MICROSTRUCTURE EVOLUTION AND MECHANICAL PROPORTIES OF SUS304 BY ULTRASONIC AND AIR BLAST SHOT PEEING

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ABSTRACT

The objective of this work is to investigate the evolution of microstructures and resulting mechanical properties in SUS304 stainless steel treated by air blast and ultrasonic shot peening. X-ray diffractometer, optical microscope, scanning electron microscope and transmission electron microscope with energy dispersive spectrometer were used for microstructure analyses. Microhardness and abrasive wear test were carried out for the determination of the mechanical properties. A nanostructured surface layer with grain size of ~ 20 nm and mechanical twin were observed after shot peening. As the depth into the matrix increases, the multidirectional mechanical twins were gradually replaced by single-direction twins. The α -martensite and very few $M_{23}C_6$ precipitates were observed after air blast shot peening. The deformation induced α -martensite transformation appeared to occur in twin-twin intersection only after ultrasonic shot peening. The microhardness increased due to deformation induced martensite transformation and nanocrystalline surface structure. The improvement of wear properties after shot peening results from work hardening and nanocrystalline surface layer generation.

KEY WORDS

SUS304, Air blast shot peening, Ultrasonic shot peening, Nanocrystalline, Hardness, Abrasive wear

INTRODUCTION

In many cases, failures of a work piece may initiate at the surface, such as fatigue, wear and corrosion. So nanocrystalline (NC) materials (grain size less than 100 nm) show some attractive novel properties, such as higher corrosion resistance and wear resistance (X.P. Yong, 2003; J.L. Liu, 2007; T.S. Wang, 2006; I. Altenberger, 1999). Surface nanocrystallization has been achieved in various metallic materials by severe plastic deformation (SPD) (N.R. Tao, 1999; T.S. Wang, 2007; G. Liu, 2001; H.W. Zhang, 2003; G. Liu, 2000; Y. Todaka, 2004). The austenitic stainless steel is susceptible to martensite transformation under plastic deformation. For instance, the modes of γ (fcc) $\rightarrow \epsilon$ (hcp) $\rightarrow \alpha'$ (bcc) and γ (fcc) \rightarrow deformation twin $\rightarrow \alpha'$ (bcc) were proposed to be the transformation mechanisms. In both cases, the crystallographic relationships among γ , ϵ and α' followed the Kurdjumov-Sachs orientation

relationships: $\{111\}_{\gamma}$ // $\{0001\}_{\varepsilon}$ // $\{011\}_{\alpha'}$ and $\langle 110\rangle_{\gamma}$ // $\langle 1120\rangle_{\varepsilon}$ // $\langle 111\rangle_{\alpha'}$ (C.X. Huang, 2007; H.C. Choi, 1999; J.Y. Choi, 1997). In this study, SUS304 stainless steel, of which the stacking fault energy is 21 mJ/m² (M. Hadji, 2002) was chosen for shot peening (both air blast shot peening, ABSP, and ultrasonic shot peening, USSP) with an eventual aim for application of the treated parts to power plants. The

objective of this work is to investigate the evolution of microstructures and mechanical properties by ABSP and USSP in austenitic stainless steel.

METHODS

The material used in this investigation was SUS304 stainless steel (70x50x10mm³). The specimens (Table 1) were fabricated by ABSP and USSP. The crystal structures of the samples were examined using an X-ray diffractometer (XRD). The specimens were etched with a solution of HCl+HNO₃+H₂O₂ for analyses using optical microscope (OM) and scanning electron microscope (SEM). For transmission electron microscope (TEM) observation, specimens were ground to 100µm in thickness, punched as 3mm discs, and then twin jet electropolished with an electrolyte of 95% acetic acid and 5% perchloric acid. Statistical evaluation of the area fraction and mean diameter of precipitates were carried out by using Image-Pro plus software on the TEM micrographs. The average grain size (D) and mean microstrain ($\langle \varepsilon^2 \rangle^{1/2}$) were calculated using the following equations (S.Y. Ma, 2005):

$$\langle \varepsilon^2 \rangle^{1/2} = \beta_g^3 / (4 \tan \theta)$$

 $D = \lambda / (\beta_c^f \cos \theta)$

where θ is Bragg angle, λ is the wavelength of Cu K α_1 , β_g^f is Gauss integral broadening and β_c^f is Cauchy integral broadening. Microhardness and abrasive wear tests (ASTM G65) were carried out to study the mechanical properties.

 Table 1. Classification of samples. Table 2. Average grain size and mean microstrain of the surface layer after shot peening

Treatment	Specimen
As received	SUS304
SUS304 + ABSP	ABSP304
SUS304 + USSP	USSP304

Specimen	D (nm)	<ε ² > ^{1/2} (%)
ABSP304	22 ± 3	0.4181 ± 0.004%
USSP304	27 ± 4	0.3151 ± 0.004%

RESULTS AND DISCUSSION XRD study

Figure 1 shows XRD profiles of SUS304 and shot peened samples. It can be found that the as received samples consist of mono-austenite (fcc structure, a=0.359 nm). The formation of α '-martensite (bcc structure, a=0.287 nm), characterized by (110)_{α '}, (200)_{α '}, (211)_{α '} and (220)_{α '} peaks, was detected after shot peening. The average grain size and mean microstrain of the peened samples were calculated in terms of the diffraction line broadening of Bragg reflection peaks using equation (1) and (2) and the results are listed in Table 2. The average grain size was found to be about 22 nm and 27 nm after ABSP and USSP, respectively. It's obvious that both ABSP and USSP have been successfully applied in achieving surface nanocrystallization.

Microstructural characterization

The typical microstructure of SUS304 is shown in Figure 2, in which the average grain size was determined as about 80 μ m. Figure 3 shows the typical cross-section morphology of shot peened samples. The microstructure consists of mechanical twins, δ -ferrite and small volume fraction of carbides. With depth increasing, multiple-direction mechanical twin near the surface are gradually replaced by single-direction, while the densities of mechanical twins decrease. The distance between mechanical twins in ABSP is shorter than that in USSP at same depth due to the higher strain in ABSP. The mechanical twins disappear at about 160 μ m beneath the top surface.





Fig. 1. XRD profiles of specimen.

Fig. 2. OM image of SUS304.



Fig. 3. OM (a, b) and SEM (c-h) images: (a),(b) are cross-sectional observation of ABSP304 and USSP304; (c),(d) are area A in (a),(b); (e),(f) are area B in (a),(b); (g),(h) are area C in (a),(b) respectively; the arrow marks the mechanical twins consisting of multi or single directions.



Figure 4(a) shows TEM image of as received sample. The stacking fault and some dislocations can be seen clearly. The inset, selected area diffraction pattern (SADP) indicates that the matrix is austenite. The top surface layer after ABSP and USSP is shown in Figure 4(b) and (c). The mean grain size is about 20-30 nm by averaging over several TEM micrographs yielded. This result is close to the results calculated from XRD. This nanograin morphology is quite similar to those formed with other SPD processes (R.Z. Valiev, 2000). Figure 4(d) and (e) show the typical intersection of twins in ABSP304 and USSP304. The ABSP304 have thinner twin lamellae compare to USSP304. Inset is the corresponding SADP indicating that some of these blocks are of bcc martensite phase. It means that α '-martensite is formed at intersections of two mechanical twins. The size of martensite is depending on the size of twins operated. In the surface layer, the strain causes the formation of nanometer thick mechanical twins that generate nanometer sized parts. So a refined structure starts to be formed in to the nanometer scale. Strain induced martensite transformation takes place at the same time (H.W. Zhang, 2003). Figure 4(f) shows

the precipitate on grain and twin boundaries generated after ABSP, the corresponding SADP indicating that these precipitates are fcc structure $M_{23}C_6$. The mean diameter and area fraction of $M_{23}C_6$ is about 240 nm and 0.5%. It should be pointed out that the precipitation of $M_{23}C_6$ makes the material susceptible to the α '-martensite formation (L.F.M. Martins, 1998). Figure 4(g) reveals the twin-matrix alternative lamellar structure in USSP304, a high density of dislocation can also be observed inside the twins and close to their boundaries. The corresponding SADP shows a typical fcc twin. Same phenomenon also observed in ABSP304.

Mechanical properties and strengthening mechanisms

Figure 5 shows the mircohardness result after USSP and ABSP. As the distance below the treated surface increases, the microhardness decreases to approach the microhardness of the matrix (~200 Hv). The curve decreases gently after the depth reaches about 160 μ m. This agrees with the observation of cross-section images. The high hardness value can be explained by the presence of martensite at the top surface. With an increase of the depth from the top surface, strain amplitude and strain rate decrease accompanied by a significant reduction of the martensite transformation, so the hardness decreases with increasing of depth. Further more, a strong refinement of the grain size can lead to additional increase of strength due to the Hall-Petch relationship (T. Roland, 2007), which relates the hardness of a material, H_{u} , to the grain size, d, through the expression

$$H_{v} = H_{0} + \frac{K_{h}}{\sqrt{d}}$$
(3)

where H_0 and K_h are appropriate constants. After USSP and ABSP, the surface of SUS304 becomes NC microstructure. So the large reduction in grain size is reflected in the significant increase of the hardness value. The microhardness of ABSP304 (368.8 Hv) is little higher than USSP304 (348.8 Hv) because the grain size of ABSP304 (~22 nm) is smaller than USSP304 (~27 nm).



Fig. 5. Microhardness variation along depth from the surface (Left). Fig. 6. Wear resistance relative to load of SUS304, ABSP and USSP (Right).

Figure 6 shows the wear resistance improved after ABSP and USSP as expected. The improvement is a little bit higher for the specimens of ABSP, which matches well with the microhardness in Fig 5. This indicated the smaller grain size due to higher deformation of ABSP (Table 2) resulted in the higher hardness and higher wear resistance compared to those of USSP. It is well known that α '-martensite has a strong effect on work hardening of austenitic stainless steels (J. Talonen, 2005). If work hardening is previously done, it may give effect to the wear resistance of materials. Microstructure refinement is expected to increase hardness, which can be

used to roughly explain the tendency of abrasive wear resistance according to Rabinowicz's abrasive wear equation (W.L. Yan, 2007), i.e.

$$W = K \frac{P}{H}$$
(4)

where W is the wear volume per unit sliding distance, P the applied load, H the hardness of the worn surface and K is the wear coefficient. The increased wear resistance can be attributed to the increased hardness by shot peening. And also the nanocrystallized microstructure tends to increase the wear resistance of the material.

SUMMARY

- (1) Both ABSP and USSP process induce the NC in the SUS304 stainless steel.
- (2) The multi-direction mechanical twins replaced by single-direction twins with depth increasing. The narrower twins in ABSP304 due to bigger strain.
- (3) The nanograined microstructure with size of about 27 nm and deformation induced α '-martensite can be produced by USSP. In case of ABSP, the average grain size of top surface is about 22 nm. The α '-martensite and few M₂₃C₆ precipitate on grain and twin boundaries are generated after ABSP. The α '-martensite transformation occurs in twin-twin intersection.
- (4) The microhardness increased due to martensite transformation and NC surface generation. The improvement of wear properties after shot peening results from work hardening and NC surface layer.
- (5) The higher deformation and microstrain caused the increase in the wear resistance in the ABSP specimens.

ACKNOWLEDGMENTS

This project was funded by Electric Power Industry Foundation Fund of The Ministry of Commerce, Industry and Energy via Korea Electric Power Research Institute. The authors gratefully acknowledge the sponsor.

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