# Microstructure Changes and Stress Relaxation of 18CrNiMo7-6 Steel after Triple Shot Peening During Isothermal Annealing

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## Abstract:

Variations of micro-structure, residual stress and micro-hardness for 18CrNiMo7-6 steel after shot peening (SP) against annealing temperature were studied. Riveted software 'Maud' was used to identify changes on the top surface micro-structure for 18CrNiMo7-6 steel after SP. Results show that top surface micro-strain of 18CrNiMo7-6 steel after SP decrease with the increase of annealing temperatures while domain size increases with increasing annealing temperatures. Compressive residual stress (CRS) at the top surface of 18CrNiMo7-6 steel after SP decreases with increase of annealing temperatures. Meanwhile, changing rate of microstructure and residual stress are bigger at higher annealing temperatures.

Keywords: 18CrNiMo7-6 steel; Shot peening; Residual stress relaxation; Microstructure

## Introduction

18CrNiMo7-6 is widely used in many industrial components, specifically in high-speed heavyduty gear manufacturing [1, 2]. Shot peening (SP) is one of the most commonly used mechanical surface treatments laye in order to improve the surface hardness and surface quality, this kind of material and [3, 4]. In the process of SP, impact indentations of the shot induce local plastic deformation at the surface, which can create beneficial CRS, increase dislocation population and induce both microstructure changes and work hardening near surface layers [5-7]. CRS, microstructure refinement and work hardening produced in peened layers can improve fatigue life of the material [8-11]. In previous works [12], authors have reported that triple SP with intensity of 0.50 + 0.30 + 0.15 mmA can more efficiently refine the domain size, improve CRS, micro-strain and micro-hardness of 18CrNiMo7-6 steel. In this work, 18CrNiMo7-6 steel after triple SP with intensity of 0.50 + 0.30 + 0.30 + 0.15 mmA is used as research object.

Now, researches about 18CrNiMo7-6 steel have almost focused on mechanical properties and microstructure characterization at room temperatures [3, 4, 12, 13]. But little investigation was made on characterization of recovery, and recrystallization behavior and thermal stability of 18CrNiMo7-6 steel after SP at increased temperatures. Meanwhile, XRDLPA is a well-known reliable and non-destructive technique. It is widely used to characterize residual stress, crystallite size, micro-strain, dislocation density etc. [14-20]. In this study XRDLPA refined software 'Maud' [21] was used to identify changes in micro-structure and relaxation behavior of residual stress on top surface of 18CrNiMo7-6 steel after triple SP during isothermal annealing was also discussed.

## Experimental

## Materials

Samples studied are manufactured from case-hardened steel 18CrNiMo7-6 (EN 10084). Its Chemical composition is: 0.170 C, 0.190 Si, 0.560 Mn, 1.520 Ni, 1.650 Cr, 0.320 Mo, 0.006 P, 0.003 S, 0.028 Al, 0.020 Cu, 0.002 Sn and balance Fe (weight%). All specimens ( $15 \times 15 \times 7$  mm) were austenitized at 950 °C for 50 hours, subsequently heated at 860 °C for 2 hours and quenched in oil, followed by tempering at 180 °C for 3 hours and cooling in air.

## SP and isothermal annealing treatments

In this study, SP treatments with intensity of 0.50 + 0.30 + 0.15 mmA were performed on an air blast machine (Carthing Machinery Company, Shanghai). Intensities of SP were measured

by arc height of Almen specimen (A type). Diameter of peening nozzle was 15 mm and distance between nozzle and sample was 100 mm. Triple SP is a process of going through SP treatment three times on the same surface of sample with different SP intensities and shots. Firstly, the sample was SP treated with intensity of 0.50 mmA and cast steel balls, then treated again under intensity of 0.30 mmA and cast steel balls, at last, the same surface were treated under intensity of 0.15 mmA and ceramic shots. Triple SP treatments can not only strengthen the sample surface, but also make the surface smoother after dual SP, so low SP intensities or small shots were selected to carry out on the second and third SP treatments. Cast steel balls with diameter of 0.6 mm and hardness of 610 HV and Al<sub>2</sub>O<sub>3</sub> ceramic balls with diameter of 0.3 mm and hardness of 700 HV were selected as shot media in SP techniques. Coverage of SP was 100%.

After SP treatment, isothermal annealing treatments were carried out by annealing flat specimens with temperatures between 200 and 650°C for annealing times of 5 minutes, respectively. During annealing, samples were buried in alumina powders in order to create a uniform thermal environment. After cooling in air, residual stresses and microstructures of all specimens were measured and analyzed by various measurements.

#### Measurements and calculations

XRD patterns were measured by the Rigaku Ultima IV diffractometer (Cu-K $\alpha$  radiation) with a D/tex1D high-speed detector. Voltage, current, scanning velocity and step are 40 kV, 30 mA, 2 °/min and 0.02 °, respectively. K<sub>a2</sub> components of Cu radiation were eliminated and backgrounds were corrected when analyzing. Residual stresses of the samples were measured by X-ray stress analyzer (LXRD, Proto, Canada) with Cr K<sub>a</sub> radiation (Ni filter). Voltage and current were 30 kV and 25 mA, respectively. Shifts of BCC {211}<sub>a</sub> peaks were detected in measurements and then residual stresses were determined by using sin<sup>2</sup> $\psi$  method [22, 23]. Peaks of {211}<sub>a</sub> lattice plane were measured at high 2 $\theta$  value of 156.4°, and the elastic constant of (1/2)s<sub>2</sub> = 5.81×10<sup>-6</sup> MPa<sup>-1</sup> was selected for stress determination. MAUD [21], a Rietveld software, was used to accurately fit the whole X-ray pattern for microstructural characterization, including grain size and micro-strain.

#### Results and discussions

#### X-ray diffraction analysis

XRD patterns of peened surface at different annealing temperatures have been shown in Fig. 1. It can be found that all specimens after annealing at different temperatures have only martensite state, and peak breadth decreases with increase of annealing temperature, which reflects the microstructure change of the surface of specimens at different annealing temperatures. In order to further study the effects of annealing on microstructure of the surface for 18CrNiMo7-6 steel after SP, whole peak diffraction profiles of triple SP treated samples after annealing at different temperatures have been analyzed via Maud software. The Rietveld plot of SP treated sample annealed at 200 °C obtained by Maud software was as shown in Fig. 2. Refinement error: sig = 1.51, Rw (%) = 0.65, Rexp (%)=0.38.

#### **Micro-structure characteristics**

Domain size and micro-strain of the top surface of triple SP treated samples at different annealing temperatures were shown in Fig. 3. It can be observed from Fig. 3 that domain sizes increase and micro-strains decrease with the increase of annealing temperatures, respectively. With the increase of annealing temperature from 30 °C to 500 °C, domain growth rate and micro-strain reduction rate are slower than that at above 500 °C. Intensity of thermal recovery and dynamic recrytallization increases as temperature increases. After SP process, elasticplastic deformation is introduced in the surface layer of samples. During isothermal annealing, micro-strain relaxation occurs and domain size increases. This happens in the processing of recovery and recrystallization, as both are driven by stored energy of deformed state (caused by SP in this paper) [20].

### **Residual stress distribution**

Fig. 4 indicates variations of residual stress on the top surface of 18CrNiMo7-6 steel before and after SP with annealing temperatures. In case of hardened 18CrNiMo7-6 steel before the SP, the residual stress decreases slightly with increasing in annealing temperatures, and almost constant CRS of approximately -100 MPa are present when the annealing temperature increases to 650 °C. Residual stresses of 18CrNiMo7-6 steel are generated during martensitic hardening by dislocations which are produced during diffusionless transformation and by solute carbon atoms which remain in their octahedral sites without diffusion [24]. The stored energy is smaller than energy changes associated with phase transformations and diffusions, so residual stresses of 18CrNiMo7-6 steel before SP change slightly increasing along with the increase of annealing temperatures. It is also shown that from Fig. 4 the surface of 18CrNiMo7-6 steel after triple SP have an obvious CRS, which is beneficial to the improvement of mechanical properties [8-11]. With increasing annealing temperatures, CRS decreases gradually owing to the recovery and dynamic recrystallization of deformation surfaces. And CRS has been closed to the value of the matrix without SP treatment when annealing temperatures increase to 650 °C. Meanwhile, the rate of CRS change in at high temperatures is greater than that of at lower temperatures.

## Conclusion

The residual stress relaxation behaviors and variations of micro-structure in the shot peened layer of 18CrNiMo7-6 steel during isothermal annealing were investigated. XRDLPA software 'Maud' was used to identify the change of micro-structure and residual stress relaxation behaviors of top surface for 18CrNiMo7-6 steel after SP was also discussed. Results shown that residual stress and micro-strain of top surface of 18CrNiMo7-6 steel decreases while annealing temperature increases along with an increase in domain sizes. Meanwhile, high integrity recovery occurs as change rates of each parameter increase at higher annealing temperatures.

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Fig. 1 The surface XRD patterns of 18CrNiMo7-6 steels after triple SP at different annealing temperature



**Fig. 2** The Rietveld plot of the SP treated sample annealed at 200 °C obtained by Maud software: sig =1.51, Rw = 0.65%, Rexp = 0.38%



Fig. 3 Variations of Domain sizes and microstrain obtained by Maud software with annealing temperatures



Fig. 4 Variations of top surface residual stresses for 18CrNiMo7-6 steel before and after SP with annealing temperatures