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The effect of processing temperature and time on the surface properties of plasma-radical nitrided SKD61 steel

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Plasma-radical nitriding was performed to harden the surface of SKD 61 steel for 1-10 hours at a temperature range of 450-550°C. This process involved the use of NH_3 gas instead of N_2 gas which is employed for the well-established plasma-nitriding method. An NH radical, which played a key role to produce a nitrogen diffusion layer without the formation of the brittle compound layer, was generated in a gas mixture of NH_3 and H_2. One of the main advantages of the plasma-radical nitriding is to improve the surface hardness by maintaining the roughness of the initial polished surface. The microstructures and material properties of the radical- nitrided layer were characterized in order to investigate the effects of various radical-nitriding processing parameters. In addtion, radical nitriding produces better surface roughness, compared with conventional ion nitriding.

Key words: plasma radical nitriding, diffusion layer, compound layer.

Introduction

Recently, plasma ion nitriding has been receiving a great attention due to its reduced distortion and the fact that it is an environment-friendly process. The plasma ion nitriding is a thermochemical process to improve the surface properties, such as wear resistance, corrosion resistance, and fatigue strength, of various engineering steels [1-4]. Plasma-radical nitriding technology is a process in which a gas mixture of NH_3 and H_2 is introduced into the chamber, where the plasma nitriding process is usually carried out in a gas mixture of N_2 and H_2. This radical nitriding process leads to the formation of a diffusion layer about 30-150 μm in thickness without the formation of a brittle compound layer [5]. The roughness of the initial surface is maintained during this process, so that after the treatment, the components do not need grinding before the final application. Conventional plasma nitriding produces a compound layer which should be removed by mechanical grinding before a coating deposition. Therefore, a duplex surface modification treatment combining radical nitriding and subsequent a physical vapor deposition (PVD) CrN(TiN) coating will be a viable technology for coating hot working tools in real industrial conditions.

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In this paper, the effect of processing temperature and time on the microstructure and characteristics of the radical nitrided layer produced on SKD 61 steel have been investigated in detail.

Experimental Procedure

The material used for this investigation is SKD 61 steel which is an alloy steel commercially used for hot forging die for automotive parts. The chemical composition of SKD 61 steel is listed in Table 1. Cylindrical samples (ϕ20 mm × h8 mm) were heated at 1020°C for 1 hour, oil quenched, tempered at 570°C for 3 hours (three times), and air cooled. The surfaces of the samples to be exposed in the plasma were polished and cleaned. The hardness of the samples prior to plasma treatment was 580 $HV_{0.1}$. The vacuum chamber was pumped down to 6.65 Pa and then, back-filled with a gas mixture of NH_3 and H_2 up to 133 Pa (NH_3:H_2 mixing ratio; 1:4). The radical nitrtriding process was immediately carried out with a pulsed DC potential 500 Volts at 450-550°C for 1-10 hours in the glow discharge of the plasma. The samples were heated to the processing temperature by a heating element and

Table 1. Chemical composition of SKD61 Steel (wt.%)

	C	Si	Mn	Cr	V	P	S	Mo
SKD 61	0.40	1.02	0.41	5.15	0.83	0.022	0.0011	1.11

Table 2. Experimental parameters for plasma radical nitriding processing

Temperature (^oC)	Time (Hr)	Discharge Voltage (V)	Discharge Current (A)	Gas ratio	Process pressure	Cooling Condition
450, 500, 550	1, 5, 10	500	1	H_2:NH_3=4:1	133 Pa	Furnace cooling

the ramping rate was maintained by 10 K minute^{-1}. The typical experimental conditions for this work are summarized in Table 2. After the radical nitrtriding, the vacuum chamber was pumped down to 6.65 Pa and the samples have been cooled to room temperature. The microstructures of radical nitrided layers on the surface were observed by optical microscopy and scanning electron microscopy. The phases formed on the radical nitrided surfaces of the specimens were characterized by a Rigaku D/Max-200 X-ray diffractometer using CuKα radiation. Microhardness measurements were carried out with a Micro Vickers microhardness tester using a load of 0.98 N (100 g) and a loading time 15 seconds. The surface residual stress was measured by AIS3000 equipment. A Form Talysurf Series 2 has been employed to measure the surface roughness. By fixing the sampling length as 1.8 mm, Ra (arithmetical mean deviation of the profile) values, which represent the variations of the roughness, were estimated.

Results and Discussion

Figure 1 shows the typical morphology of diffusion layers produced on SKD 61 steel by the radical-nitriding treatment, which shows the effects of plasma treatment time and temperature on the diffusion depth. It can be seen that the plasma-radical nitriding process produced a diffusion layer up to about 150 μm in thickness on the surface of SKD 61 steel, and the diffusion depth increased with increasing treatment temperature and time. This observation is in good agreement with the results obtained from microhardness profiles of radical-nitrided surface layers (Fig. 2). On the other hand, for the experiment carried out at 500°C for 10 hours, the compound layer was formed. It is expected

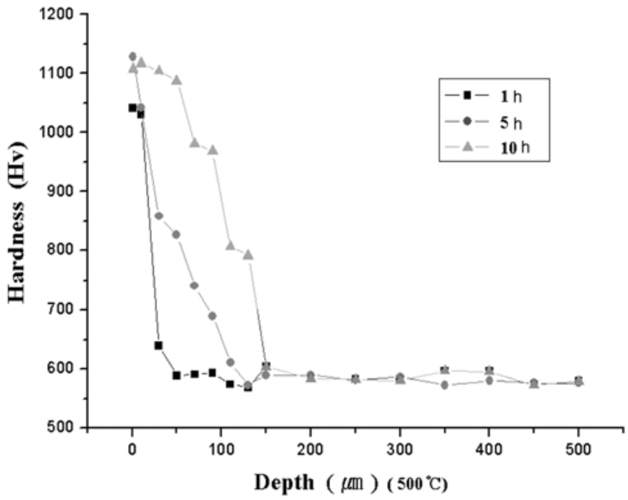

Fig. 2. Microhardness profiles of the surface hardened layer with variation of treatment time.

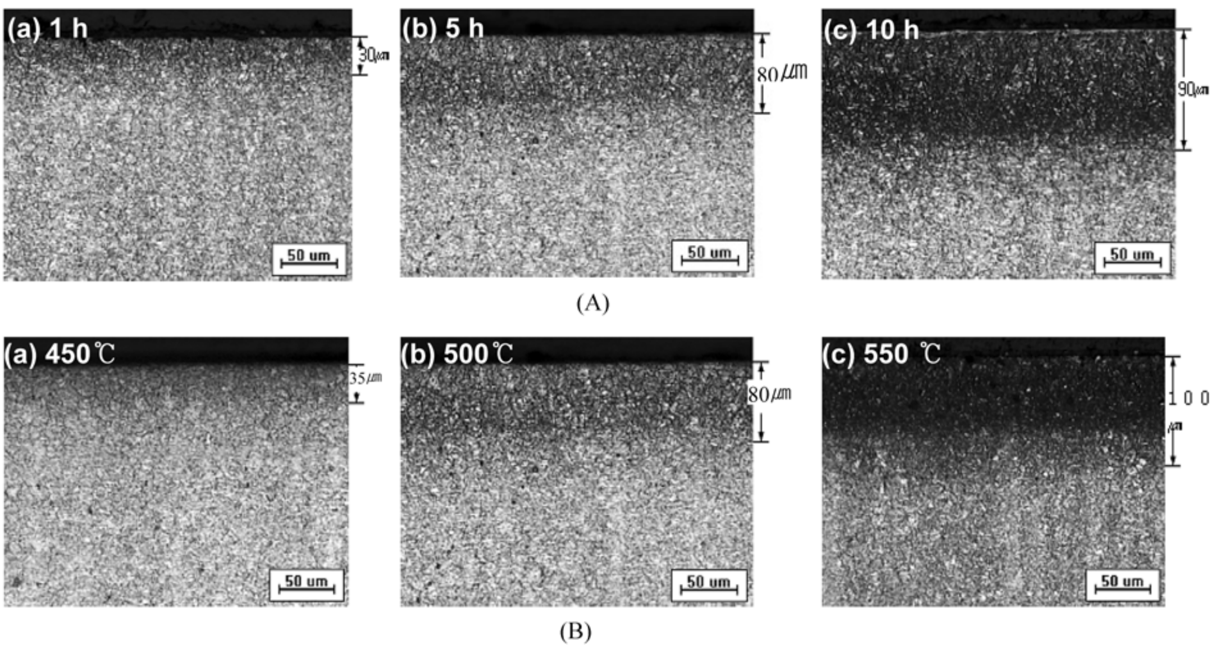

Fig. 1. (A) Optical micrographs showing the effect of plasma-radical nitriding time on the thickness of the diffusion layer at 500°C. (B) Optical micrographs showing the effect of plasma-radical nitriding temperature on the thickness of the diffusion layer for 5 hours.

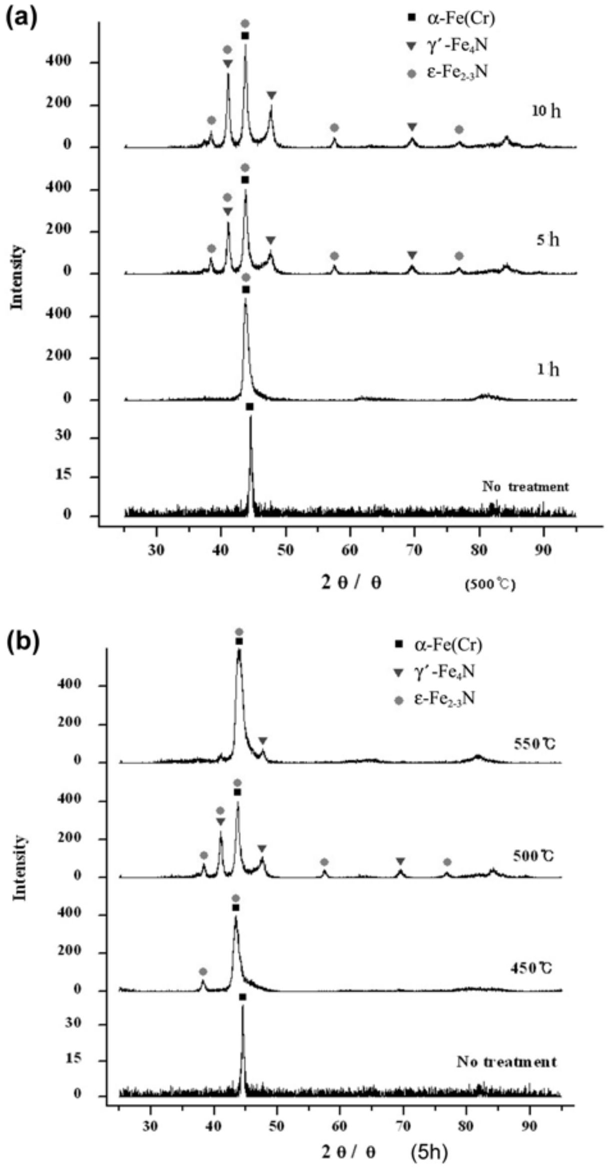

Fig. 3. (a) XRD patterns of radical nitrided layers on SKD 61 steel treated at 500°C for various oxidation times. (b) XRD patterns of radical nitrided layers on SKD 61 steel treated for 1 hour at various oxidation temperatures.

that the treatment time is required to be no longer than 5 hours to avoid the formation of the compound layer. The plasma-nitriding process normally generates a compound layer (white layer) and a diffusion layer on the surface. The diffusion layer contains iron and alloy element nitride precipitates dispersed in the nitrogen-diffused interstitial solid solution of iron. The precipitates were located along the grain boundary in the diffusion zone. It is expected that grain boundaries are a major diffusion path for nitrogen diffusion.

Figure 3 gives XRD spectra of radical nitrided layers produced on SKD 61 steel, which show the effects of plasma treatment time and temperature on nitride formation. The main nitride precipitates in the diffusion layer were identified to be ε-$Fe_{2\text{-}3}N$ and γ'-Fe_4N. It is

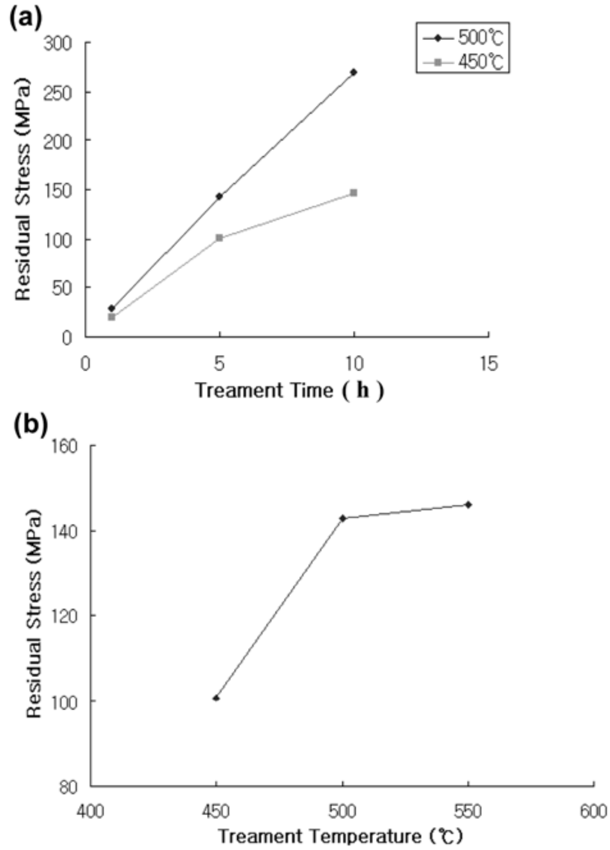

Fig. 4. The variaton of residual stress as a function of treatment time and temperature.

clear that these precipitates play a key role in improving the wear resistance by increasing the surface hardness, due to the restriction of the dislocation motion by the precipitate particles. The amount of the precipitates increases with increasing treatment time and temperature. This can be ascribed to the nitrogen diffusion lenth during the plasma process. However, the experimental results carried out at 550°C for 5 hours, show a decrease in the number of precipitates compared with the treatment at 500°C. It seems that at this treatment temperature, the solubility of nitrogen in the matrix is high, so that there are not many excess nitrogen atoms left to form precipitates.

According to surface roughness profiles of radical nitrided/plasma nitrided SKD 61 steel compared with an unnitrided sample, Ra values of the radical nitrided sample are lower than those of the nitrided sample. Thus the radical nitriding process provides a much smoother surface than the nitriding process.

Figure 4 shows the variation of residual stress as a function of treatment time and temperature. The residual stress in the diffusion layer also increased with increasing treatment temperature and time. This can be associated with the increase of precipitates with increasing treatment temperature and time. The stress is known to inherently arise from the phase boundaries between precipitates and matrix. Therefore, the more precipitates, the higher would be the residual stress. Compared with the interval of 450-500°C, the rate of increase of the residual stress in the interval of 500-550°C is much less. This can be also explained by the higher solubility of nitrogen in the matrix at the higher processing temperature, resulting in a decrease in the number of precipitates and the rate of increase of the residual stress.

Conclusions

It was confirmed that no compound layer was formed during the plasma-radical nitriding process except for the experiment carried out at 500°C for 10 hours. The diffusion depth increased with increasing treatment temperature and time (up to about 150 μm). The main phases produced in the diffusion zone were identified to be ε-$Fe_{2-3}(N,C)$and γ'-$Fe_4(N,C)$. It is expected that the treatment time is required to be no longer than 5 hours to avoid the formation of the compound layer. The value of surface hardness was determined to be 1150 Hv. Plasma-radical nitrided specimens showed better surface roughness compared with conventional plasma nitriding. The residual stress of plasma radical nitrided samples also increased with increasing treatment temperature and time.

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