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Kinetics and mechanical study of plasma electrolytic carburizing for pure iron

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ABSTRACT

In this work, plasma electrolytic surface carburizing of pure iron in aqueous solution consisting of water, glycerin and NH_4Cl was investigated. Surface carburizing was carried out in 20% glycerin solution treated at 750 °C, 800 °C, 900 °C and 950 °C temperatures for 5, 10 and 30 min. The formation of hard carbon-rich layer on the surface of pure iron was confirmed by XRD analysis. Metallographic and SEM studies revealed a rough and dense carburized layer on the surface of the pure iron. Experimental results showed that the thickness of the carburized layers changes with the time and temperature. The average thickness of the carburized layer ranged from 20 to 160 μm. The hardness of the carburized samples decreased with the distance from the surface to the interior of the test material. The average hardness values of the carburized layers on the substrate ranged 550–850 HV, while the hardness of the substrate ranged from 110 HV to 170 HV. The dominant phases formed on the pure iron were found to be a mixture of cementite (Fe_3C), martensite (Fe + C) and austenite (FCC iron) confirmed by XRD. Wear resistance in all plasma electrolytic carburized samples is considerably improved in relation to the untreated specimen. After carburizing, surface roughness of the samples was increased. Friction coefficients were also increased because of high surface roughness.

1. Introduction

Surface hardening techniques have been widely used for ferrous metals to improve hardness, wear resistance, corrosion resistance, fatique and oxidation endurance. In order to achieve these properties, surface hardening is performed by either surface transformation (flame, induction, laser and electron beam hardening methods) or surface modification (carburizing, nitriding, boriding, carbonitriding). Surface transformation methods change the microstructure of the surface, whereas surface modification methods change not only the microstructure of the surface but also the composition of the surface. In other words, chemical or thermochemical reactions is produced at the surface of the material [1]. Alternatively to the conventional methods discussed above, novel methods such as ion implantation, chemical or physical vapor deposition, sputtering and plasma have been applied for many years. These new methods have several advantages as reliability, reduced treatment times, improved wear resistance, superior control of microstructure and environmental cleanliness [2].

One way of the surface hardening can be accomplished by increasing the surface concentration of carbon in a process termed carburizing. Conventionally, a low carbon steel is exposed to an elevated temperature above 910 °C in an atmosphere rich in carbon to form austenite. Once the carbon atom dissolves at the surface, it tends to diffuse into the core to produce a carbon enriched layer. Following carburizing, it is quenched to a relatively low temperature to increase the hardness of the carbon enriched surface layer [3–6]. Solid, liquid, and gaseous carburizing media can be used depending on the nature and scope of the work involved. Owing to the fact that carburizing is diffusion controlled process, the rate of layer depth is dependent on both temperature and time [7].

Luk et al. used a method, called electrolytic surface hardening process, as another way of surface hardening of medium or high carbon steel. The method is a special hardening process employing electrolysis in an aqueous solution under particular conditions. In this process, the work piece is connected to DC or pulse DC power supply as a cathode in an electrolysis cell. Once a stable hydrogen film is formed on the work piece by applied high voltage, then electrical discharge, including electrons and ion avalanche, occurs across the hydrogen film. Then, the work piece is heated violently to above the austenite temperature due to the generation of resistance heat under the applied voltage in a short time. After that, the hydrogen film around the work piece will break by shutting off the power of the electrolysis cell. Finally, especially the surface of the work piece will self-quench and/or be quenched by the cold electrolyte surrounding the work piece, leading to hardening of the work piece [8].

The aqueous electrolyte surface hardening process has been used in the literature for surface hardening of medium or high carbon steels (AISI 1050) [8–11]. Plasma electrolysis for surface

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hardening by means of carburization, nitriding, or nitride/carbide formation on mild steel (0.4% C + 1% Cr) or low carbon steel (SAE 1020) and oxidation of aluminum alloys were studied extensively, and the review paper was published in the literature [12]. Moreover, plasma electrolytic nitrocarburizing treatment for stainless steels was studied as well [13]. Plasma electrolytic nitriding for cast iron G3500 and cast steel S0050A was also studied [14]. Furthermore, plasma electrolytic surface carburizing of pure iron was studied [15]. Besides, carbonitriding of Q235 steel in an ethanolamine electrolyte was studied [16].

This study was driven by the lack of studies on the plasma electrolytic surface carburizing of pure iron. In the previous study, we investigated surface carburizing of pure iron performed at only 850 °C for different time periods in an aqueous solution of 10% glycerin [15]. However, the kinetics study of this process and wear rate of the coating are stil missing. Therefore, at the present study, the plasma electrolytic surface carburizing of pure iron was carried out in aqueous solution of 20% glycerin treated at 750 °C, 800 °C, 900 °C and 950 °C temperatures for 5, 10 and 30 min to examine the effect of temperature, time and the amount of glycerin on the kinetics of this process and to study the wear rate of the coatings.

2. Experimental procedure

Substrate material used in this study was pure iron (99.97% weight) that had a shape of approximately 10 mm × 12 mm × 2 mm. All the samples were ground by 500 grit SiC paper and cleaned by ethyl alcohol before the plasma electrolytic surface carburizing treatment.

Carburizing was performed in an aqueous medium with a nominal chemical composition of 10 l of water, 1 kg of NH_4Cl, and 2 l of glycerin ($C_3H_8O_3$). The solution was prepared by solving first NH_4Cl in water and then glycerin was added to the solution. In this aqueous solution, electrolytic conductivity was attained by addition of NH_4Cl, and glycerin acted as a carbon source. Treatments were performed in a DC power supply unit at different applied voltages of 180 V, 200 V, 220 V, and 240 V equivalent to the temperatures of 750 °C, 800 °C, 900 °C and 950 °C for 5, 10, and 30 min. Temperature measurement of the samples was carried out using a thermocouple inserted into the sample approximately 1 mm to the surface by drilling a hole. At mentioned temperature or voltage above, the sample was immersed slowly into the electrolyte by a step motor drive. Then, a gas film rapidly formed in the vicinity of the sample during the slow dipping process. Due to the resistance of the gas film, the samples were heated up to temperatures of 750 °C, 800 °C, 900 °C and 950 °C in a short time. After the samples were carburized in the solution for 5, 10, and 30 min, the samples were quenched in the solution to room temperature by shutting down the power supplied. Following quenching, the samples were removed from the clamping device and cleaned by ethyl alcohol.

The cross-sectional microhardness profiles of the plasma electrolytic carburized samples from the surface to the interior were measured by a ZEIS Axio Imager microhardness tester equipped with a Vickers diamond indenter with a load of 100 g. Before the mesurements, samples were ground on wet emery paper down to 1200 grit, then polished by 1–3 μm diamond paste and etched with 3% Nital.

Rigaku X-ray diffractometer (DMAX 2200) with a Cu Kα radiation source of a wavelength of 1.540 over a 2θ range from 40° to 90° was employed for the phase characterization of carburized layer of the samples. Philips field emission scanning electron microscope (SEM) was used to examine the microstructure of the cross-section and the surface of the carburized layer of the samples. In addition, the thickness of the carburized layers was measured under an optical microscope.

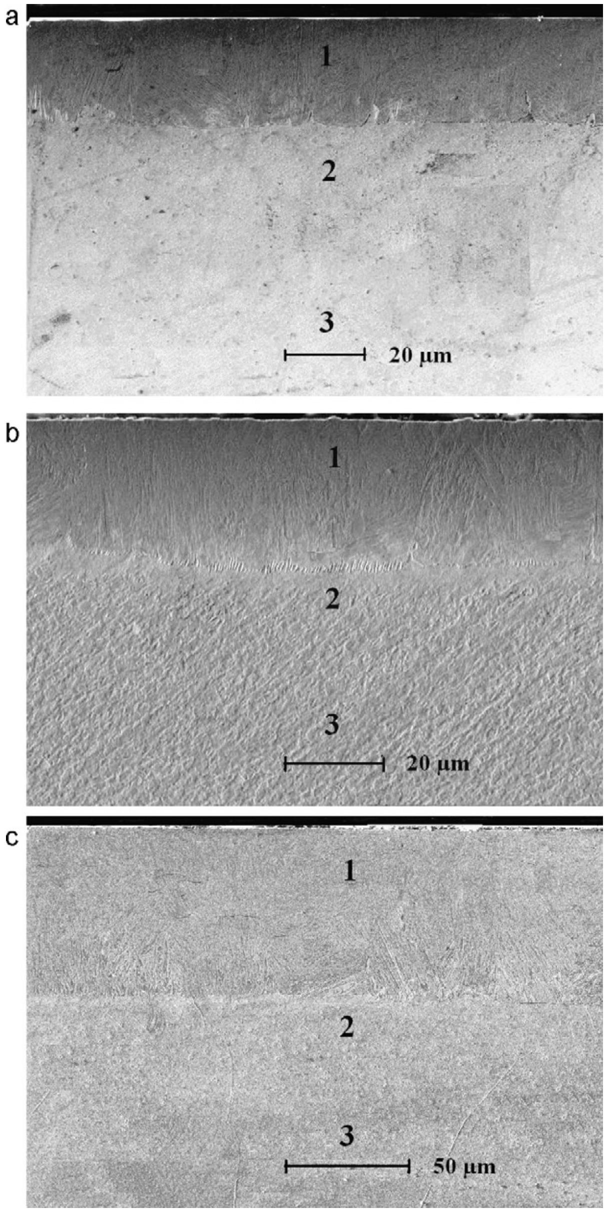

Fig. 1. SEM images of carburized pure iron samples showing the carbon rich layer (1), the transition layer (2), and the base metal (3); treated at (a) 800 °C for 5 min, (b) 800 °C for 10 min, (c) 800 °C for 30 min.

Surface roughness was measured with Dektak Programmable Surface Profilometer by scanning 1000 μm in length from the center of the samples. Wear tests on untreated and treated samples were performed against Al_2O_3 balls under the testing conditions of 10 N load, 100 m distance and velocity of 0.2 m/s without lubricant. The wear rates and the average friction coefficients were obtained by CSM tribometer.

3. Experimental results

3.1. Microstructure

Optical microscope and SEM examinations of carburized pure iron revealed dense and needle-like coating. SEM examinations of the carburized layers formed at 800 °C for 5, 10 and 30 min are shown in Fig. 1(a–c), respectively. Three regions appear in Fig. 1(a–c), namely (1) a layer consisting of a dense needle-like structure, (2) a diffusion/transition zone between the layer and the