the untreated and the treated specimens were determined, which shows that the hardness of the untreated specimen (including region I) is about 250HV, while the hardness is uniformly increased from region II to region III, and the largest value appears on the surface. The untreated and the 600 N-treated specimens were determined and observed by X-ray diffractometer (XRD) and confocal laser scanning microscope (CLSM), which shows that the SMAT treated specimen exhibits a layer of ultra-fined γ-Fe grains on the treated surface.

1. Introduction

Austenitic stainless steels (ASSs) have been widely used in the fields, such as petroleum, chemical engineering, power generation and food processing because of their superior corrosion resistance. One of the major drawbacks of ASSs is their low yield strength in the annealed states, which limits their applications [1]. There are a lot of strengthening mechanisms for ASSs including grain refinement, solid solution, and work hardening [2,3]. While among these mechanisms, strengthening ASSs without compromising other useful properties is still a challenge. For example, the strengthened ASSs containing dispersed precipitates are usually less corrosion resistant [4]. In recent years, with outstanding mechanical properties in terms of high strength, elevated fatigue strength, and improved corrosion resistance [5–8], ultra-fine grain (UFG) materials by severe plastic deformation (SPD) have attracted the attention of many researchers [9–12]. SPD is usually performed via equal-channel angular pressing (ECAP) [13], high pressure torsion (HPT) [14], surface mechanical attrition treatment (SMAT) [15,16], and cold-rolling (CR) [17–19], which can fabricate polycrystals with submicron-sized or even nanometer-sized grains [20–22]. In engineering applications, many failures of ASSs occur on their surfaces, including fatigue fracture, wear and corrosion etc. [23]. Therefore, in these cases, optimization of the surface structure is necessary, while the properties of the matrix can be remained. For this reason, SMAT is now an effective method for surface modification [24]. Shot peening, burnishing and sand blasting, and spherical shot bombardment [25–29] are some typical SMAT methods. During most of them, the surface is repeatedly bombarded with spherical shots or particles to induce SPD at high strain rates. However, martensite phases are frequently induced by these methods in ASSs, which is detrimental to the corrosion resistance. Therefore, a synergistic SMAT method of producing UFG surface as well as eliminating martensite phase transformation is a challenging issue.

Most of the studies on SMATed ASSs have been carried out only for the treated surface that forms UFG and mechanical twins [30]. Although not many investigations have been conducted on the cross-sections of SMATed ASSs, it is known that other microstructural evolutions beneath the surface, such as the change in the grain boundaries and the rotation of grains are also frequently induced by plastic deformation in ASSs. These phenomena are of considerable importance in the overall performance of ASSs. Traditionally, optical microscopy (OM) and transmission electron microscopy (TEM) have been used to study the plastic deformed specimens, which both have their limitations. The former have

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limitations in characterizing the substructure and grain boundary character, while the latter is limited to small parts of the specimen [31]. Electron backscattered diffraction (EBSD) associated with field emission scanning electron microscope (FESEM) is a powerful tool to study the plastic deformed specimens, by which, highly detailed data such as misorientation, grain boundary character, and grain size are acquired from a large area of the polished specimen [32]. Cooperating with TEM, the deformed specimens can be studied systematically.

AISI304 stainless steel, which is a typical metal with low stacking fault energy (SFE) [33], has been considered as one of the strongest candidate ASSs because of its comprehensive performance and low price. The strengthening mechanism of grain refinement plays an important role in the development of AISI304 stainless steel that is of technologically importance. The objective of the present work is to strengthen AISI304 stainless by a new SMAT method without inducing martensite phase transformation. Meanwhile, a point of particular interest is to determine the influence of the strain conditions on the microstructural evolution beneath the treated surface. For this reason, cross-sections of the specimens born various normal loads were observed by EBSD and the detailed data acquired by EBSD were compared to describe qualitatively the influence of strain conditions on the microstructural evolution. TEM observations along the depth direction were performed to further understand the deformation behavior and the refinement mechanism.

2. Experimental

2.1. Material

AISI304 steel is representative among ASSs in having primary δ-Fe, followed by a δ+γ region with partial peritectic and eutectic reactions, and finally having the equilibrium structure with single γ-Fe at room temperature. However, some δ-Fe usually remains in case of non-equilibrium solidification. In the present study, the as-received AISI304 steel was prepared by heat treatment at 1050 °C for 25.6 min, which makes the dissolution of δ-Fe. The chemical composition of the AISI304 stainless steel is given in Table 1.

2.2. Methods

The AISI304 stainless steel was cut into disks (⌀43 mm × 3 mm) and rods (⌀4 mm × 18 mm). Then, two symmetrical circular holes (⌀5 mm) were cut on the diameter of each disk. The surfaces of the disks and the rods were prepared by grinding and mechanical polishing. The steel disks were installed on the base of the MMU-5G wear tester via the holes on the disks and the pins on the base, while the steel rods were inserted into the holding fixture, which are schematically shown in Fig. 1(a). Different normal loads (50 N, 300 N, 400 N, 600 N) were applied to the steel rods, which make them contact with the disks. Then the steel rods were circularly moved with the holding fixture, conducting SMAT to the disks. The duration of SMAT process is 100 s.

Cubic specimens (Fig. 1(b)) of the untreated and the treated steels were prepared by grinding, mechanical polishing and electrolytic polishing (6 vt% perchloric acid+1 vt% glycerin+93 vt% ethyl alcohol, 16 V, 0.5 A, 30 s). The cross-sections were observed by EBSD associated with Hitachi S3400N FESEM. The scanning step is 0.80 μm. The EBSD results in this paper were analyzed by TSL OIM Analysis 6 software. According to the working principle of the TSL OIM Analysis 6 software, the image quality (IQ) reflects the intensity of the Kikuchi signal. The Kikuchi signal is poor when the storage energy and dislocation density are high, which results in low IQ values and dark IQ images [34]. Such images as phase images, boundary images and quaternion misorientation images were superimposed contrast IQ.

Table 1
Chemical composition of AISI304 stainless steel (wt%).

Fe	Cr	Ni	Mn	Si	Co	V	C	P	Al	Cu	S
72	17.9	8.06	1.23	0.501	0.226	0.084	0.06	0.045	0.031	0.029	0.011

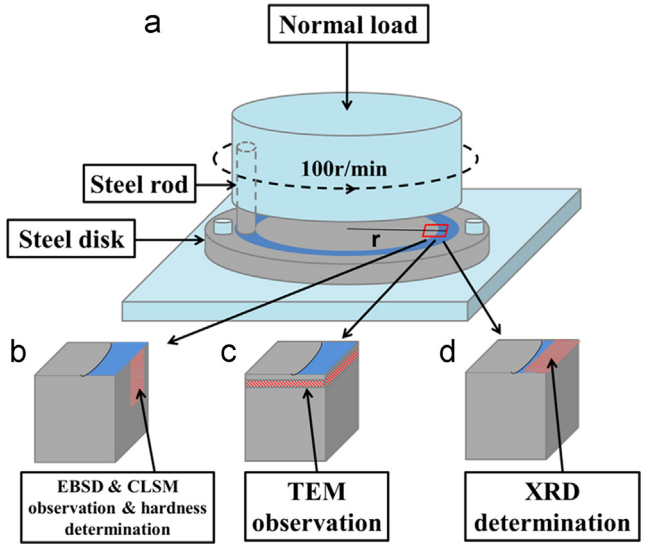

Fig. 1. Schematic diagram of SMAT process and experimental specimens.

Cubic specimens (Fig. 1(b)) of the untreated and the treated steels were also prepared by grinding and mechanical polishing. Then the cross-sectional hardness was determined by FM-700 Vickers with the loading force of 10 gf and the loading time of 10 s. The hardness determinations were started from the surfaces along the depth direction.

The foils at different depths (Fig. 1(c)) of the treated specimens were prepared by grinding and ion beam thinning (7 vt% perchloric acid+93 vt% glacial acetic acid, 25 V, 9 °C), and then were observed by JEM-2010 TEM.

Cubic specimens of the untreated and the treated steels (Fig. 1(d)) were examined by D/max-2500/PC X-ray diffractometer (XRD) with Cu Kα radiation (40–120°, 1°/s) to determine the phase constitution and the mean grain size of the surfaces. Because the SMAT-affected region is very small, the XRD specimen of the 600 N-treated steel was only prepared by ultrasonic cleaning, while that of the untreated steel was prepared by descaling.

Cubic specimens (Fig. 1(b)) of the 600 N-treated steel were prepared by grinding, polishing and corrosion (25 vt% nitric acid+75 vt% hydrochloric acid), and then were observed by LEXT/OLS3100 confocal laser scanning microscope (CLSM).

3. Results

3.1. Cross-sectional observations of specimens with different loads

Fig. 2 shows the cross-sectional IQ images of the untreated and the treated specimens. Because the storage energy of the grain boundaries is high, the grain boundaries are shown as black in Fig. 2. Fig. 2(a) shows a bright IQ image of the untreated specimen, which consists of equiaxed grains as well as a large number of annealing twins. Fig. 2(b)–(e) shows the cross-sectional images of the treated specimens, in which the right borders are the treated surfaces. It can be observed that Fig. 2(b)–(e) can be divided into