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The effect of temperature on plasma nitriding behaviour of DIN 1.6959 low alloy steel

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Abstract

A series of experiments have been conducted on DIN 1.6959 low-alloy steel using a 5 kVA DC plasma nitriding apparatus with the aim of elucidating the role of treatment temperature in plasma nitriding process. Treatments were carried out in 75%N_2–25%H_2 atmosphere of 4 mbar for 5 h at temperatures ranging from 350 to 550 °C. Optical microscopy, scanning electron microscopy, X-ray diffraction, along with surface roughness and microhardness measurements were utilized to characterize the treated samples. The depth, microstructure, hardness profile and phase constituents of the nitrided layers as well as the surface roughness of the samples were assessed as a function of treatment temperature. The results suggested that the compound layers were mostly dual phase consisting of gamma prime and epsilon iron nitride phases. Increasing treatment temperature increases compound layer and diffusion layer thicknesses. However, maximum surface hardness and roughness were found on the samples treated at 500 and 550 °C, respectively.

Keywords: Plasma nitriding; Ion nitriding; Temperature effect; Low-alloy steel; DIN 1.6959 steel

1. Introduction

Plasma nitriding, also referred to as ion nitriding, is a thermochemical treatment that improves the wear [1,2], corrosion resistance and fatigue properties of the product being nitrided [3,4]. The process was developed in the early 1930s by Egan [5] and Berghaus [6,7] as an alternative to the gas nitriding process. It involves diffusion of nitrogen atoms into the metal surface in presence of a plasma environment. The experimental apparatus consists of a vacuum chamber which is connected to anode and a cathode, where the parts are placed. A nitriding atmosphere, consisting of a mixture of nitrogen and hydrogen gasses, is established at pressures that may vary from 1 to 10 mbar. The voltage applied between anode and cathode typically lies in the range of 300–800 V. The applied potential ionizes the gas mixture. The plasma generated by the ionization, envelops the surface of the workpiece with a blue-violet glow. The charged positive (nitrogen) ions are accelerated and hit the negatively charged workpieces with a relatively high level of kinetic energy. A large proportion of this (up to 90%) is then transferred to heat energy and thereby heats the workpiece to its required nitriding temperature. As a result, nitrogen is transferred to the workpiece, which then penetrates inside by diffusion, leading to the formation of a wear and corrosion resistant layer. This layer, which is called "white" or "compound" layer can consist of iron nitride phases, gamma prime, Fe_4N, or epsilon, $Fe_{2-3}N$ [8]. Below the compound layer there is a diffusion layer or zone where the steel matrix is supersaturated by the in-diffusion of nitrogen atoms. The diffusion zone with its induced compressive residual stresses, enhances fatigue strength of the workpiece. Detail of plasma nitriding process and its parameters have been described by Kovacs and Russell [9].

The response of steel to plasma nitriding mainly depends on its composition [10]. Further to this, though plasma nitriding is influenced by process factors such as time, temperature, chemical composition of the treatment gas, working pressure, width of the glow sheath, the amount of electrical power and the geometry of the chamber.

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Significant data, relating properties and nitriding parameters for different types of materials have been published in the past [11–14]. The purpose of this work is to contribute towards understanding the effect of one of these parameters, i.e. the treatment temperature on properties of the nitrided layer of DIN 1.6959 low-alloy steel.

2. Experimental

The samples to be treated were machined in the form of 25 mm diameter and 7 mm thick discs from DIN 1.6959 low-alloy steel whose chemical composition is shown in Table 1. All samples were hardened, quenched and tempered, (Q & T) to the hardness of 400 HV prior to plasma nitriding. One of the surfaces of the specimens was polished to 1 μm finish using standard metallographic techniques.

Plasma nitriding treatments were carried out using a 5 kVA DC plasma nitriding unit designed and constructed at Amirkabir University of Technology. Treatments were performed with power regulation to maintain the desired temperature. Treatments were carried out for 5 h at different temperatures of 350, 400, 450, 500 and 550 °C. A gas mixture of 75% N_2–25% H_2 under a total pressure of 4 mbar was used for all treatments. Cross-sectional microhardness profiles were obtained on polished surfaces, etched with 2% Nital, using a Zeiss microhardness tester equipped with a Vickers indenter with a load of 100 g. This was also used for surface hardness measurements of the treated samples. The white layer thicknesses and the depth of the diffusion zones were determined by optical microscopy and microhardness profiles, respectively. X-ray diffraction technique was used by employing Cu Kα radiation for the determination of the phase structure of the compound layer. Surface roughness was characterized by measuring the average roughness *Ra* with a Taylor Hobson Surtronic 3+ instrument.

Table 1
Chemical composition of DIN 1.6959 low alloy steel

Element	C	Cr	Mn	Mo	Si	Ni	V
Wt %	0.34	1.1	0.4	0.6	0.15	3	0.1

3. Results and discussion

Typical micrographs of the plasma nitrided samples at 350, 450 and 550 °C can be seen in Fig. 1, showing a continuous compound layer. Nevertheless, the interface of the compound layer and the diffusion layer formed on the sample treated at higher temperature (550 °C) is more uniform than those treated at lower temperatures (350 and 450 °C) in which at some areas along the interface, the compound layer extended into the grain boundaries. This can be attributed to the faster penetration of nitrogen atoms along the grain boundaries and precipitation of iron (carbo) nitrides at there, leading to a non-uniform and uneven compound layer/diffusion layer interface morphology. This non-uniformity can be credited to the temperature dependence of the grain boundary and lattice (volume) diffusion coefficients. At low treatment temperatures, the mobility of nitrogen atoms is low and penetration is done more easily at less compact diffusion routes such as grain boundaries [15]. Therefore, nitrogen atoms diffusing along the boundaries will be able to penetrate much deeper than atoms that only diffuse through the more compact areas such as grains (lattice diffusion). As the treatment temperature increases so dose the nitrogen atoms mobility. Thus, lattice diffusion dominates and the penetration of nitrogen atoms through grains occurs more easily, leading to the formation of a more uniform compound layer, Fig. 1.

Figs. 2 and 3 show the results of the compound layer and the diffusion layer thicknesses as a function of treatment temperatures, respectively. The general trend is an increasing thickness with increasing temperature, as expected for diffusion-controlled growth. Growth of the compound layer is controlled by diffusion of nitrogen through this layer [16]. At low treatment temperature (350 °C) the diffusion coefficient of nitrogen atoms is low and therefore the compound layer and diffusion layer are very thin. Increasing treatment temperature results in an increase in the nitrogen diffusivity, leading to the formation of thicker layers.

The surface hardness measurements of the samples treated at different temperatures are displayed in Fig. 4. It is seen that the highest surface hardness was achieved for treatment temperature of 500 °C. This can be attributed to the temperature dependence of the dispersion hardening of

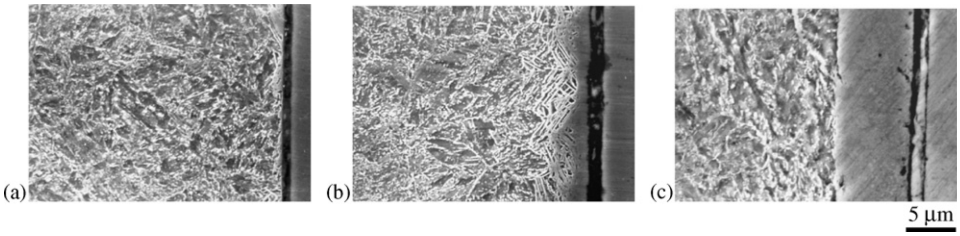

Fig. 1. Scanning electron micrographs of the surface layers of the samples plasma nitrided at various temperatures (a) 350, (b) 450 and (c) 550 °C. Note the difference in the thickness and morphology of the compound layer.