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Investigation on the thermostability of residual stress and microstructure in shot peened SAF 2507 duplex stainless steel

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was precipitated in the deformed layer after isothermal annealing.

1. Introduction

Shot peening (SP) is widely used as a kind of mechanical surface treatments to improve the fatigue properties and wear resistance of metallic components by introducing the compressive residual stress (CRS) and work hardening [1–[4\]](#page--1-0). Duplex stainless steels (DSS) containing almost equal fraction of austenite and ferrite have full-blown applications in the petrochemical, seawater cooling and nuclear industries duo to their excellent corrosion resistance and mechanical properties. In order to enhance the overall performance and expand the application range of the alloy, SP has been implemented on the DSSs in recent years. Chen et al. [\[5\]](#page--1-1) investigated the residual stress and microstructure evolutions of 2507 DSS by multiple SP treatments and introduced high CRS into the surface layer without extensively enhancing the surface roughness. Selvabharathi et al. [\[6\]](#page--1-2) improved the tensile strength of the 2205 DSS by means of SP treatment. Sanjurjo et al. [[7](#page--1-3)] studied the effects of the shot peening on the fatigue behavior of 2205 DSS, they confirmed that SP can effectively improve the fatigue properties of the material.

It is considered that the beneficial effects of SP prevail, only if the near-surface CRS or work hardening remains stable under thermal exposure, cyclic loading and thermomechanical loading conditions [8–[10](#page--1-4)]. Therefore, the stability of residual stress, work hardening and microstructure refinement against thermal and mechanical loading is

critical to the fatigue characteristics and strength of shot peened materials. The relaxation behavior of residual stresses and microstructure in the shot peened material at elevated temperature has been relatively well investigated $[11-14]$ $[11-14]$. Previous investigations indicate that the relaxation is affected by kinds of factors, such as the initial residual stress state, material type and its microstructure, as well as the annealing temperature and time [[15,](#page--1-6)[16\]](#page--1-7). Meanwhile the Zener–Wert–Avrami function has been successfully applied to characterize the relaxation behavior of residual stress in dependence on the time and temperature in some types of steel [\[17](#page--1-8)–19]. It is well-established that the annihilation of metastable lattice defects, dislocation rearrangement and subgrain growth which driven by the reduction of stored energy are responsible for relaxation process.

However, there is little research available in the literature relating to the high-temperature behavior of the SP-processed DSSs. This is critical issue especially for the shot peened workpiece which serving under the elevated temperatures. It is the purpose of this work to investigate the thermal relaxation behavior of the shot peened SAF 2507 DSS at evaluated temperatures (600–750 °C) for different times. The thermal stability of surface residual stress and microstructure of peened 2507 alloy were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM) techniques.

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Table 1

2. Material and experimental procedures

Type SAF 2507 DSS supplied by Baosteel Co. (China) was investigated in this study. The obtained material was fabricated into hotrolled plate with a thickness of 30 mm. Then the steel plate was fully solution annealed at 1030 °C for 1 h and quenched in water. The chemical composition of the studied material is given in [Table 1.](#page-1-0) Specimens with a dimension of $20 \times 20 \times 8$ mm were cut from top layer of the steel plate and ground up to 800-grit SiC prior subjecting to SP process. After that, a three-step SP procedure was carried out by using air blast machine (Carthing Machinery Company, Shanghai, China). The first SP treatment was performed by injection using steel shot S230 (mean diameter 0.6 mm) at Almen intensity of 0.51 mm A. Then the dual peening operation was implemented using a smaller steel shot S110 (mean diameter 0.3 mm) at an intensity of 0.19 mm A. Finally, the dually peened specimen was blasted using ceramic beads B60 (mean diameter 0.125 mm) at an intensity of 0.11 mm A. Detailed SP parameters are listed in [Table 2.](#page-1-1) The benefit of the multiple SP over the traditional (one-step) SP is that it can develop deep layer of higher CRS without excessively increasing the surface roughness. After SP treatments, isothermal aging was carried out at temperatures of 600, 650, 700 and 750 °C for different times from 1 min to 2 h. While annealing, the samples were sunk in the alumina powders for even thermal environment, then samples were air cooled at a cooling rate of ∼8 °C/s.

Surface residual stresses after annealing were measured by X-ray stress analyzer (LXRD, Proto, Canada) using $\sin^2\!\psi$ method at ψ angles in the range -30° ≤ ψ ≤ 30°. The Mn K_a radiation with a wavelength of 2.10314 Å and Cr K_a radiation with a wavelength of 2.2897 Å were used to determine the austenite (311) and ferrite (211) diffraction peak, the stress constants are −328 and −289 MPa/deg. for ferrite and austenite, respectively. The surface XRD patterns were measured using Rigaku UItima IV diffractometer (Cu K_a radiation) with a D/tex1D highspeed detector. The voltage, current, scan speed and step were 40 kV, 30 mA, 2°/min and 0.01°, respectively. Microstructural observations were carried out using optical microscope (OM, Olympus-BX53), scanning electron microscope (SEM, Hitachi, S4800) and transmission electron microscope (TEM, JEOL, JEM-2100F). The cross section of specimens for OM and SEM was polished and then etched using a boiling solution of $10 g K_3Fe(CN)_6$, $15 g KOH$ and $100 ml$ distilled water. Samples for TEM studies were prepared by thinning from the unpeened side to thicknesses of ∼50 μm using SiC papers, followed by punching, dimpling and ion milling. Ion milling was accomplished using Gatan PIPS instrument at an accelerating voltage of 5 kV and angle of 10°. The depth profiles of the microhardness of the annealed specimens were performed using a Zwick hardness tester with experimental load of 25 gf and loading time of 15 s.

3. Results and discussion

The optical micrograph of the as-received SAF 2507 alloy is shown in [Fig. 1\(](#page--1-9)a). The microstructure is comprised of about 60% of γ phase (white phase) and 40% of α phase (gray phase). Aside from γ and α phase, the as-received material contains no precipitations like σ phase, $χ$ phase and $M₂₃C₆$ as confirmed by the XRD pattern in [Fig. 1](#page--1-9)(b). The surface morphology of the as-peened specimen is shown in [Fig. 2.](#page--1-10) It can be seen that the surface was severely deformed after triple SP, and the calculated average roughness was approximately 2.54 μm.

3.1. Relaxation of residual stress

In order to pick out the suitable isothermal annealing parameters, the response of residual stress at temperatures from 250 to 900 °C with holding time of 5 min was first determined before the isothermal annealing, and the results are illustrated in [Fig. 3](#page--1-11). The initial CRS on the surface introduced by SP was about -1020 MPa in γ phase and −850 MPa in α phase. As can be seen, the stress relaxation rate is strongly dependent on temperature. Higher temperature resulted in greater relaxation rate. After annealed at 600 °C for 5 min, the residual stress was about -723 and 728 MPa in γ and α phase, which was 29 and 14% less than the initial state, respectively. It is evident from [Fig. 3](#page--1-11) that α phase possesses higher stress relaxation resistance than $γ$ phase. After annealing at 900 °C, the residual stress diminished about 95% (−66 MPa) in γ phase and only 48% (−441 MPa) in α phase.

Temperatures in the range of 600–750 °C at intervals of 50 °C were chosen to conduct the isothermal annealing. The quantitative evolution of the surface stress as a function of annealing times is shown in [Fig. 4](#page--1-9). All specimens exhibited a noticeable stress relaxation after annealing. The stress reduction rate is time and temperature dependent. Two time stages can be distinguished concerning the relaxation: (i) Most of the stress reduction occurred within the initial 30 min. Comparing with the as-peened state, about 58, 73 and 95% of the CRS were released in γ phase after annealing at 600, 650 and 700 °C for 30 min. At 750 °C, the residual stress induced by SP in γ phase was completely disappeared. (ii) After isothermal annealing for 30 min, the surface stress decreased slowly with time and finally achieved stability. Comparing [Fig. 4\(](#page--1-9)a) with (b), it can be seen that stress relaxation rate in α phase is lower than that in γ phase. Only about 37, 54, 61 and 84% of the stress was diminished in α phase at 600, 650, 700 and 750 °C for 30 min. In addition, a considerable of thermal tensile stress was introduced into γ phase after annealing at 700 °C or higher temperature. These residual thermal stresses are thought to generate during cooling from elevated temperature. Since the coefficient of thermal expansion (CTE) of α phase is lower than that of γ phase (10.4 vs. 17.2 \times 10⁻⁶/°C, 100 °C) [[20\]](#page--1-12) and the unrelaxed CRS contributes to partially offset the generated tensile stresses, the surface stress in α phase remains in compressive state after annealing.

Thermal stress relaxation is a well-known phenomenon and is in dependence on the time and temperature. The relaxation behavior of residual stress can be described by Zener-Wert-Avrami function [\[9,](#page--1-13)[15](#page--1-6)]:

$$
\frac{\sigma^{RS}}{\sigma_0^{RS}} = \exp\left(-\left(C \cdot t \cdot \exp\left(-\frac{\Delta H}{kT}\right)\right)^m\right) \tag{1}
$$

Transform Eq. [\(1\)](#page-1-2) into the following form:

$$
\log\left(-\ln\frac{\sigma^{RS}}{\sigma_0^{RS}}\right) = m \cdot \log C + m \cdot \log t - \frac{m \cdot \Delta H}{\ln 10} \cdot \frac{1}{kT} \tag{2}
$$

Where $\sigma^{\rm RS}$ is the value of surface stress after isothermal annealing at T temperature for t time, σRS 0is the initial residual stress in the room temperature before annealing, k is Boltzmann constant, ΔH is the activation energy of stress relaxation, C and m are constant, which is in dependence on the corresponding relaxation mechanism. From Eqs. [\(1\)](#page-1-3) [and \(2\),](#page-1-3) a plot of log ($\text{-ln}(\sigma^{\text{RS}}/\sigma_0^{\text{RS}})$) as a function of log t at constant

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