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Enhanced corrosion resistance of AISI H13 steel treated by nitrogen plasma immersion ion implantation

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Abstract

Electrochemical corrosion measurements of AISI H13 steel treated by PIII process in 3.5% (wt) NaCl solution were investigated. So far the corrosion behavior of AISI H13 steel by PIII has not been studied. The electrochemical results are correlated with the surface morphology, nitrogen content and hardness of the nitride layer. Ion implantation of nitrogen into H13 steel was carried out by PIII technique. SEM examination revealed a generalized corrosion and porosity over all analyzed sample surfaces. Penetration of nitrogen reaching more than 20 μm was achieved at 450 °C and hardness as high as 1340 HV (factor of 2.7 enhancement over standard tempered and annealed H13) was reached by a high power, 9 h PIII treatment. The corrosion behavior of the samples was studied by potentiodynamic polarization method. The noblest corrosion behavior was observed for the samples treated by PIII at 450 °C, during 9 h. Anodic branches of polarization curves of PIII processed samples show a passive region associated with the formation of a protective film. The passive region current density of PIII treated H13 samples (3.5×10^{-6} A/cm^2) is about 270 times lower than the one of untreated specimens, which demonstrates the higher corrosion resistance for the PIII treated H13 samples.

Keywords: AISI H13 steel; Plasma immersion ion implantation; Hardness; Corrosion; Nitride layer

1. Introduction

AISI H13 (DIN X40CrMoV51) is a high strength steel that is frequently employed in industry due to its extraordinary shock resistance. H13 finds applications in hot die work, die casting and extrusion dies. It is also applied in molds for high temperature manufacturing of industrial components. During its production process, H13 steel passes through two sequential treatment steps, tempering and annealing, which allow it to reach 500 HV hardness. Thus it is important to enhance AISI H13 steel wear and corrosion resistance and also increase its hardness by some kind of surface treatment. There are several techniques adequate for this purpose, however in this work plasma immersion ion implantation (PIII) has been used since it can produce homogeneous implantation over all faces of a three dimensional object [1,2] and also because the PIII treatment allows avoiding the formation of a white layer constituted of iron nitrides with different phases [3]. In a previous work [4–6] enhanced nitrogen retention and diffusion in shock resistant steels to depths greater than 1 μm at elevated temperatures have been noted. Hutchings et al. [4] demonstrated that PIII processing can produce duplex layers on 5% chromium tool steel (H13) whereby the non-equilibrium implanted layer is supported by a substantial diffusion zone. So far the corrosion behavior of AISI H13 steel by PIII has not been studied. The aim of the present work is to study the electrochemical corrosion behavior of AISI H13 steel treated at distinct experimental conditions of PIII processing in 3.5% (wt) NaCl solution. The electrochemical results are also correlated with the surface morphology, the nitrogen content and the hardness of the superficial layer.

2. Experimental

AISI H13 steel rod (ϕ=2.5 mm) from Villares Metals which had been tempered and annealed at 550–600 °C, was sliced into 3 mm thick disc samples and then polished to mirror-like finish.

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Table 1
Experimental conditions of nitrogen PIII treatment of AISI H13 steel

Samples	M6	13
Temperature (°C)	450	300
Pressure (Pa)	0.2	0.2
Time (h)	9	12
Pulse length (μs)	60	60
Pulse voltage (kV)	11	11
Frequency (kHz)	1.5	1.5

These samples were fixed onto a sample holder for PIII processing. PIII treatment was performed using the pilot plant system for three dimensional ion implantation described elsewhere [7,8]. The experimental conditions are shown in Table 1.

The morphology and hardness profile of the samples were obtained using a scanning electron microscope LEO 440 and a triboidenter from HYSITRON Incorporated, respectively. Glow Discharge Optical Spectroscopy (GDOS) analysis was carried out using equipment from INA.

The corrosion behavior of the samples was studied by an electrochemical method. Potentiodynamic polarization curves were obtained by means of an EG&G PAR 283 potentiostat using a conventional three-electrode glass cell. AISI H13 steel slices (cross-section ~ 0.07 cm^2) were employed as the working electrode. Two different sets of working electrodes, untreated and treated by PIII processing, were tested. The counter electrode was a graphite rod and a calomel electrode served as a reference electrode. All experiments were conducted at room temperature (~ 25 °C). Polarization curves were acquired in 3.5% NaCl solution, pH = 6 and recorded in electropositive direction at a sweep rate of 0.33 mV s^{-1}, starting from −0.8 up to 0.1 V. The electrochemical measurements were performed in naturally aerated solutions.

3. Results and discussion

3.1. SEM, GDOS and hardness analysis

Fig. 1 shows the surface morphology after electrochemical corrosion of untreated and treated AISI H13 steel samples. Corrosion attack of the samples was carried out in 3.5% NaCl solution by applying one potential step from −0.8 up to 0.1 V. SEM examination of the samples revealed generalized corrosion and porosity over all analyzed sample surfaces. However, the micrograph in Fig. 1b depicts lower surface porosity than the one in Fig. 1a, mainly in the bottom of the image as can be noted in Fig. 1b. The number of pores was determined visually over a representative area of the SEM micrographs of treated and untreated H13 steel samples. For the PIII treated sample at 450 °C there is approximately one pore per every 3 μm^2 while for the untreated sample, the pore number is duplicated for the same area. Similar characteristics were observed for other randomly selected areas of the same sample and also for other treated samples.

This observation is coherent because the H13 steel surface was treated by nitrogen ion implantation (PIII). The nitrogen content profile was determined in another work by GDOS

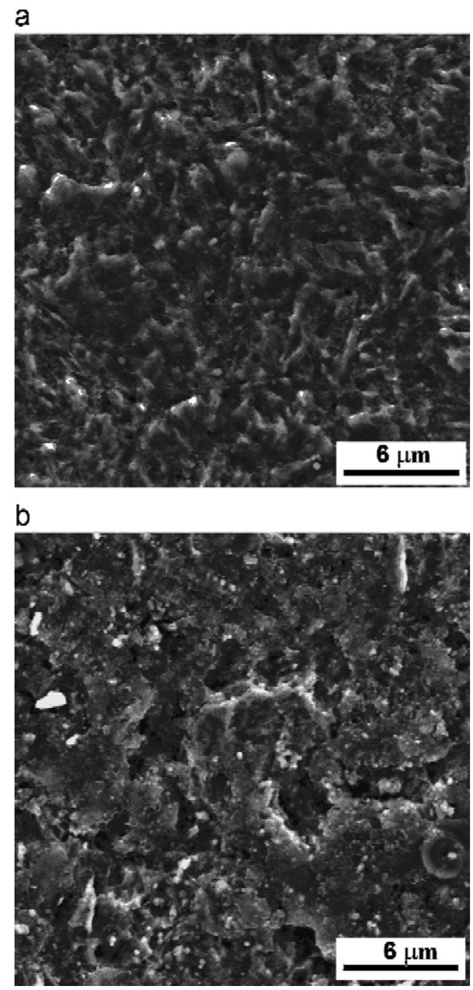

Fig. 1. SEM images of AISI H13 steel after electrochemical corrosion in 3.5% NaCl by applying one potential step from −0.8 up to 0.1 V. (a) Untreated sample, (b) nitrogen PIII processed sample. Magnification: 5000×.

and Auger spectroscopy techniques [9]. Fig. 2 depicts the nitrogen profiles obtained by GDOS for implanted H13 samples under different temperatures and processing times (Table 1). A temperature rise from 300 °C up to 450 °C caused a strong

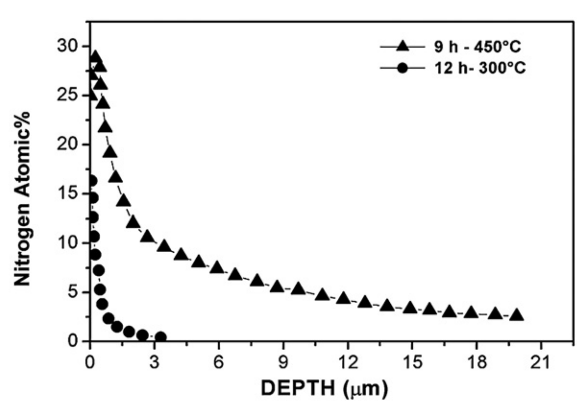

Fig. 2. Profiles of nitrogen atomic concentration of H13 steel implanted by PIII at different treatment times, as a function of depth.