Vacuum 133 (2016) 54–57

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Short communication

A novel duplex plasma treatment combining plasma nitrocarburizing and plasma nitriding

Bin Miao [a, c], Yating Chai [a, b], Kunxia Wei [a, c, **], Jing Hu [a, b, *]

[a] Jiangsu Key Laboratory of Materials Surface Science and Technology, Changzhou University, Changzhou 213164, People's Republic of China
[b] Materials Research and Education Center, Auburn University, AL 36849, USA
[c] Jiangsu Collaborative Innovation Center of Photovoltaic Science and Engineering, Changzhou University, Changzhou 213164, People's Republic of China

ARTICLE INFO

Article history:
Received 1 June 2016
Received in revised form
6 July 2016
Accepted 6 July 2016
Available online 5 August 2016

Keywords:
Surfaces
Plasma nitriding
Plasma nitrocarburizing
Microstructure
Diffusion

ABSTRACT

In this study, a novel duplex treatment (DT) combining plasma nitrocarburizing (PNC) and plasma nitriding (PN) was primarily developed for AISI 1045 steel. The modified samples were investigated by optical microscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), microhardness test, and pin-on-disk tribotest. The results showed that the nitriding efficiency was remarkably improved by DT, and the compound layer was much thicker than that treated by PNC or PN alone. Furthermore, higher cross-sectional microhardness and significant enhancement of wear resistance were achieved. The possible enhancement mechanism could be the phase transformation of γ' to ε, and the reduced amount of cementite could be because of the activating and ionizing effect of the subsequent PN on cementite.

1. Introduction

AISI 1045 steel is a widely used type of medium carbon steel because of its low cost and excellent combined properties [1–5]. In real-time applications, however, surface modification is necessary to further enhance its properties to meet the requirements in various service environments [6–10]. Plasma nitriding (PN), plasma carburizing (PC), and plasma nitrocarburizing (PNC) are proved to be effective surface modifications and have been adopted by most industries.

In the past decades, PN and PNC have become popular surface modification techniques, as they are more environment friendly than those using gas sources and solid powders [11,12]. It is well known that PN offers many advantages over traditional gas nitriding, especially in terms of gas consumption and properties control. However, it requires long cycle time [3,13]. Generally, the technique consumes several hours to yield the desired thickness and properties in most applications, which results in low efficiency and high production cost [14,15]. The developed PNC is a thermochemical treatment, in which nitrogen and carbon atoms diffuse simultaneously into the workpiece surface layer to obtain a higher efficiency [16–19]. However, it has been found that the formation of a loose layer is unavoidable in the utmost surface because of its long duration, which causes side effects on surface performance such as higher brittleness [19]. Therefore, developing a new way to overcome these problems is of paramount importance.

To make good use of the advantages of both PNC and PN, a novel duplex treatment (DT) combining PNC and PN was primarily developed for AISI 1045 steel in this study, and it was found that the developed technique could not only increase nitriding efficiency but also improve related properties.

2. Experimental procedures

The material used in this study was AISI 1045 steel with the following chemical compositions (wt%): C, 0.45; Si, 0.18; Mn, 0.52; S, 0.031; P, 0.032; and Fe: remaining. The specimens were prepared with dimensions of 10 × 10 × 5 mm, and quenched at 1123 K for 8 min, cooled in water and tempered at 833 K for 30 min, and air cooled. All the surfaces of specimens were grounded by silicon carbide paper, polished by chromic oxide slurry, and cleaned in

* Corresponding author. Jiangsu Key Laboratory of Materials Surface Science and Technology, Changzhou University, Changzhou 213164, People's Republic of China.
** Corresponding author. Jiangsu Collaborative Innovation Center of Photovoltaic Science and Engineering, Changzhou University, Changzhou 213164, People's Republic of China.
E-mail addresses: weikunxia@cczu.edu.cn (K. Wei), jinghoo@126.com (J. Hu).

http://dx.doi.org/10.1016/j.vacuum.2016.07.007
0042-207X/

dehydrated alcohol for 15 min in an ultrasound device before DT treatment.

All the plasma treatments were performed in the same 20-kW pulsed DC PN equipment. After placing the specimens in the equipment, it was evacuated to 10 Pa and then sputtered for 30 min by hydrogen at a flow rate and gas pressure of 500 mL/min and 300 Pa, respectively. After sputtering, PNC, PN, or DT process was subsequently run at 783 K for 4 h at a gas pressure of 400 Pa, with the gas flow rate as mentioned below: (1) PN: hydrogen flow rate of 600 mL/min and nitrogen flow rate of 200 mL/min in the whole process; (2) PNC: nitrogen flow rate of 591 mL/min, propane flow rate of 9 mL/min, and without hydrogen in the whole process; (3) DT: PNC 3 h for the first step and PN 1 h for the second step, with the same gas flow rate as in (1) or (2) for each step. The reasons for choosing the above gas flow for each process are as follows: (1) As reported previously, the suitable ratio of nitrogen and hydrogen in PN is 1:3 [20]; according to this ratio, hydrogen flow of 600 mL/min and nitrogen flow of 200 mL/min were used by our group [21]; (2) In our previous research, we found that the optimum concentration of propane is 1.5% in the PNC process [19]; thus, we used the optimum ratio in the PNC step. After each process, the nitriding furnace was pumped to 10 Pa and cooled to ambient temperature.

The surfaces of the specimens were metallographically polished and etched using ethanol solution containing 4% nitric acid. An optical microscope (DMI-3000M) was used for observing microstructures. The phases were determined by X-ray diffraction (XRD) with Cu-Ka (λ = 1.54 Å) radiation at a scan rate of 0.2°/min, 2θ ranging from 30° to 80°. The surface morphology was observed by scanning electron microscopy (SEM). In addition, the cross-sectional hardness was measured by a HXD-1000TMC microhardness tester with the test load of 50 g and the holding duration of 15 s. At least three measurements were carried out for each sample for assessing the hardness. Finally, a HT-1000 ball-on-disk high-temperature friction and wear tester was used to evaluate wear resistance of AISI 1045 steel at dry sliding with 50 N load and 200 rpm against GCr15 ball with a diameter of 4 mm for durations of 30 min. The wear tests were performed at room temperature (~20 °C) with a relative humidity of approximately 50%.

3. Results and discussions

3.1. Microstructure and depth analysis

The cross-sectional microstructures of samples with plasma treatment at 783 K for 4 h are shown in Fig. 1a–c. The figure clearly shows the formation of the modified layer; the compound layer obtained by PN is the thinnest (Fig. 1a) and that obtained by DT is the thickest (Fig. 1c). Especially, the outer layer (marked in red zone) of the images show that a porous layer is formed in Fig. 1b after PNC, while the layer disappears and a dense surface is formed after DT.

3.2. XRD analysis

Fig. 2 shows the XRD patterns of the samples with plasma treatment at 783 K for 4 h. The figure shows that both ε-$Fe_{2\text{~}3}$(C,N) and γ′-Fe_4N are formed in the samples treated by PN, PNC, and DT. It can also be observed that the relative amount of cementite is decreased for DT sample because of the activating and ionizing effect of the subsequent PN step on cementite, and the relative amount of ε-$Fe_{2\text{-}3}N$ is increased because of the transformation of some γ′-Fe_4N to ε-$Fe_{2\text{-}3}N$ with higher nitrogen content.

3.3. Surface morphology

Fig. 3 shows SEM micrographs of the surface morphologies of the samples with plasma treatment at 783 K for 4 h. Block structure with some carbon black can be seen in Fig. 3a for PNC-treated sample, while fine particles are uniformly distributed and without carbon black in Fig. 3b for DT-treated sample, which is attributed to the spallation of cementite by plasma bombardment during the PN step [22].

3.4. Microhardness analysis

Fig. 4 shows the microhardness profile of samples with plasma treatment at 783 K for 4 h. The figure shows that the hardness in the modified layer decreases gradually from the surface to core. Further, the hardness of samples treated by DT reaches a high level of 788 $HV_{0.05}$, much higher than that by PN and PNC. In addition, it is obvious that the modified layer by DT presents a wide hardened region as compared to that by PN and PNC, with the effective hardening depth of the order of 26, 45, and 70 μm, respectively. The

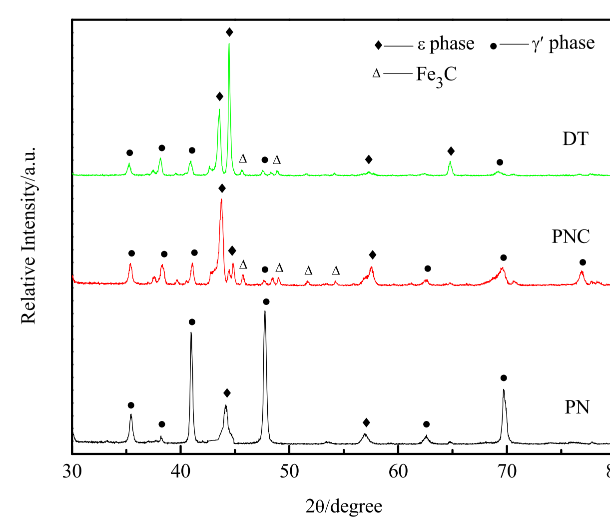

Fig. 2. XRD patterns of samples treated by different plasma techniques (783 K for 4 h).

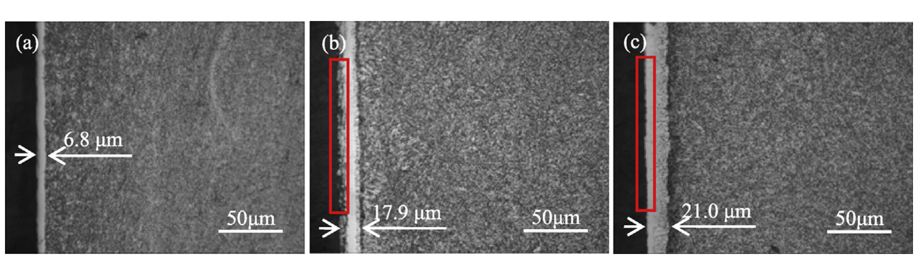

Fig. 1. Cross-sectional microstructure of samples treated by (a) PN, (b) PNC, and (c) DT at 783 K for 4 h.