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The Influence of Plasma Nitriding Time on Mechanical Properties of Plasma Nitriding steel

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Abstract

The aim of this study is to investigate the effect of plasma nitriding time on mechanical and tribological behavior of nitriding coatings produced by plasma-assisted chemical vapor deposition (PACVD). The heat treatment of quench and temper was carried out on hot work AISI H12 (DIN 1.2606) steel samples. A group of samples was plasma nitrided at 470 °C for 2 h in an atmosphere containing 25 vol. % nitrogen and 75 vol. % hydrogen and another group of samples was nitrided on for 4 h, 8 kHz frequency, and 33% duty cycle. The microstructural, mechanical, and tribological properties of the coatings were investigated using optical microscopy, microhardness tester, and pin-on-disc wear test. The load of wear test was 10 N and the samples were worn against different pins, ball-bearing steel (DIN1.3505). The results indicate that the difference of hardness between the samples with different time was 450 HV and the former samples showed a significant amount of wear resistance in comparison to the latter ones.
Keywords: mechanical testing, surface engineering, tool steels, tribology

1. Introduction

H12 hot work steel is recommended for hot tooling applications where maximum resistance to cracking is required. Such applications include hot punches, die casting dies, forgings die, hot shear blades, and extrusion tooling. [1, 2].

Nitriding consists of introducing nitrogen into metallic materials to improve their surface hardness, wear and corrosion resistance, as well as fatigue life. During nitriding of steels, two different structures are formed from surface to core, known as the compound layer and diffusion region. The compound layer consists of iron nitrides of ε phase (ε-$Fe_{2\text{–}3}N$), gamma phase (γ'-Fe_4N) or a mixed phase (ε + γ') developed at the surface. Wear characteristics of the compound layer depend on many factors such as compound layer composition (epsilon/gamma) compound layer thickness, mode of mechanical loading, etc. [3, 4] On the other hand, the diffusion region brings about an improvement of fatigue strength when compared to an untreated material. In this structure, N atoms also dissolved interstitially in excess in the ferritic lattice, give rise to formation of nitride precipitates [5, 6].

Different nitriding techniques have been used in the last years: liquid nitriding, gas nitriding, plasma nitriding. Although these methods are well established, gas and plasma nitriding have some disadvantages from an engineering viewpoint, for example, they may require the use of rather complicated and/or expensive apparatus [7].

The aim of this work has been to investigate the influence of the plasma nitriding time on the wear behavior of H12 steel, used in forging dies.

2. Experimental details

PN treatment with compositional gradients were deposited on a H12 hot-work tool steel substrate using a plasma nitriding system equipped with a voltage-controlled pulse generator. The spectrometric analysis of the substrate is shown in Table 1.

Samples of 50 mm diameter and 10 mm thick were cut for this study. Samples were thermal treated in an industrial furnace as follows. First, samples were austenitized at 1040 ◦C during 45 min followed by quenching at oil. Second, samples were subsequently tempered at 550 ◦C for 2 h.

During plasma nitriding (PN), process parameters such as gas flow, wall heating and voltage duration of pulse-on and pulse-off time and total pressure were monitored. H_2, Ar, and N_2 vapor were used as process gases for PN. Total pressure was kept at 2 mbar and substrate temperature was controlled at 420°C in order to avoid exceeding the tempering temperature of the hot-work tool steel. The processing parameters for plasma nitriding are listed in Table 2. The crystalline structure of the sample was determined by grazing incidence X-ray diffraction (GIXRD) in the continuous scanning mode using CuK_α radiation (λ = 0.154056 nm). The full-width at half-maximum (FWHM) of the Bragg peaks is used to

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approximate grain size base on the Scherrer formula [8]:

$$D = \frac{0.9\lambda}{\beta.\cos\theta'} \quad (1)$$

Table 1.
Chemical composition of hot work steel DIN 1.2606

ELEMENT	Wt%.
CARBON	0.49
MANGANESE	0.50
TUNGESTEN	0.02
NICOLE	0.24
SILICON	0.48
CHROMIUM	5.42
VANADIUM	0.52
MOLYBDENUM	2.65

Table 2
PN parameters.

Parameters	Value
Pulsed voltage	650 V
Duty cycle	33%
Temperature	470°C
PN time	2,4 hr
$N_2/(N_2+H_2)$ flow ratio	25%
Total pressure	2 mbar

Where D is grain size, β is the FWHM of the Bragg peak, and θ is the Bragg reflection angle. The film morphology measured by scanning electron microscopy (SEM). The Vickers hardness of the nitriding layer was measured using a micro-hardness test, within the loading range of 50 g; five micro-hardness tests were performed for each sample to obtain the average values of the hardness.

3. Results and Discussion

Figure 1 shows GIXRD plots for PN treatment for various time durations. Peaks are wide which suggest that is semicrystalline a mixture of amorphous and microcrystalline. S. Li et al. [5] have reported a semicrystalline on steel. A single broad peak indicates that the deposit is microcrystalline with preferred orientation (200). This orientation is dense, so PN layer shows high hardness. By increasing deposition time, structure was dense and also hardness increased (fig.4). Figures 2(a) and 2(b) show crass section of PN treatment for time intervals of 2 and 4 hours, respectively. PN for 120 min (Figure 2(a)) is incomplete. The nuclei of PN over substrate materials are clearly visible. Compared to that, PN deposited for 240 minutes and above covers the substrate almost completely. Black part in Fig. 2(a) was interpreted as a porosity. The existence of the porosity means the PN deposit is incomplete, i.e., it does not cover the surface completely. On the other hands, Fig. 2(b) almost does not have black part. This means that PN deposit in Fig. 2(b) covers the substrate almost completely. During nitriding of steels, two different structures are formed from surface to core, known as the compound layer and diffusion region for 2h.

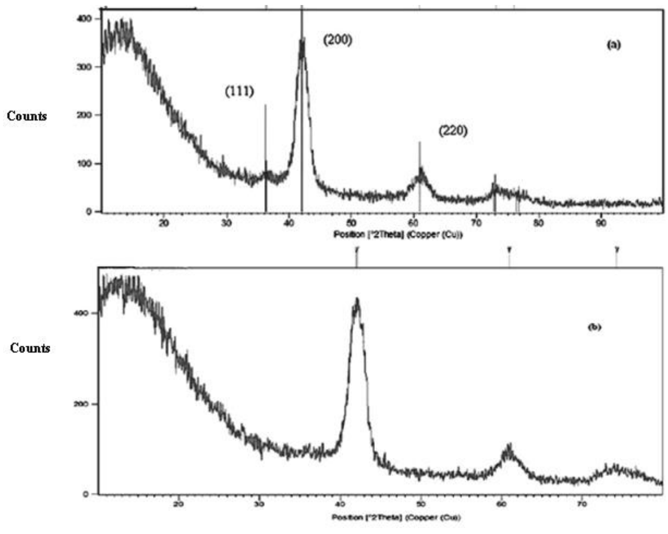
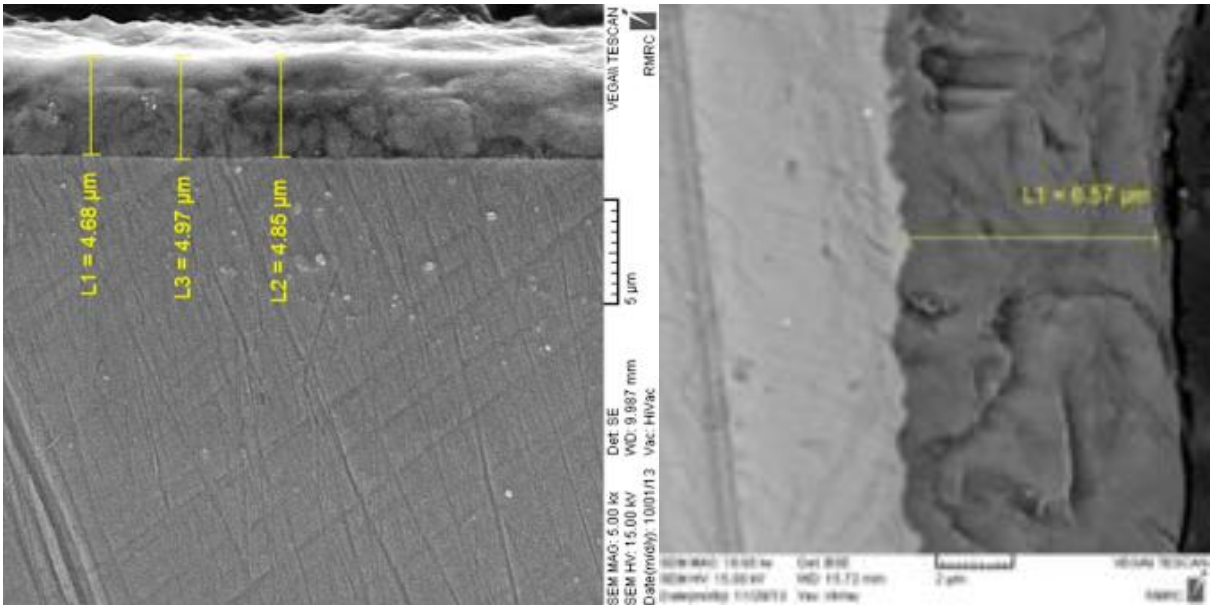


Fig 1: GIXRD plots for PN treatment for various times. a) 2h and b) 4h.



a) b)

Fig 2: SEM images of PN treatment for various time a) 2h and b)4h.

A similar friction behavior was observed for the PN treatment with 4h with a constant friction coefficient of approximately 2h. The friction coefficient starts at a value of approximately 0.2, where it remains constant for a sliding distance of approximately 2800 m. After this running-in period, where the friction coefficient is mainly a result of ploughing and polishing of the surfaces in contact, a sharp drop to a value of 0.2 occurs. Then, the friction coefficient remains constant until the end of the test period of 2800 m without significant fluctuations. (Fig.3)

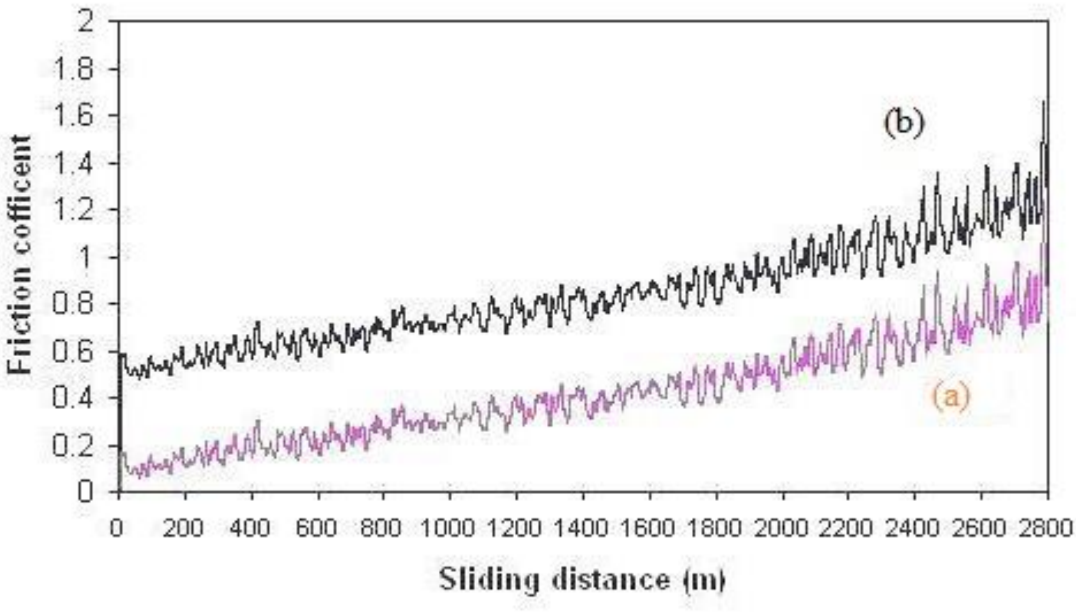


Fig 3: results of wear test a) PN and b) substrate steel.

The resulting hardness values of the are illustrated in Fig. 4 The hardness of the increased with increasing time. The increasing of hardness can be attributed to two reasons, the grain size had also decreased (as shown in Fig.1). Due to the decrease in the grain size, the hardness increased.

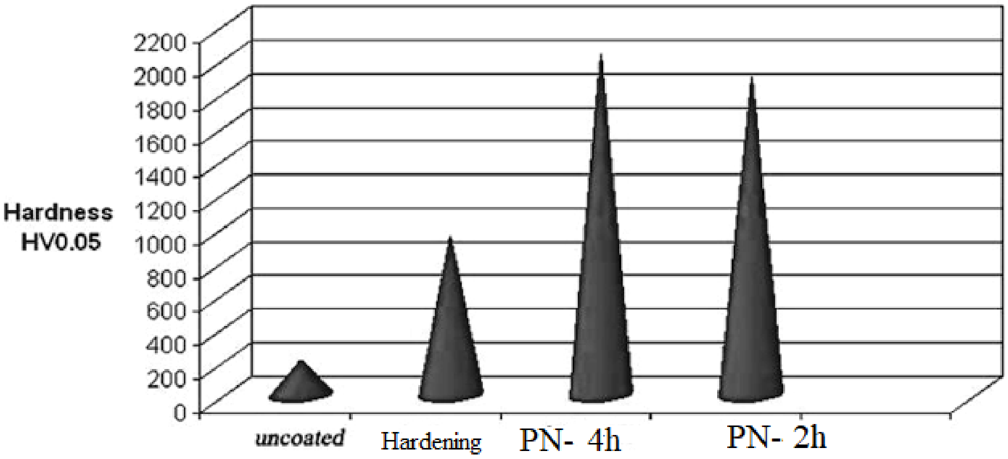


Fig 4: results of microhardness.

4. Conclusions

- PN treatment on steel for different time durations. Very fine nuclei in the deposit were observed the form of islands.
- These results show that growth of PN layer includes of three stages. The first stage is primary growth of nuclei, and then these nuclei join together in second stage. In third stage, secondary growth of these nuclei happens.
- GIXRD plots show a single broad peak indicates that the deposit is microcrystalline with preferred orientation (200). This orientation is dense, so PN layer shows higher hardness than. By increasing deposition time, structure was denser and also hardness increased.

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