X-ray Diffraction Rietveld Analysis of Shot-peened Duplex Stainless Steel during Isothermal Annealing

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Abstract:
Lattice parameters of shot-peened duplex stainless steel (DSS) S32205 samples at isothermal annealing are obtained by using modified Rietveld method. Lattice parameters of both α-phase and γ-phase are found in decrease with prolonged annealing time. The weight fraction of α-phase, microstructure of both α-phase and γ-phase are discussed during isothermal annealing. The dislocation densities of shot-peened DSS samples at isothermal annealing are calculated on the basis of Williamson-Smallman approach using modified Rietveld method.

Keywords: Duplex stainless steel; Shot peening; Thermal relaxation; Rietveld method; Lattice parameter

Introduction

Transmission electron microscopy (TEM) and X-ray diffraction (XRD) techniques are now routinely used to obtain information on microstructure. In recent years, TEM with impressive resolution power on a local scale has been most widely used to investigate microstructure, especially the dislocation distribution. However, the possibility of loss and rearrangement of dislocations during thin-foil preparation and/or measurement and a poor statistical basis must be given due consideration [1-3]. X-ray line broadening analysis (LPA) of deformed crystals has been developed theoretically [4, 5] and experimentally [6-8] to a considerable extent, and this method has been proved to be suitable to obtain dislocation densities that correlate well with TEM data. However, the use of this technique requires careful experimentation, sample preparation and calculation. Anisotropy in peak broadening has been attributed to various factors such as crystallite shape, anisotropic strain, planar faults especially stacking faults or twinning [11]. However, we have determined the anisotropy in the domain size and strain as a function of the annealing DSS in the present investigation. In this study, the lattice parameter, domain size and microstrain in shot-peened DSS S32205 at elevated temperature are obtained by X-ray diffraction Rietveld method. The calculation of dislocation density from parameters such as domain size and strain using Williamson-Smallman [9, 10] approach is presented.

Experimental procedure

The material used in this investigation is DSS S32205 provided by Shanghai Baosteel Group Corporation. Microstructure containing nearly equals amounts of α-phase and γ-phase produced when material was solution-mill annealed (1050 °C, 2.5 hours) and then water-quenched. The samples studied in this work are 20 mm in diameter and 2 mm in thickness which were cut directly from 10 cm-diameter ingots followed by polishing. The chemical composition is C (0.029), S (0.006), Si (0.42), Mn (1.27), Cr (22.10), Ni (5.17), Mo (3.10), N (0.18), P (0.021), and the rest is Fe (all in wt. %). The arc height of Almen specimen (A type) of 0.36 mm is set as SP intensity in this work, which is influenced by jet pressure of nozzle, peening time and average diameter of shot balls. In order to investigate the recovery and recrystallization behavior of shot-peened DSS, isothermal annealing treatment was carried out at 600 and 650 °C, respectively. The samples were sunk in the alumina powder for even thermal environment during annealing. After cooling to ambient temperature, XRD investigation was conducted in the same zones of samples by Rigaku Ultima IV X-ray diffractometer with Cu-Kα radiation (λ = 1.54056 Å) and D/tex 1D high-speed detector operated at 40 kV/ 30 mA. XRD was analyzed using Materials Analysis Using Diffraction (MAUD) software [12]. Instrumental parameters like 2θ correction, peak asymmetry, and peak broadening parameters (U, V, W)

[13,14] were determined from Si standard received from Rigaku, Japan, which does not contribute to size and strain broadening according to the procedure indicated by Lutterotti et al. [12,15] and latter reported by several researchers [16-19]. α-phase (ao = 2.881 Å, Im-3m) and γ-phase (a_o = 3.596 Å, Fm-3m) calculations were performed using lattice parameters of pure Fe as an initial structural model for Rietveld refinement of the shot-peened samples. Popa anisotropic size-strain model [20] was adopted and refined with other structural parameters. The domain size (D) and the root mean square (RMS) microstrain ($<\varepsilon^2>^{1/2}$) values were evaluated from the Popa model.

Results and discussion

A typical Rietveld plot of shot-peened DSS annealed at 600 °C for 60 min after refinement is shown in Fig. 1. It can be found that both α-phase and γ-phase exist in DSS after annealing at 600 □ for 60 min. The phase transformation of shot-peened DSS at elevated temperature is shown in Fig. 2. It can be obtained that weight fraction of α-phase decreases with increasing temperature and decreasing content of α-phase translates into γ-phase. Intensive α → γ phase transformation commences at 650 °C after 16-min annealing.

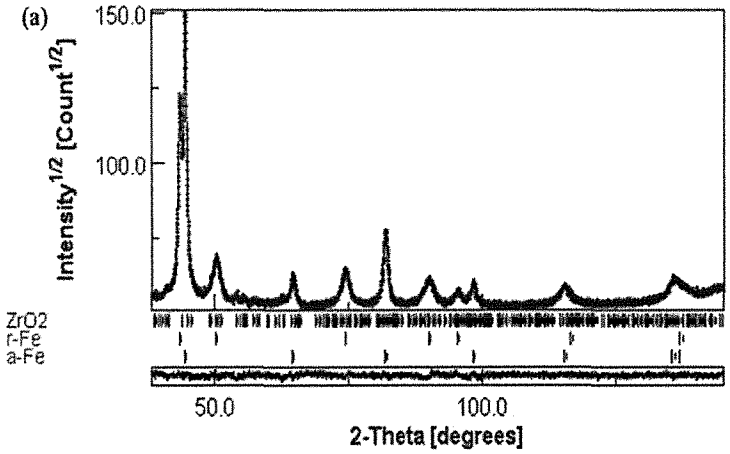

Fig. 1 Rietveld plot of shot-peened DSS annealed at 600 °C for 60 min after refinement: Sigma=1.42, Rp: 9.33%

Before SP, lattice parameters of α-phase and γ-phase in DSS obtained by X-ray diffraction Rietveld are 2.8791 and 3.6057 Å, respectively, however, they increase to 2.8858 and 3.6063 Å, respectively, after SP. In the absence of external load acting on sample, the variation of lattice parameter implies (i) the formation of solid solutions with C, N et al. interstitial atom and/or different size atoms to Fe as solutes in α-phase and γ-phase host, (ii) the introduction of macroscopic residual in-plane compressive stresses, or (iii) both (i) and (ii) [21-23]. The formation of solid solutions containing C, N et al. or Mo, Mn would increase the interplanar spacings of α-phase and γ-phase alloy, but this is unlikely to happen because SP is conducted at room temperature in this work and rapid diffusion at room temperature is impossible. Therefore, the lattice distortion can be concluded that SP processing has introduced large macroscopic residual compressive stress at the surface region of DSS.

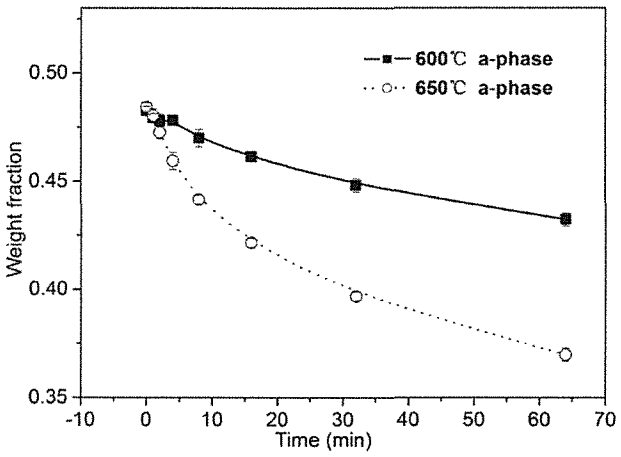

Fig. 2 Weight fraction of α-phase decreases with the increasing annealing time at different temperature.

Lattice parameters of α-phase and γ-phase as a function of annealing time at 600 and 650 °C are shown in Fig. 3. It is noticed that lattice parameters of both phases decrease with the prolonged annealing time at a fixed temperature, which is attributed to the relaxation of residual stress at 600 and 650 °C. As for γ-phase, the higher annealing temperature, the more obvious lattice-parameter decreases, this reveals that annealing temperature is a critical factor for decrease of lattice parameters. The process of thermal recovery and recrystallization in γ-phase is the reason that lattice parameter decreases at higher annealing temperature. However, in α-phase, it can be obtained that the decreasing rate of lattice parameter at 650 °C is less than that at 600 °C after annealing for more than 16 min, this may be attributed to interaction between α-phase and γ-phase in the process of thermal recovery and recrystallization.

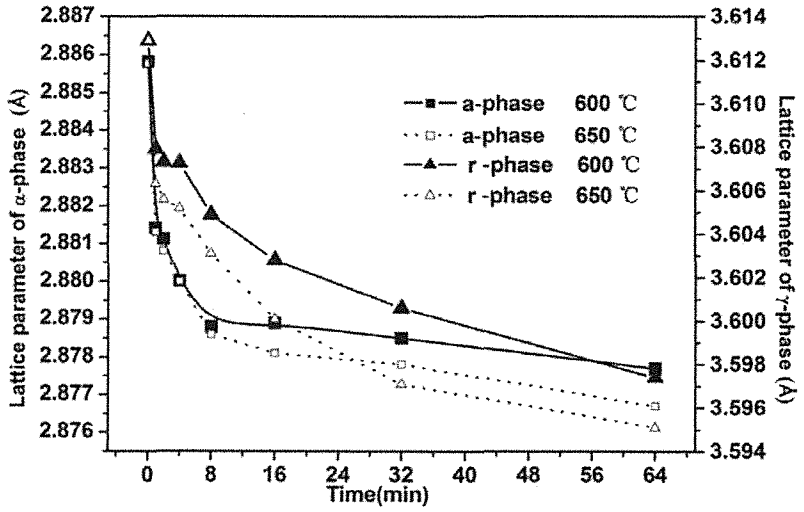

Fig. 3 The distributions of lattice parameters along the variation of annealing time at different temperature.

Domain size and microstrain can be obtained from the strongest peak {110} in α-phase and {111} in γ-phase as a function of annealing time at 600 and 650 °C. It is noticed that domain size increases and the microstrain decreases in the processes of annealing with prolonged annealing time. According to the results of domain sizes and microstrain, the dislocation densities in the surface deformation layers can be determined via Williamson method [9, 10], which can be expressed as below:

$$\rho = 2\sqrt{3}[<\varepsilon^2>]^{1/2} / [|\vec{b}|.D] \qquad (4)$$

Where ρ represents the dislocation density, $<\varepsilon^2>$ is the weighted average of ε^2 after multiple measuring, D is the domain size and $|\vec{b}|$ represents the mold of burgers vector in calculation. The burgers vectors are introduced as ref. [24]. Fig. 4 is the dislocation densities in α-phase and γ-phase versus the annealing time, indicating that the dislocation density decreases with prolonged annealing time. The typical dislocation density of shot-peened DSS before annealing is 2.3×10^{15} and $8.5\times10^{15}/m^2$ in α-phase and γ-phase, respectively. It must be mentioned that there is a significant decrease in the dislocation density with increasing temperature in DSS. It is noticed that the two factors, namely domain size and microstrain, collectively decrease the dislocation density slightly in the process of annealing. This is clearly understandable from domain size has increased significantly, however, the strain has been found to decrease with prolong of annealing time. The relaxation of microstrain and growth of recrystallization nucleus depends largely on the stored energy induced by SP [25]. High dislocation density in γ-phase means high store energy. During annealing, the stored energy was released for recovery and recrystallization, which is ascribed to the thermally activated gliding of dislocations in the thermal recovery and recrystallization process, and then dislocation density decreases.

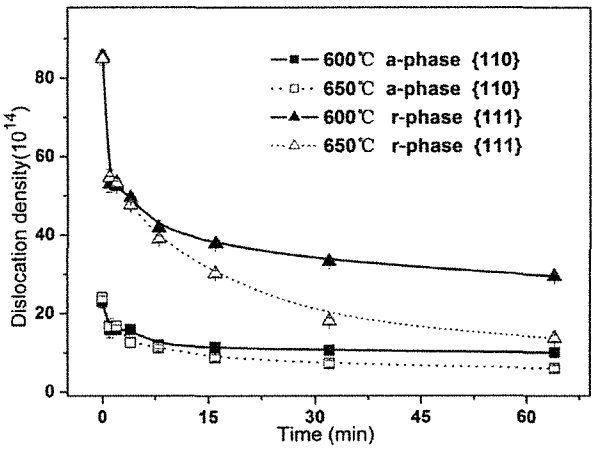

Fig. 4 Distributions of the dislocation densities in α-phase and γ-phase with the increasing annealing time at different temperature.

However, for all thermal treatments, the decrease of dislocation density in α-phase takes place at shorter time as same as γ-phase, as shown in Fig. 4, despite of the higher driving force induced by dislocation for recovery and recrystallization of γ-phase. This can be contribution to the decrease of dislocation density also correlates to diffusion activated phenomena, and the diffusion in α-phase is much faster than that in γ-phase [26]. For recovery and recrystallization, the lower stored energy during SP within α-phase is overcompensated by a higher diffusivity in the bcc lattice, resulting in the decrease of dislocation for shorter time in α-phase.

Conclusions

Lattice parameters of both α-phase and γ-phase in shot-peened DSS under isothermal annealing were obtained by using Rietveld method, and they were found to decrease with prolonged annealing time. The lattice-parameter decrease rate in α-phase at 650 °C is less than that at 600 °C after annealing at 16 min, which is contrary to the result found in γ-phase. Dislocation density of both α-phase and γ-phase in DSS were determined from domain size and microstrain values using Rietveld method. It has been observed that the dislocation density decreases as a function of annealing time due to the thermally activated gliding of dislocations in thermal recovery and recrystallization process.

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