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Analyses of quenching process during turn-off of plasma electrolytic carburizing on carbon steel

Jie Wu^{a,b}, Run Liu^{a,b}, Wenbin Xue^{a,b,*}, Bin Wang^{a,b}, Xiaoyue Jin^{a,b}, Jiancheng Du^{a,b}

^a Key Laboratory for Beam Technology and Materials Modification of Ministry of Education, College of Nuclear Science and Technology, Beijing Normal University, Beijing 100875, China

^b Beijing Radiation Center, Beijing 100875, China

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1. Introduction

In recent years, plasma electrolytic saturation (PES) technology has been studied as an efficient approach of surface hardening on metals [1–3]. During the saturation process, a continuous vapor envelope is broken down at a critical voltage, resulting in the plasma discharge at near-cathode region and an enhanced interstitial diffusion of active species. Specific processing methods have extended from plasma electrolytic carburizing (PEC) [4] to plasma electrolytic carbonitriding (PEC/N) [5,6] and plasma electrolytic borocarburizing (PEB/C) [7,8]. It is generally believed that the hardening effect derives from two aspects. The first is the saturation of carbon, nitrogen or boron atoms to form solid solution or compound layer with high hardness, and the other is the rapid quenching into the electrolyte to form some hard phases at the end of PES treatment. However, the latter has not been confirmed by experimental evidence, so it is interesting to evaluate the so-called quench hardening effect in the turn-off process.

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ABSTRACT

Plasma electrolytic carburizing (PEC) under different turn-off modes was employed to fabricate a hardening layer on carbon steel in glycerol solution without stirring at 380 V for 3 min. The quenching process in fast turn-off mode or slow turn-off mode of power supply was discussed. The temperature in the interior of steel and electron temperature in plasma discharge envelope during the quenching process were evaluated. It was found that the cooling rates of PEC samples in both turn-off modes were below $20 \,^{\circ}C/s$, because the vapor film boiling around the steel sample reduced the cooling rate greatly in terms of Leidenfrost effect. Thus the quench hardening hardly took place, though the slow turn-off mode slightly decreased the surface roughness of PEC steel. At the end of PEC treatment, the fast turn-off mode used widely at present cannot enhance the surface hardness by quench hardening, and the slow turn-off mode was recommended in order to protect the electronic devices against a large current surge.

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The fast turn-off mode of power supply at the end of PES discharge process was widely used since it was considered to be a trigger of quench hardening effect which can effectively enhance the surface hardness [9]. Here, the fast turn-off mode refers to turning off the power supply immediately once the PES process is completed. It means that the applied current and voltage of sample instantaneously shrink to zero, and the plasma discharge around the steel sample immediately stops. But till now, the detailed descriptions of this quenching process and the influence of different turn-off modes on microstructure of steel sample have not been reported. Nie et al. [10] found that an expanded austenite diffusion layer below a top hardening layer on the stainless steel surface was observed after PEC/N treatment. They ascribed this diffusion layer to quench hardening effect, which occurred when the steel surface with high temperature of several hundred degrees Celsius was quenched into the electrolyte after switching off the power supply. However, they have not given more evidence such as cooling rate measurement to support the assumption about quench hardening effect.

The sample temperature is key characteristic parameter to evaluate quenching process. Meanwhile, for the slow turn-off mode in which the voltage decreases gradually, the plasma electron temperature is also important. It can be used to evaluate the characteristics of plasma discharge, which is the source of heat transfer. In this work, we prepared a carburizing layer on T8 high-carbon steel by PEC method. In order to investigate the quenching process, a







^{*} Corresponding author at: Key Laboratory for Beam Technology and Materials Modification of Ministry of Education, College of Nuclear Science and Technology, Beijing Normal University, Beijing 100875, China. Tel.: +86 10 62207222; fax: +86 10 62231765.

E-mail addresses: xuewb@bnu.edu.cn, xuewb2013@163.com (W. Xue).

thermocouple was employed to measure the temperature change inside the steel sample while optical emission spectroscopy (OES) was utilized to determine the electron temperature in plasma discharge envelope. The microstructures, phase constituents and hardness profiles of the PEC treated samples were analyzed. The influence of different turn-off modes on the quenching process was discussed.

2. Materials and methods

Plasma electrolytic carburizing was performed on T8 highcarbon steel (C: 0.75–0.84, Si: \leq 0.35, Mn: \leq 0.40, P: \leq 0.035, S: \leq 0.030, wt.%, Fe balance) cylinder with dimensions of Φ 10 mm \times 20 mm. The cylinder sample was set as cathode and it was mechanically polished, cleaned in ethanol and dried before the experiment. A stainless steel container served as anode and an aqueous solution containing 80 vol.% glycerol was chosen as electrolyte. During the PEC treatment, the negative bias voltage on T8 steel cathode was fixed at 380 V for a stable discharge and the discharge duration is 3 min. Then the power supply was slowly shut down with the applied voltage reducing to zero within 40 seconds, which is the so-called slow turn-off mode. Also, the fast turn-off mode in which the voltage immediately decreased to zero was compared.

In the stable discharge stage and slow turn-off process, the active species in the plasma envelope were detected using optical emission spectroscopy (OES, AvaSpec-3648). Meantime, a thermocouple was employed to detect the temperature distribution inside the T8 steel close to the sample surface. Details of the measurement method are described in Ref. [11]. In addition, Fig. 1 shows the schematic diagram of PEC treatment. The stainless steel double groove was used as electrolyte bath, and the electrolyte was cooled by running water. The stirring system in electrolyte bath was absent, because the stirring of solution will disturb the plasma envelope around the steel sample and affect the carburizing stability, though it may improve the cooling rate at end of PEC treatment [12]. Futhermore, the morphology and structure of the PEC treated steels were characterized by scanning electron microscopy (SEM, Hitachi S-4800) and X-ray diffraction (XRD, X' Pert Pro MPD). The compositions were analyzed by energy dispersive spectroscopy (EDS). Microhardness measurements of PEC samples were performed with a HX-1000 Vickers hardness tester under 10 g load.



Fig. 1. Schematic diagram of the apparatus used for PEC process on T8 steel.

3. Results and discussion

3.1. Current, voltage and sample temperature

Fig. 2a shows the typical dependence of current and voltage on discharge time in the slow turn-off mode. The current and voltage changes in the whole PEC treatment are also given at the top right, and the interval between two dash lines is amplified as the turn-off process. In the stable discharge stage, the current is kept steady, and then it starts to reduce when the voltage decreases uniformly from 380 V. After 25 s, the current drops quickly because the voltage is too low to sustain the discharge process and the discharge stops. Then it attenuates to zero at 40 s and the slow turn-off process finishes.

Fig. 2b displays the temperature changes in the interior of steel sample close to surface in both turn-off modes. The temperature was measured by inserting a thermocouple into a small hole in the cylinder sample. The distance of the hole bottom to the lower surface of cylinder was 0.2 mm. In the fast turn-off mode, the sample temperature decreases from 700 °C to 80 °C within 55 s though the voltage and current immediately reduce to zero. However, in the slow turn-off mode, the sample surface still keeps high temperature close to 700 °C in the early 25 s. Then it begins to decline rapidly to about 150 °C within 30 s. Furthermore, as shown in Fig. 2b, the cooling rates in rapid decline stage of the two turn-off modes at 15–40 s and 25–50 s respectively are approximately the same with 18.2 °C/s. It implies that these two turn-off modes substantially experience a similar quenching process. This cooling rate is much lower than that of the traditional quenching process in aqueous



Fig. 2. (a) Dependence of current and voltage on discharge time in the slow turn-off mode. (b) Dependence of sample temperature with cooling time in the two turn-off modes.

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