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Influence of pulsed power supply parameters on active screen plasma nitriding

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ABSTRACT

It is well known that active screen plasma nitriding (ASPN) is driven using a pulsed D.C. power supply, due to several advantages such as improved control over temperature, nitrided layer phase composition and active species generation. In addition, the pulsed power supply helps to avoid the transition to the arc regime. The ASPN configuration in particular is preferred over conventional plasma nitriding due to complete elimination of edge effect, as plasma is not in direct contact with the samples. In this work, the influence of pulsed power supply parameters such as duty cycle, peak voltage, peak current and time averaged current is studied in an ASPN setup, on nitrided layer phase composition and surface hardness of austenitic stainless steel (ASS). In addition, optical emission spectroscopy (OES) is used to measure the excitation, vibrational and rotational temperatures (T_{exc}, T_{vib}, T_{rot}), nitrogen dissociation fraction and concentration of active species $N_2(C^3\Pi_u)$ and $N_2^+(B^2\Sigma_u^+)$ (in terms of emission intensities) to clarify the results obtained from surface characterization. The results show that keeping time-averaged current constant, the surface hardness decreases with the duty cycle of the pulsed D.C. power supply. The results from OES and surface analysis are correlated, and as a result plasma parameters (T_{exc}, T_{vib}, T_{rot}) and active species concentration (N, N_2, N_2^+) are found to be important precursors in surface hardening of ASS.

1. Introduction

Nitriding is a thermochemical surface treatment technique which improves the mechanical properties of various materials. Aided with the abnormal glow discharge, conventional plasma nitriding (CPN) remained a standard industrial technique for many decades [1,2]. The CPN is quite favorable over other previous techniques such as gas and salt bath nitriding due to certain features like excess of nitrogen concentration, no environmental hazards, short processing time and low energy consumption [3–5]. Despite these features, plasma is in direct contact with the specimens which results in certain shortcoming such as surface damages due to arcing, edge effect and certain limitations to treat specimens of various dimensions in one batch [4,6,7]. To overcome these drawbacks associated with the CPN, active screen plasma nitriding (ASPN) was introduced [8–16].

The ASPN is based on transfer of glow discharge from specimens to a cathodic cage, the so called active screen (AS), and this screen covers the specimens along with the worktable. The AS plays dual role i.e. it distributes the temperature uniformly over the work load/specimen by radiations, and it generates active species for nitriding process [5,11,13,17]. Since the specimen and glow discharge are isolated, therefore the problems associated with the CPN can be avoided. The fundamental mechanism of ASPN has been explained by a number of models: Georges [18] proposed that neutral nitrogen species produced on AS play the significant role, while according to Hubbard et al. [12,19,20] nitrogen ions and neutrals originating from the AS and sufficient substrate biasing results in uniform nitrided layer formation. The previously proposed model by Li and Bell [21,13] which involves sputtering and re-deposition mechanism was found insufficient by Hubbard. According to Zhao et al. [13] and Gallo and Dong [23], the sputtered iron nitride particles from the AS are the nitrogen carriers which plays significant role in hardening effect. The ASPN process mainly occurs in the volume surrounded by AS which has low electric field, and this is the region of main interest [24]. The AS confines the electrons electrostatically and therefore its

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crucial to study and control the number density and hence the active species in the process volume [24]. The number density and active specie production is based on the mixture of gases, discharge conditions and plasma source.

The plasma can be generated by numerous sources for laboratory and industrial purposes such as direct current, microwave, radiofrequency etc. Among these plasma sources, pulsed D.C. discharges are quite favorable due to various advantages such as additional control on temperature, nitrided layer phases, and active species generation as a function of duty cycle [25–27]. The commercial nitriding equipment is usually powered by pulsed D.C. power sources, to avoid the overheating and arcing [28,29]. In addition to ASPN, pulsed D.C. power sources are broadly used in CPN [1,30–36], plasma enhanced chemical vapor deposition (PECVD) [37–39], pulsed magnetron sputtering [40], hollow cathode discharge (HCD) [41,42] and many other industrial applications. It is found that while the plasma reactive species remain in the vacuum chamber during whole cycle, the ion induced sputtering is dominant only during the voltage pulse [43]. Therefore, the pulse duration (i.e. duty cycle) is of significant interest in pulsed power sources applications and it needs to be optimized for improved results.

Menthe and Rie [44] examined the influence of pulsed duty cycle on the nitrided layer formation on AISI 304. It was found that phases of nitrided layer can be altered by the pulse off-time in addition to other parameters. Alves et al. [28] investigated the influence of pulse width on the microstructure of nitrided layer.They processed AISI-409 at 50–100% duty cycles, 500 °C temperature, and 600 Pa pressure. It was found that the penetration depth of diffusion zone is mainly affected in range of 80–100% duty cycle, which was attributed to enhanced ionized species during afterglow. In these conditions, the energetic neutrals were found to be crucial precursors for plasma nitriding. Díaz-Guillén et al. [45] evaluated the influence of pulsed duty cycle from 10 to 80% on surface characteristics of AISI-4340. They found that the phase composition of nitrided layer is independent of duty cycle; however layer thickness and surface hardness increase with duty cycle up to 50%, and decrease beyond 70%. The role of pulsed duty cycle from 10 to 90% with unipolar power supply is investigated by Jeong et al. [46] for duplex coating, where the surface roughness and hardness increase with the duty cycle. Taherkhani and Mahboubi [47] studied the influence pulsed duty cycle (40–80%) and frequency (8–10 kHz) on the nitriding of AISI H13 steel. They observed that the compound layer thickness, micro hardness and surface roughness increase with the duty cycle. Recently, the influence of duty cycle on surface characteristics of plasma processed AISI 316L is examined by Díaz-Guillén et al. [48]. They reported that the hardness and nitrided layer thickness increase with duty cycle up to voltage pulse time of 5 μs and then decrease with the duty cycle. It is important to report that all the above works were made in CPN and the effect of duty cycle have no definite trends on nitriding quality and depends on processing conditions. Although ASPN results have been reported with pulsed power supply, the influence of duty cycle has not been reported so far to our knowledge.

The influence of discharge parameters can be observed non-invasively using optical emission spectroscopy (OES) [49–51]. The effectiveness of surface processing occurring in the plasma depends on the plasma parameters such as electron temperature, electron number density and vibrational temperature [52,53]. To attain improved results in plasma nitriding, it is necessary to optimize plasma parameters, nitrogen atomic and molecular species which are responsible for the enhancement of plasma-ferro-metal reaction [54]. Hugon et al. [55] deduced the favorable conditions of pulsed D.C. nitriding system in term of duty cycle using OES. He observed that the population of N_2^+ state increases with the duty cycle. Kim et al. [56] investigated the influence of duty cycle on the nitriding of AISI H13 and active species concentration (N_2^+,N_2^*) in term of emisison intensitites of nitrogen bands. The emission intensities are found to increase with the duty cycle as well as surface hardness and γ'-Fe_4N phase formation. In another study, Kim and Han [57] investigated the influence of duty cycle (25–75%), and maximum enhancement in N_2^+ species was found at 50% duty cycle.

The above reports suggest that the duty cycle has a significant influence on the generation of nitrogen active species, sputtering process, surface characteristics and the influence of duty cycle in ASPN should be examined. In current study, the effect of duty cycle as well as other parameters such as peak voltage, peak current and time averaged current are examined in an ASPN system on the plasma parameters and surface properties of AISI 304 steel.

2. Experimental details

2.1. Experimental apparatus

The schematic diagram of experimental setup used in the current study is shown in Fig. 1, which consists of stainless steel vacuum chamber having height of ~33.5 cm and a diameter of ~31.5 cm. The grounded chamber walls acts as anode, and a ~17 cm diameter ~18 cm high screen cage made with 2 mm thick AISI-304 plate, acting as the counter electrode. The screen cage is punched with uniformly distributed round holes of ~8 mm diameter on the walls and ~6 mm on the top surface; as reported in the literature [41,58]. The screen cage is connected to pulsed power supply, whereas samples placed on a ceramic plate which attains floating potential. The specimens were 55 mm away from the top of the cage.

The nitriding process was made as follows. First the chamber was evacuated down to the base pressure ~ 0.1 Pa with the help of a rotary vane pump. The gas flow was controlled using flow meters. The temperature was monitored and controlled with an external heater and thermocouple, respectively.

2.2. Materials

In the current study, AISI 304 specimens (chemical composition given in Table 1) having dimension of 10 mm × 10 mm and thickness of ~2 mm are used. The samples are mirror polished by silicon carbide papers of different grit size (320, 400, 600, 1000, 1200, 1500 and 2000) and finished using micron alumina powder. Later, they are cleaned in ultrasonic acetone bath for 15 min.

2.3. Processing

The specimens are processed at optimum composition of gases $N_2/H_2 = 60/40$, as reported earlier [59]. The complete processing conditions are given in Table 2. The specimens are placed in the chamber and evacuated down to the base pressure of 0.1 Pa. After achieving the processing temperature at the electrode, the argon gas with the flow rate of 50 sccm is used for the sputtering of specimen for 30 min. Nitrogen and hydrogen gases are then injected for processing. After processing, the specimens are cooled down to the room temperature under vacuum before being taken out from the chamber.

2.4. Characterization

The surface hardness of the specimens is obtained using Vickers micro hardness tester equipped with 136° diamond indenter. The phase structure of specimens is examined using X-ray diffractometer with CuKα radiation. To examine the surface morphology and cross sectional images, scanning electron microscope (SEM) is used. For spectroscopic diagnostics, a customized computer controlled Avantes USB spectrometer having five channels with spectral resolution 0.08–0.12 nm is utilized. The measured emission intensities are normalized for quartz window (as provided by manufacturers) while the emission intensity of spectrometer along with the optical fiber is calibrated with the tungsten ribbon lamp.