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Influence of bias voltage on the formation and properties of iron-based nitrides produced by plasma nitriding

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ABSTRACT

A series of experiments have been conducted on AISI 5140 low alloy steel using a hollow cathode discharge-assisted plasma nitriding apparatus with the aim of elucidating the role of substrate bias voltages in plasma nitriding process. For comparison, some samples were nitrided with applied substrate bias (−50 V) while other samples were nitrided at floating potential. Treatments were carried out in NH_3 atmosphere of 150 Pa at temperatures ranging from 450 °C to 550 °C for 2 h, 4 h, and 6 h. The nitrided samples were characterized by optical microscopy, X-ray diffraction and micro-hardness measurement. The corrosion behaviors were evaluated using anodic polarization tests in 3.5% NaCl solution. The results showed that the microstructure and phase constituents of the nitride layers were strongly influenced by the bias voltages. It was also demonstrated that the better corrosion resistance with a thicker nitrided layer was obtained on the biased sample.

1. Introduction

Iron nitrides play an important role in metallurgy. In particular, nitriding of iron and steel can improve the tribological properties, the corrosion resistance and the fatigue endurance [1–6]. By the nitriding process, the α-Fe solid solution becomes enriched with nitrogen and, if the chemical potential of nitrogen in the nitriding atmosphere is sufficiently high, different iron nitrides may form. It is well known that plasma nitriding offers many advantages over traditional gas nitriding and bath nitriding, particularly, in terms of reduced gas consumption, reduced energy consumption, and the complete removal of any environmental pollution. However, in conventional plasma nitriding process, the treated components are submitted to high cathodic potentials and the plasma acts directly in surface of the components. This brings some inherent shortcomings for plasma nitriding, such as damage caused to parts by arcing, the “edging effect”, and difficulty in maintaining a uniform component temperature, particularly in full workloads of components with varied dimensions. Considerable effort has been made over past ten years to overcome these problems. Post-discharge nitriding [7], RF nitriding [8], plasma immersion implantation [9,10] and through cage (TC) plasma nitriding [11–20] have been developed. A basic consideration of these modified nitriding processes is to decouple the plasma generation from the components to be nitrided. For the TC nitriding technique, the components are enclosed by a metal screen and maintained at a floating potential. As the cathodic potential is applied to the metal screen, and not to the components to be treated, the above-mentioned problems are completely avoided [11–21]. Radiation from the heated screen supplies the heat that brings components to the required temperature for treatment. Hubbard et al. [21] have shown that the bias voltages are essential for a satisfactory nitriding response in the commercially available TC system.

In this research, the samples to be treated were placed in a floating potential or subject to a relative lower negative bias voltage of 50 V. The aim of this paper was to investigate the structure and properties of the AISI 5140 steel which were plasma nitrided assisted by hollow cathodic discharge, and to compare the effect of bias in nitriding process.

2. Experimental

The material used in this study was AISI 5140 steel with the following chemical compositions (in wt.%): 0.37–0.45 C, 0.80–1.10 Cr, 0.50–0.80 Mn, 0.20–0.45 Si and Fe balance. The specimens were cut from a hot rolling bar and then machined to 20 mm diameter and 5 mm thickness. The samples were mechanically gritted with silicon carbide paper and polished to a mirror surface.

The nitriding was carried out using a 20 kW ion nitriding furnace. The schematic arrangement of specimens and apparatus is shown in Fig. 1. In our experiments, the plasma was formed by a pulsed dc glow discharge. The active screen consists of two low carbon steel cylinders with different diameter. The diameter of inner cylinder is 440 mm and that of the outer cylinder is 468 mm. The thickness of the cylinder is 6 mm. The holes with diameter of 10 mm are well distributed in the inner cylinder, these holes supply good entry way for the transfer of chemical elements. The space between two cylinders is 8 mm. The discharge current and voltage between the two-cylinder active screen and the anode (furnace well) was 4–6 A and 500–670 V. The nitriding gas was NH_3, and the working pressure was maintained at 150 Pa.

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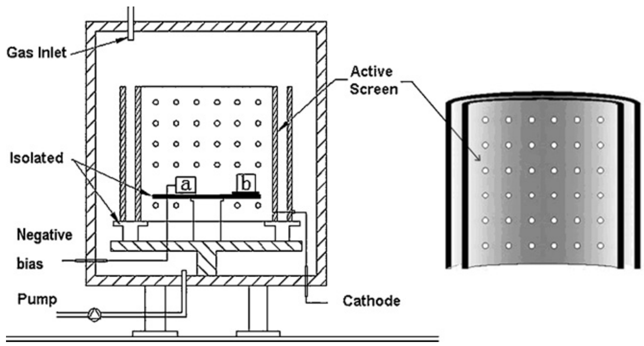

Fig. 1. Schematic diagram of the hollow cathode discharge-assisted plasma nitriding apparatus.

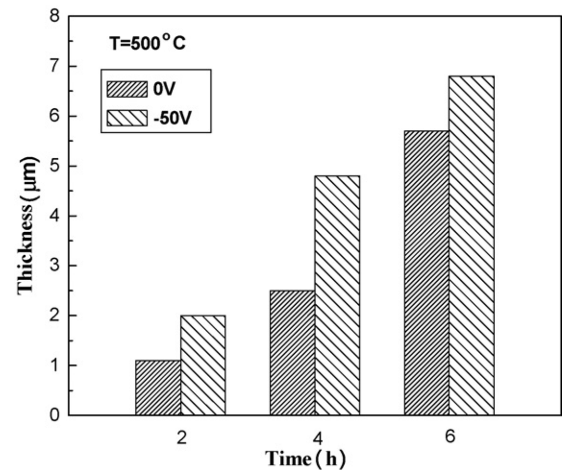

Fig. 3. Variation of thickness of compound layer with different times.

The temperature of the substrates was monitored using a thermocouple. The nitriding temperature was taken from 450 °C to 550 °C range, the process duration of 2 h, 4 h, and 6 h. Some of samples to be treated were placed in a floating potential (Fig. 1b) and the other subjected to a relative lower negative bias voltage of 50 V (Fig. 1a).

The microstructure of the cross-section of the treated samples was studied by optical metallographic techniques. The phase composition of the nitrided layer was determined by X-ray diffraction (XRD) using Rigaku diffractometer operated in Bragg–Brentano geometry using Cu*Ka* radiation. The surface microhardness and hardness profile were determined by a Vickers' indenter with a load of 50 gf.

The corrosion resistance of treated samples was evaluated by measuring polarization curves in 3.5% NaCl solution using ZAHNER IM6e electrochemical workstations. The flat cell which is a three-electrode set-up consists of the specimen as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum sheet used as the counter electrode. The potentiodynamic curves were measured at a potential scan rate of 1 mV/s.

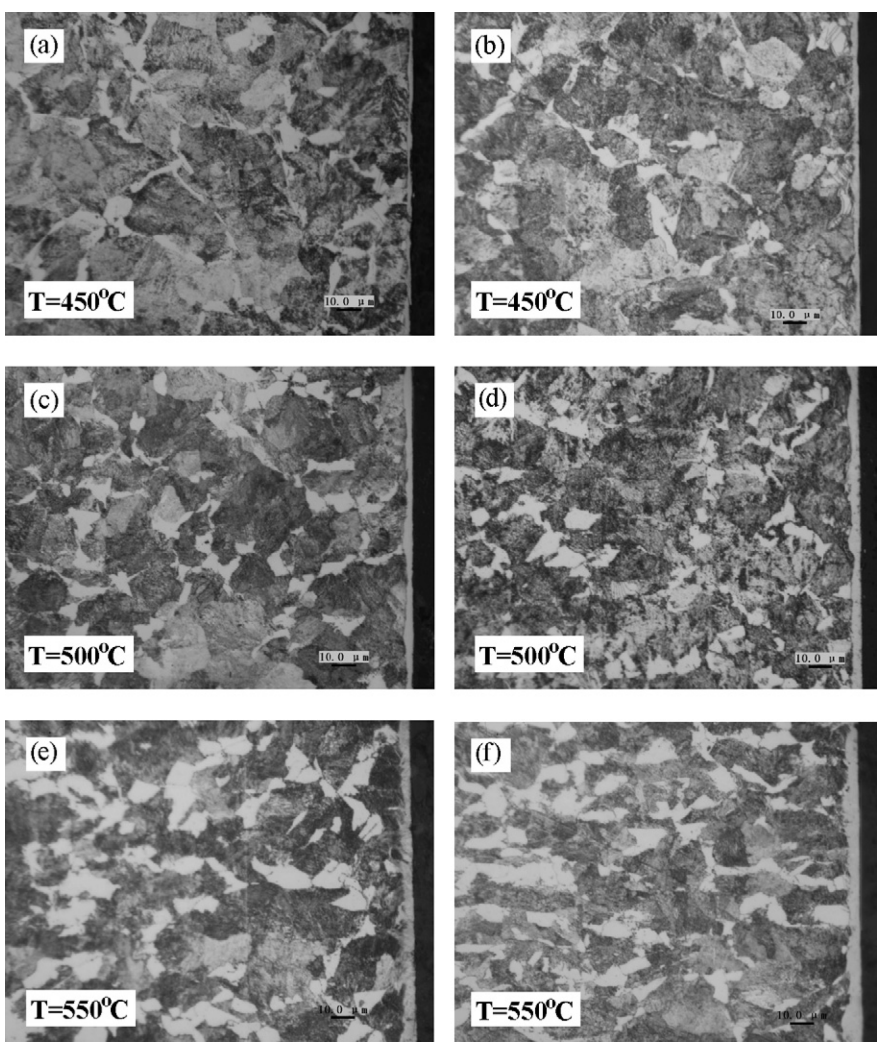

Fig. 2. Optical cross-sections images of samples nitrided with bias 0 V (a, c, and e) and −50 V (b, d, and f).