



A rapid D.C. plasma nitriding technology catalyzed by pre-oxidation for AISI4140 steel



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ABSTRACT

D.C. plasma nitriding (PN) of AISI4140 steel catalyzed by pre-oxidation was investigated primarily. The pre-oxidation was carried out in air prior to plasma nitriding. The results revealed that pre-oxidation had significant catalysis effect on plasma nitriding, the maximum compound layer thickness of 15 μm was obtained after nitriding at 500 °C for 4 h under the optimum pre-oxidation (PO) condition of 300 °C and 30 min, which was two times thicker than that without pre-oxidation. The possible catalysis mechanism is due to the formation of a uniform nano-iron oxide particle along with some nanocracks and nanopores on the surface, which has the highest SFE.

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1. Introduction

AISI4140 steel is widely used for gears due to its excellent combined properties. Meanwhile, in order to meet the design criteria of gears, surface modification is necessary to improve their fatigue strength, wear and corrosion resistance. Plasma nitriding is one of the most popular surface modification technologies for AISI4140 steel to obtain the required properties suitable for gears, since it can effectively enhance the related properties due to the formation of a compound layer and nitrogen diffusion zone beneath the compound layer. The compound layer is mainly a combination of iron nitrides, and the diffusion layer is composed of ferrite with nitrogen solution and dispersed precipitates of nitrides [1,2]. Unfortunately, dozens of hours or even longer duration must be consumed to obtain the desired layer thickness and related properties in current application, which results in the low efficiency and high production cost [3].

Pre-oxidation refers to oxidizing in air and transforming the microstructure of the surface layer by the formation of oxide. It has been reported that pre-oxidation has effective catalysis effect on gas nitriding, since the surface layer after pre-oxidation plays a crucial role on the evaluation of nitriding layer [4,5]. However, there have no reports about the effect of pre-oxidation on plasma nitriding.

In this work, the effect of pre-oxidation on plasma nitriding for AISI4140 steel was investigated primarily. The results showed that the pre-oxidation could significantly accelerate the plasma

nitriding and the enhancement mechanism of pre-oxidation was analyzed as well.

2. Experiment

The material used in this study was AISI4140 steel with the following chemical compositions (wt%): C, 0.41; Cr, 0.91; Mo, 0.18; Mn, 0.83; Si, 0.21; P, 0.014; S, 0.011 and Fe, balance. The substrate presented a microhardness of about 300HV_{0.05}. Specimens with the size of 10 mm × 10 mm × 10 mm were cut from a used gear. All the surfaces of samples were grounded and polished by using chromic oxide slurry to achieve a fine finish. And then, the specimens were ultrasonically cleaned in anhydrous ethanol prior to nitriding. Finally, the samples were placed on the working table in the chamber and served as the cathode for nitriding.

The entire treating process was composed of four steps: heating, pre-oxidizing, plasma nitriding and cooling. Firstly, specimens were heated by hydrogen ion bombardment to the designed temperature of 300 °C or 350 °C; and then the supply of hydrogen was substituted by air with a flow rate of 3 L/min to run the pre-oxidation process. After that, air was terminated and the mixture gas of nitrogen and hydrogen with a ratio of 1/3 was supplied at a gas pressure of 180 Pa to run the plasma nitriding process at 500 °C for 4 h. Finally, samples were cooled down to room temperature in nitrogen atmosphere in the furnace.

The cross sectional microstructure and the surface morphology were observed by optical metallography and scanning electron microscopy attached with energy dispersive spectroscopy, respectively. The phases were determined by X-ray diffraction (XRD) with Cu-K α (λ = 1.54 Å) radiation. Hardness measurements were

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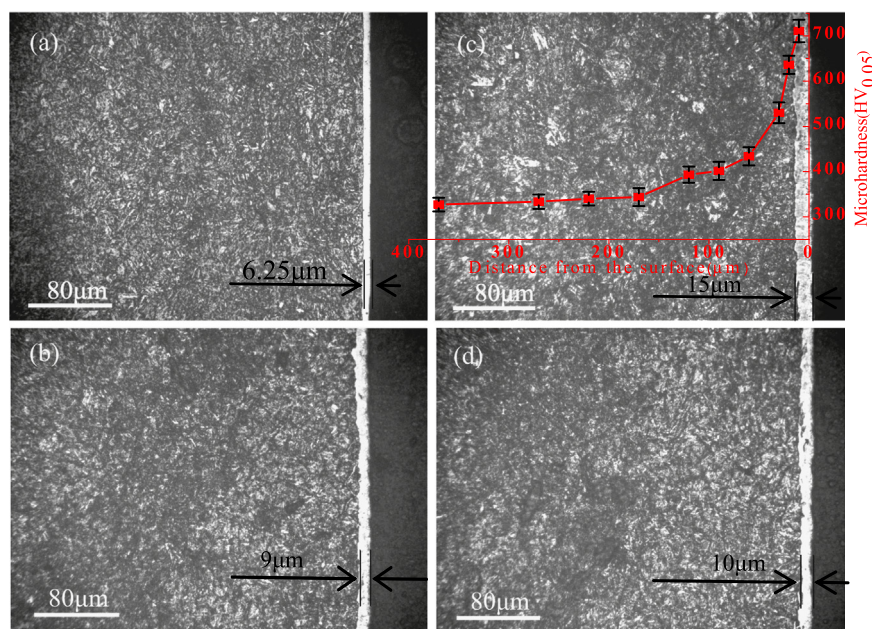


Fig. 1. The cross sectional microstructures and microhardness profile of samples nitrided at 500 °C for 4 h with and without pre-oxidation. (a) Without pre-oxidation, (b) pre-oxidized at 300 °C, 45 min, (c) pre-oxidized at 300 °C, 30 min and (d) pre-oxidized at 350 °C, 30 min.

made in a HXD-1000TMC microhardness tester, with the test load of 50 g and the holding duration of 15 s. Each hardness value was determined by averaging at least five measurements.

In order to calculate the surface free energy (SFE) of the pre-oxidized surface, the contact angles between the liquid drop (distilled water and formamide) and the examined surface were measured by a JC2000D1 system at room temperature. Drops with a volume of 10 mm³ were put on the specimens' surface with a micropipette. Four or five drops were put on each specimen and contact angles were measured for each on two sides of the drops, between six and eight times. And finally the value of SFE was determined with Eqs. (1)–(3).

3. Results and discussion

Microstructure and depth analysis: The cross sectional microstructures of samples nitrided at 500 °C for 4 h with and without pre-oxidation are shown in Fig. 1. It can be clearly seen that the thickness of compound layer was significantly increased by the additional pre-oxidation process, and the maximum thickness of compound layer (also called “white layer”) was obtained under the condition of pre-oxidation at 300 °C for 30 min. And the thickness of diffusion layer can be obtained by analyzing the cross-sectional microhardness profile as shown in Fig. 1(c). As well known, the diffusion layer has higher hardness due to the N solution strengthening, and the hardness decreases with the decrease of N solid solubility. The effective diffusion layer is that with hardness 50HV higher or more than that of the base metal. The detailed investigation on modified surface under different pre-oxidation conditions is given in Table 1.

Surface morphology and EDS analysis: Fig. 2 shows the surface morphology and elemental analysis of samples prior to plasma nitriding with and without pre-oxidation. Compared with the typical surface morphology (Fig. 2(a)) via ion sputtering without oxidation, it can be seen that an oxide layer was formed on the surface and the oxide morphology is closely related to the pre-oxidation conditions. Nano-oxide particles along with nano-cracks and pores are uniformly distributed on the surface with pre-oxidation at 300 °C for 30 min (Fig. 2(b)). However, the surface

Table 1
The comparison of the nitriding layer.

Pre-oxidation (PO)	Temperature (°C)	Time (min)	Thickness of compound layer (μm)	Thickness of effective diffusion layer (μm)
Y1	300	30	15.0	570
Y2	300	45	10.0	470
Y3	350	30	9.00	470
Y0	Without pre-oxidation		6.25	370

Note: All the samples were plasma nitrided (PN) at 500 °C for 4 h.

is nearly free of cracks and with serious aggregation of oxide particles with pre-oxidation at 300 °C for 45 min (Fig. 2(c)), and the aggregation is more serious with pre-oxidation at 350 °C for 30 min (Fig. 2(d)). From these different surface morphologies at different conditions, it can infer that pre-oxidation at 300 °C for 30 min offers the optimum surface characteristics for the following plasma nitriding, which is in good agreement with the results shown in Fig. 1. EDS analysis shows that the main elements in the spot are O and Fe, though some amount of other elements are also detected, which is attributed to the base metal surrounding the particle.

XRD analysis: Fig. 3 illustrates XRD patterns of samples under different conditions, corresponding to those in Table 1. Characteristic peaks of iron oxide were clearly shown for the sample after pre-oxidation (PO). And after plasma nitriding, the iron oxide was totally replaced by nitride for the samples with pre-oxidation at 300 °C for 30 min (Y1), but a mixture of nitride and iron oxide existed for the samples with pre-oxidation at 300 °C for 45 min (Y2), which implies that the iron oxide formed under suitable pre-oxidation conditions (here 300 °C for 30 min) is easier to be transformed to nitride during plasma nitriding, therefore, a plasma nitriding process with higher rate could be obtained. The results are in agreement with Ref. [6].

Discussion: Possible mechanism for plasma nitriding (PN) of AISI4140 steel catalyzed by pre-oxidation is as follows, firstly, the iron oxide formed on the surface during pre-oxidation can create traps for the active nitrogen and the higher nitrogen concentration

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