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THE DYNAMIC PHASE TRANSFORMATION AND FORMATION OF NANOCRYSTALLINE STRUCTURE IN SUS304 AUSTENITIC STAINLESS STEEL SUBJECTED TO HIGH PRESSURE TORSION

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Abstract. SUS304 austenitic stainless steel was deformed by high pressure torsion (HPT) at room temperature at different the rotation speed. By using X-ray diffraction (XRD) and transmission electron microscopy (TEM), the quantitative phase analysis and microstructural evolution along the progress of HPT were investigated. The present results show that the deformation-induced dynamic phase transformation (DPT) not only from austenite (γ) to martensite (α') but also α' to γ occurred and equaixed nanocrystalline γ grains are generated by HPT at high rotation speed. It is proposed that in austenitic stainless steel nanocrystalline γ grains structure is formed by combination of forward ($\gamma \rightarrow \alpha$') and reverse ($\alpha' \rightarrow \gamma$) DPT induced by HPT at high strain rate.

1. INTRODUCTION

HPT has been attracted considerable scientific interesting due to its outstanding ability of hardening and grain refining [1]. As the most common engineering materials, iron and carbon steels have been employed to study the changes in microstructure and properties after HPT treatment. It has been gradually realized that the nanometer structure is difficult to achieve in iron although large strain and strain gradient can be imposed by HPT [2]. However, nanocrystalline ferrite with the grain size of 10-20 nm has been obtained in the carbon steels by applying the similar or less amount of strain by HPT [3]. Comparing HPT-processed iron with carbon steels, one pronounced difference is the phase transformation during deformation (so-called dynamic phase transformation, DPT) which occurs only in carbon steel [3]. Recently, a number of studies have been reported that the phase transformation occurs during SPD in hard turning [4], ball milling [5], high speed drilling [6] and HPT [3,7], and the final structure has been characterized as equiaxed nanocrystalline grains. These results suggest that the DPT seems a favorable factor to attain nanocrystalline structure by deformation. SUS304 is one of the metastable austenitic stainless steels that are widely used in many machine components. Deformation induced martensitic transformations has been observed at ambient temperatures. In the present study, the SUS304 stainless steel processed by HPT was investigated to understand the roles of DPT on the formation of nanocrystalline structure.

2. EXPERIMENTAL PROCEDURE

The material used in present study was a metastable austenite stainless steel SUS304 having a composition of 0.05 C, 18.3 Cr, 8.5 Ni, 0.6 Si, 1.0

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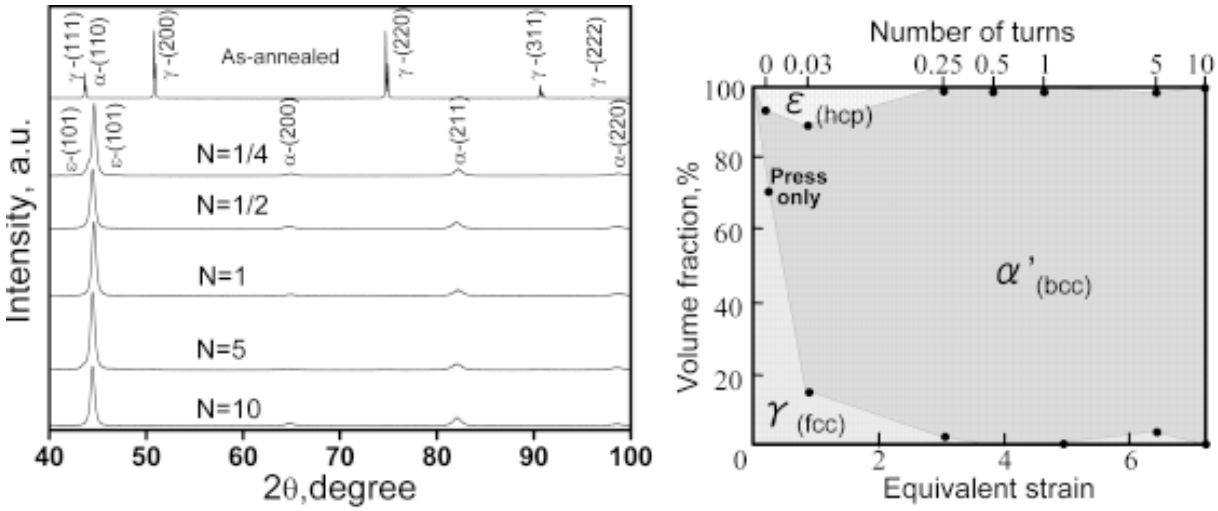

(a) (b)

Fig. 1. X-ray spectra and volume fractions of composed phases in the specimens before and after HPT at rotation speed of 0.2 rpm. (a) X-ray spectra, (b) volume fractions of composed phases.

Mn, 0.010 S, 0.03 P (in mass %) and balance Fe. Before HPT, the bulk materials were annealed at 1323K for 3.6 ks and then quenched into ice water to obtain fcc austenite phase. The disk samples for HPT treatment are 0.85 mm in thickness and 10 mm in diameter. HPT deformation was performed at room temperature under an imposed pressure of 5 GPa. The value of equivalent strain ε_e were estimated using the equation

$$\varepsilon_e = \frac{1}{\sqrt{3}} \cdot \frac{2\pi RN}{t}, \quad (1)$$

where R is the distance from the sample centre, N is the number of the anvil rotations and t is the thickness of sample. Thus the large number of turns and high rotation speed are directly related to the high strain and strain rate, respectively. The specimens were processed by HPT for 0.03 ~ 50 turns at rotation speed of 0.2 rpm or 5.0 rpm and characterized by XRD and TEM. The quantitative phase analysis was carried out by using Rietveld software QUANTO. The TEM observations were done at approximately 3 mm away from the sample center.

3. RESULTS

3.1. XRD analyses of HPT processed SUS 304 stainless steel

Fig. 1a shows the XRD spectra taken from the specimen before and after HPT processing at rotation speed of 0.2 rpm. The volume fractions of composed phases obtained from XRD spectra are show in Fig. 1b as a function of equivalent strain at the edge of sample. It can be seen that the deformation induced martensitic transformation, i.e., DPT, occurred and the bcc α'- and hcp ε- martensite were introduced with the expense of γ. In the early stage of HPT deformation, the volume fraction of γ decreases and the volume fraction of α' and ε increases rapidly. After 0.03 turns (ε_{eq} = 1.26), the volume fraction of ε reached to 11%. With the further deformation, the amount of ε starts to decrease and the volume fraction of α' continuously increases. After 0.5 turns (ε_{eq} = 15.46), nearly 100% of α' formed and almost no change with further deformation.

The XRD spectra and volume fractions of composed phases in the specimens HPT processed at rotation speed of 5.0 rpm are shown in Figs. 2a and 2b, respectively. It is seen that the volume frac-

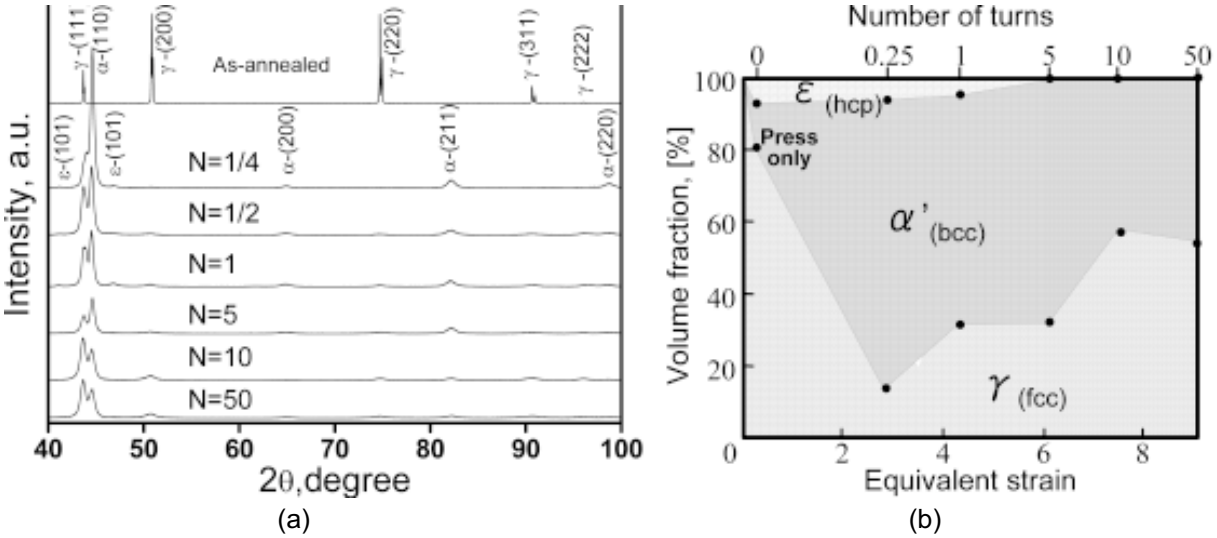

Fig. 2. X-ray spectra and volume fractions of composed phases in the specimens before and after HPT at rotation speed of 5.0 rpm. (a) X-ray spectra, (b) volume fractions of composed phases.

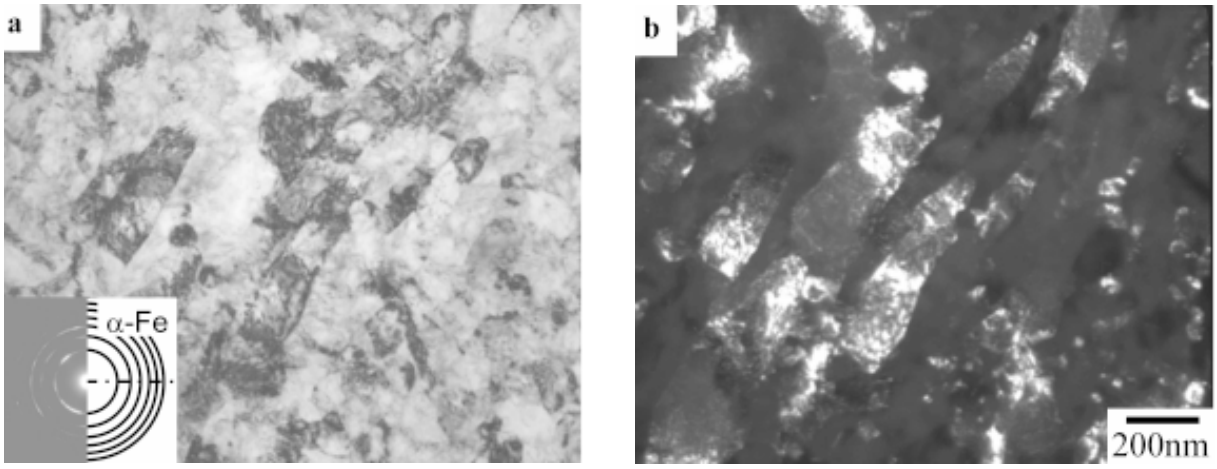

Fig. 3. TEM images after HPT treatment at the rotation speed of 0.2 rpm for 10 turns. (a) bright field image and corresponding SADP, (b) corresponding dark field image.

tion of γ decreases rapidly with the imposed strain and reaches the minimum value of 15% at equivalent strain of 3.0, and then increases with the further deformation. After 10 turns (ε_{eq} = 7.2), the volume fraction of γ reaches 59%. In contrast, the amount of the α' increases rapidly with the strain and reaches the maximum value of 79% after 0.25 turns (ε_{eq} = 3.0), and then decreases with further deformation. The volume fraction of ε phase is as high as 7% after pressing (before rotation) and then gradually decreases thereafter. After 5 turns of HPT, ε phase is hard to be detected by XRD analyses. These results suggest that the forward ($\gamma \rightarrow \alpha'$) and reverse ($\alpha' \rightarrow \gamma$) DPT occurred during HPT at the high strain rate in SUS304 austenitic stainless steel.

3.2. The microstructures of HPT processed SUS 304 stainless steel

The microstructure of HPT processed sample at different the rotation speeds was investigated by TEM. Figs. 3a and 3b show the typical bright-field and corresponding dark-field TEM images, respectively, after HPT treatment at rotation speed of 0.2 rpm for 10 turns. The microstructures are characterized by elongated grains with the about 200 nm

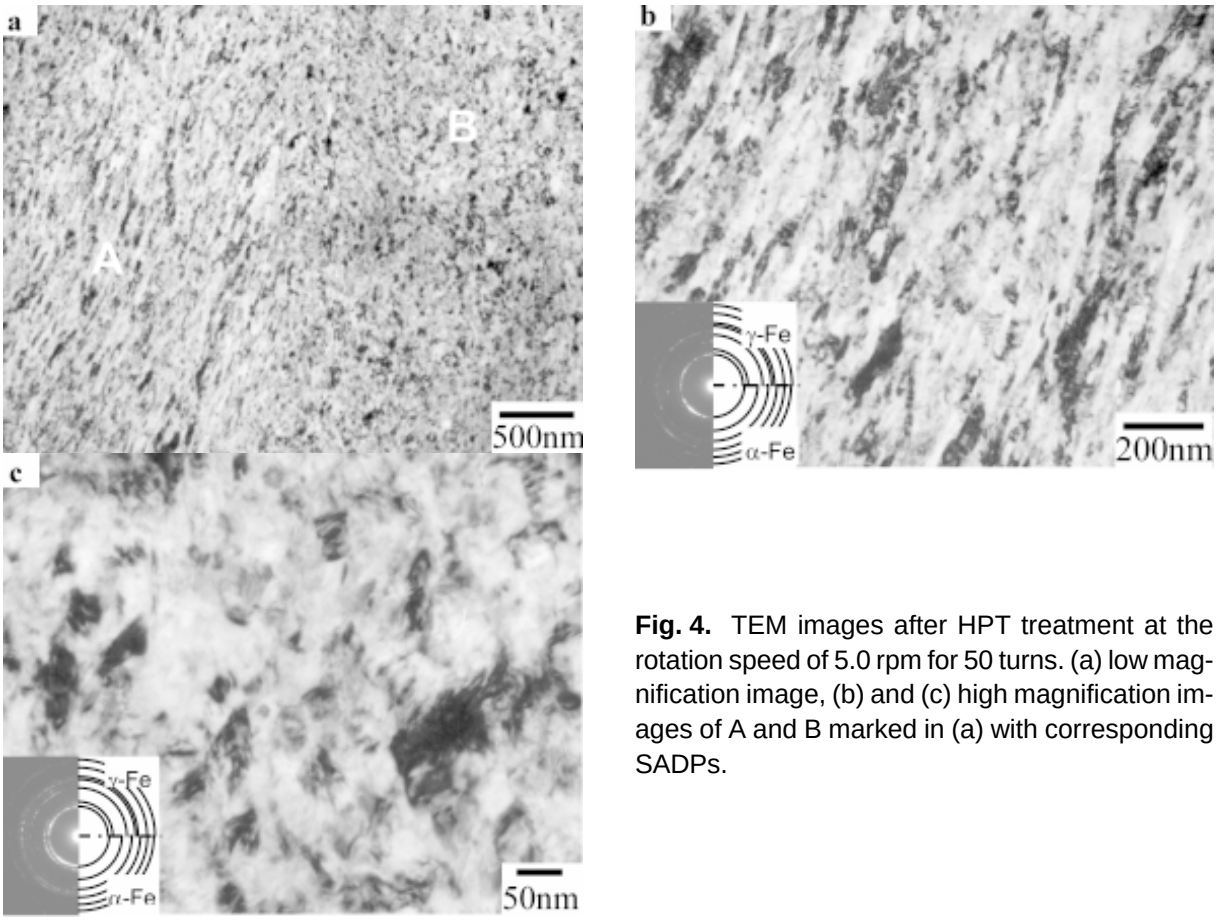

Fig. 4. TEM images after HPT treatment at the rotation speed of 5.0 rpm for 50 turns. (a) low magnification image, (b) and (c) high magnification images of A and B marked in (a) with corresponding SADPs.

in width and several 100 nm in length. High densities of dislocations are seen inside grains. The corresponding selected-area diffraction pattern (SADP) shows strong diffraction rings from bcc martensite (α'). Fig. 4 presents the bright-field TEM images of the HPT processed sample for 50 turns at rotation speed of 5.0 rpm. Two kinds of microstructures those of elongated and equiaxed grains in nanometer scale can be identified in the regions marked A and B in Fig. 4a. Fig. 4b is the high magnification image taken from the region A. The microstructure is mainly delineated by elongated grains with several 10 nm in width and several 100 nm in length. The corresponding SADP shows the strong diffraction rings from bcc martensite (α') and some weak rings from the fcc austenite (γ). The high magnification image taken from the region B is given in Fig. 4c. The equiaxed grains with the average size of several 10 nm are observed. The corresponding SADP indicates that these nanocrystalline grains are mainly composed of γ and a small amount of α'. It is further noted that many grains contains planar parallel defects that are considered to be deformation twins of γ.

4. DISCUSSION

The mechanism and microstructural aspects of the deformation induced transformation from γ to α' in austenitic stainless steels have been studied extensively [8-13]. It is generally considered that the α' nucleated at the shear bands intersections which are in the forms of ε, mechanical twins and stacking-fault bundles. It has been reported that the number of α' embryo increases with the number of intersections and the number of the intersections increases with imposed strain [14]. In the present study, substantially large strain and strain gradient

are introduced by HPT. A large strain gradient is associated with high density of geometrical necessary dislocations which are hard to annihilate. A large strain is beneficial to produce high density of the defects like deformation twins and stack faults. These defects can produce a great number of intersections and hence the effective nucleation site for α'. Therefore, the amount of α' is substantially increased by HPT which can introduce extremely large strain and large strain gradient. As a result, very fine α' grain is obtained after DPT from γ to α'. Moreover, the grains can be further refined to submicron size though the interaction of high density of dislocations.

The equiaxed nanocrystalline austenite grains are observed in the HPT processed samples after many turns at a high strain rate. XRD analyses clearly show that the deformation-induced forward (γ → α') and reverse (α' → γ) DPT occurred. To the best of the authors' knowledge, there has no report on the deformation-induced of forward (γ → α') and reverse (α' → γ) DPT in stainless steel during deformation at the room temperature. The extremely high strain imposed in the present study might be one important reason. Such a reverse transformation from bcc to fcc structure has also been reported recently during HPT of a carbon steel at room temperature [3]. It has been proposed that the driving force for this reverse martensitic transformation is provided by Gibbs free energy of ultra-fine bcc grains together with very high shear stresses [3]. Besides these factors, it is considered that adiabatic heating during HPT contributes to induce the reverse transformation. From the XRD analysis, it is found that the reverse transformation appears after imposing a large strain at high strain rate. The temperature measurements of the specimens by the thermocouple put in the anvil locating at 1mm away from the specimen surface during HPT indicated that a large temperature rise of about 200K at rotation speed of 5.0 rpm and a small temperature rise of about 10K at rotation speed of 0.2 rpm, respectively. The pronounced temperature rise at the high strain rate suppresses the forward transformation (γ → α') and promotes the reverse transformation from the α' to γ. Due to the reverse transformation from the ultrafine α' to γ, the further grain refinement is probably achieved. Deformation twins in fine g may also assist further grains refinement. At last, the equiaxed nanocrystalline γ grains were produced. Base on the present experimental results, it is proposed that in austenitic stainless steel the nanocrystalline γ structure is formed by combination of forward (γ → α') and reverse (α' → γ) DPT induced by HPT at high strain rate.

5. CONCLUSION

In SUS304 austenitic stainless steel, the equaixed nanocrystalline γ grains are generated by HPT at high rotation speed. While only the elongated submicron α' grains are observed at low the rotation speed. The XRD analyses clearly show that the deformation-induced forward (γ → α') and reverse (α' → γ) DPT occurred during HPT at high rotation speed instead of one-way DPT from γ to α' at low the rotation speed. It is proposed that in austenitic stainless steel the nanocrystalline γ grains structure is formed by combination of forward (γ → α') and reverse (α' → γ) DPT induced by HPT at high strain rate.

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