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EFFECT OF N_2:H_2 RATIO ON SURFACE MODIFICATIONS OF AISI 316 PLATES BY PLASMA NITRIDING PROCESS

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ABSTRACT

Plasma nitriding is used for improving the tribomechanical and chemical properties of the engineering components. In this study, plasma nitriding of AISI 316 substrate has been carried out using microwave plasma enhanced chemical vapour deposition (MPECVD) process by varying the nitrogen and hydrogen ratios. (90:10, 80:20, 70:30, 60:40). Whereas the other deposition parameters such as power, temperature, pressure, deposition duration were kept constant. After deposition process, each sample was then subjected to hardness test, atomic force measurement and microstructural examination. The effect of N_2:H_2 ratio on the mechanical and tribological characteristics of AISI plasma nitrated samples will be discussed in this paper.

INTRODUCTION

Plasma nitriding process is used for improving the tribomechanical and chemical properties such as hardness, wear, friction coefficient and corrosion resistance of the engineering components. The case structure of the nitrided substrate may consist of an oxide layer, a compund layer, an austenite transformed zone (γ phase), and a diffusion zone [1]. A deep nitrogen enriched layer by plasma nitriding process can be achieved at an elevated processing temperature over 500ºC [2]. However, the corrosion resistance is reduced when the processing temperature above 450ºC, even though the hardness continues to increase under this condition [1].

Stainless steels are high corrosion resistance material and maintain its strength at high temperatures but have low wear resistance and poor tribological characteristics. This phenomenon limits their use in some of the engineering applications. Plasma nitriding process can improve the hardness and tribological characteristics. Plasma nitriding at relatively low temperature (below 723 K) in austenitic stainless steel produce extremely hard layer with good corrosion resistance [8]. This phenomenon may be due to the mobilization of chromium and formation of chromium nitride precipitates. In this paper, the effect of N_2:H_2 ratio on the mechanical and tribological characteristics of AISI plasma nitrated samples will be discussed.

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EXPERIMENTAL DETAILS

A commercial stainless steel posses of the following composition (wt. %): C, 0.7; P, 0.03; S, 0.05; Mn, 1.38; Cr, 15.48; Mo, 1.79; V, 0.09; Si, 0.39; Ni, 9.68; Fe, balance was used as a substrate in this study. In this study, the samples were plasma nitrided using Microwave Plasma Enhanced Chemical Vapor Deposition (MPECVD) System. The samples were marked as A, B, C, and D corresponding to the nitrogen/hydrogen ratios of 60:40, 70:30, 80:20, 90:10, respectively. Other process parameters were kept constant as shown in Table 1. Prior to the nitriding process, samples consisting of discs with a diameter of 30 mm and 5 mm thick, were polished to 1200 grid using SiC emery papers. The substrates were then ultra-sonic cleaned with acetone for 30 minutes before being placed in the nitriding chamber.

Table 1: Nitriding parameters

Parameters	Value
Microwave power	1.5 kW
Frequency	2.45 GHz
Substrate temperature	500 ^{O}C
Chamber pressure	1 x 10^{-4} torr

Microhardness of the samples were measured with a Vicker microhardness tester model AKASHI MVK-E using a load of 100 N. The surface morphology of the nitrided samples was observed using Shimadzu AFM equipment. Sample for AFM measurement was cut to a size of 10 mm × 10 mm. Samples for microstructure examination were cut from the nitrided sample, ultrasonic cleaned in ethanol for 30 minutes and then coated with gold. The microstructure of the nitrided layer was observed with scanning electron microscopy model LEO 1525. Samples of subsurafce examination were cold mounted and polished to a surface finish of 1μm and then etched with Adler solution.

RESULTS AND DISCUSSION

Microhardness

Figure 1 shows the microhardness of the nitrided stainless steel substrates. Test results showed that the microhardness of plasma-nitrided sample had increased form 212 HV to a maximum reading of 468 HV. The maximum microhardness was recorded under the nitrogen and hydrogen ratio of 90: 10. As a result of nitriding process, two layers were formed on the substrate, i.e. compound layer and diffusion layer. This phenomenon resulted in the formation of nitrides of iron, chromium etc., in stainless steel, thus greatly enhances the surface hardness of the nitrided substrate. It was also observed that the microhardness decreases with reduction of nitrogen hydrogen ratio.

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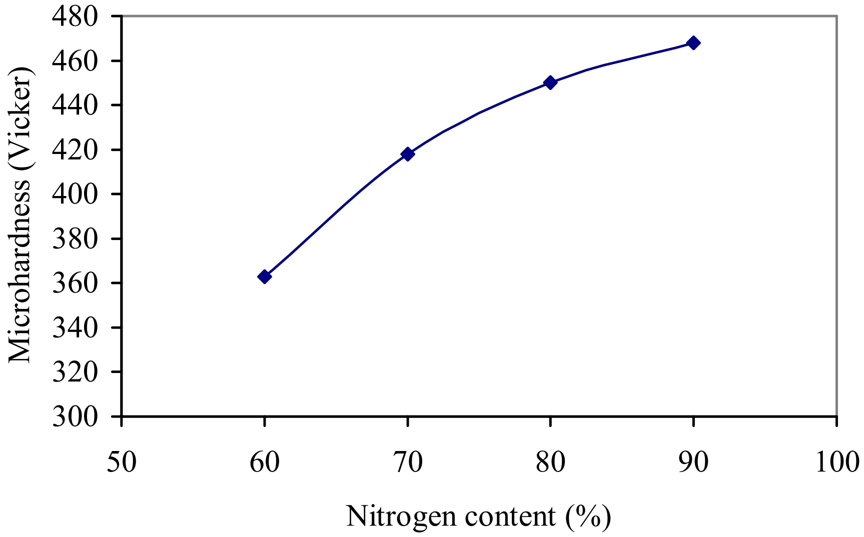

Figure 1: Graph of microhardness versus nitrogen content

Surface Roughness

Atomic Force Measurement results showed that the surface roughness (Rms) becomes smoother with increase of nitrogen ratio (Figure 2). In the early stage of nitriding process, nitrogen atoms condense into suitable nuclie site. Higher nitrogen ratio produced a smoother surface as a result of high nucleation density generation, thus, producing a smoother surface. Whereas, a lower nitrogen ratio produce less nucleation density. As the deposition process progress, the surface roughness of the substrate become smoother as a result of nuclei growth and finally forms a continuous nitriding film on the substrate. Figure 3 shows 2D image of the nitrided samples under different nitrogen hydrogen ratio. It was also observed that the grain size of the nitrided layer becomes bigger in size as the nitrogen ratio is reduced during the nitriding process as shown in Figure 4.

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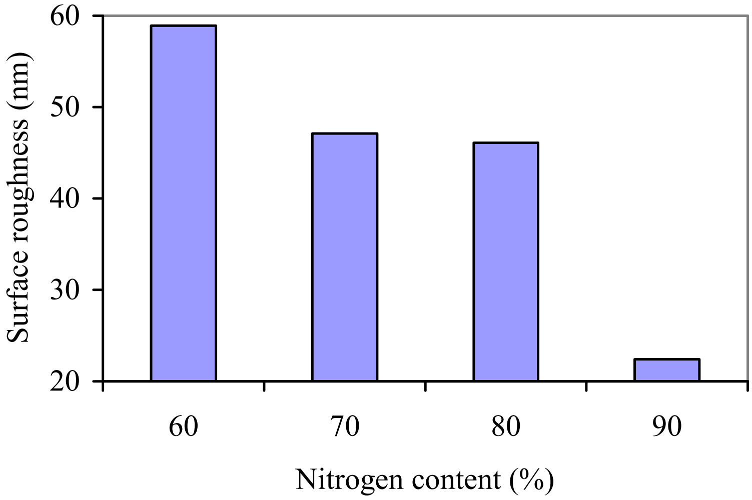
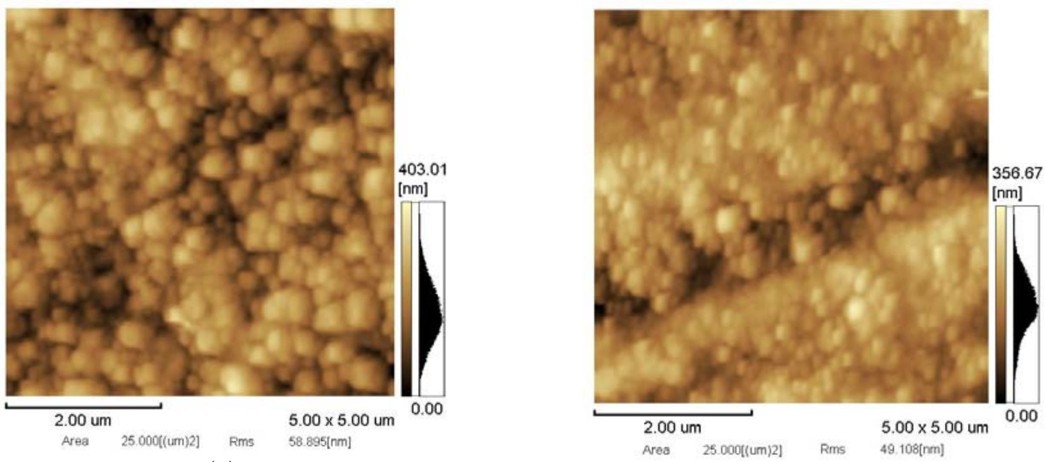


Figure 2: Graph of surface roughness versus nitrogen content

Microstructure

Figure 5 and 6 show SEM images of plasma nitrided sample at different nitrogen/hydrogen ratios. Microstructural examination showed that the nitrogen has mainly been incorporated into the existing iron lattice as interstitial atoms or as a finely dispersed alloy precipitate into the diffusion layer. Microstructural revealed that a higher nitrogen ratio, a very fine and coherent nitride precipitates are formed on the nitrided layer as shown in Figure 5. As the nitrogen ratio is reduced, it was observed that the nitrogen participates are not distributed evenly on the nitrded layer.



(a) (b)

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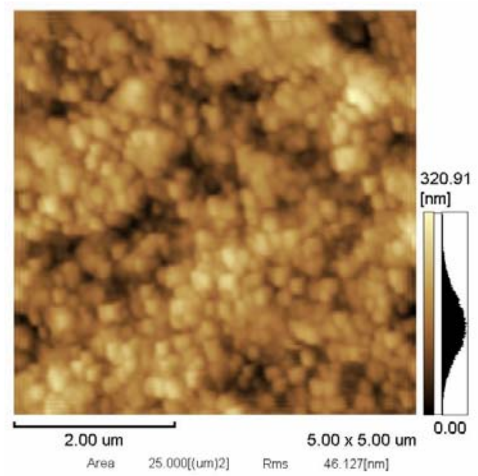
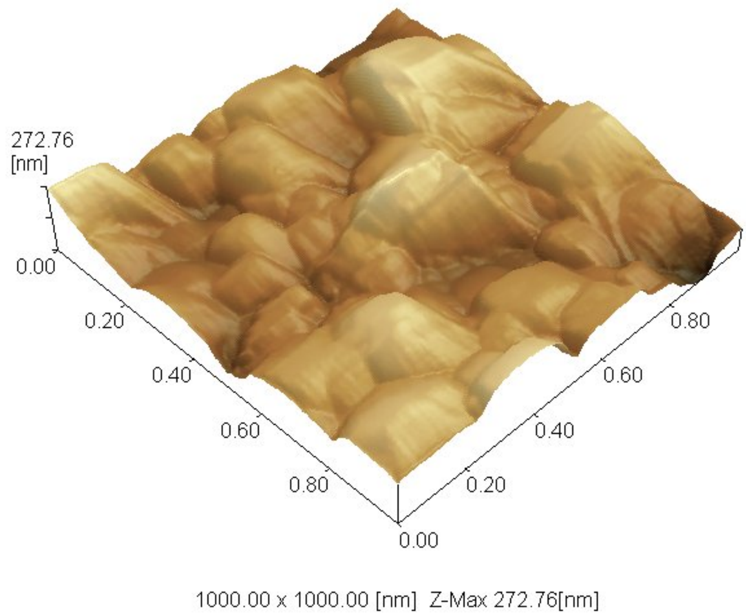


(c)

Figure 3: 2D image with surface roughness result (Rms) at the bottom; (a) Sample B, (b) Sample C, and (c) sample D



(a)

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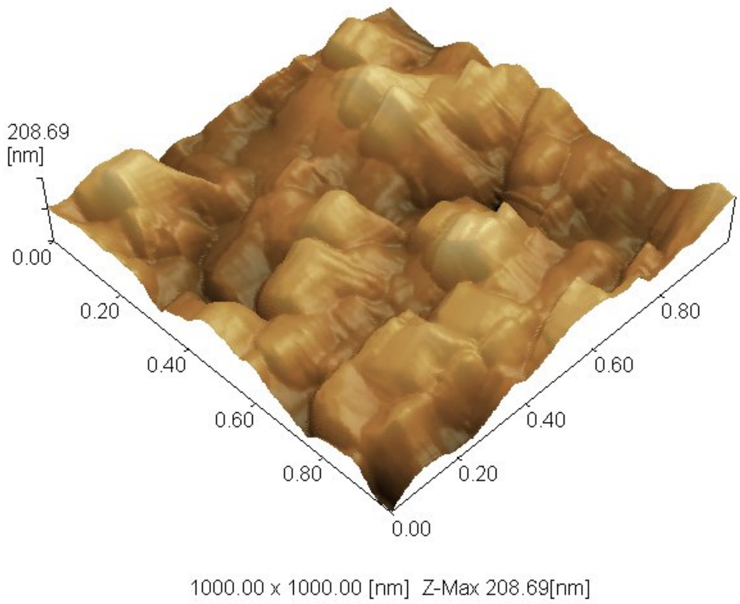

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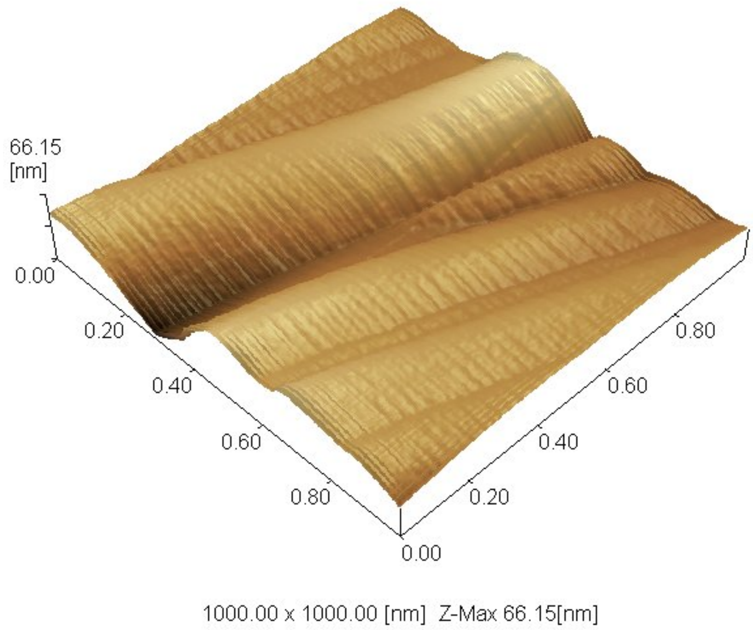
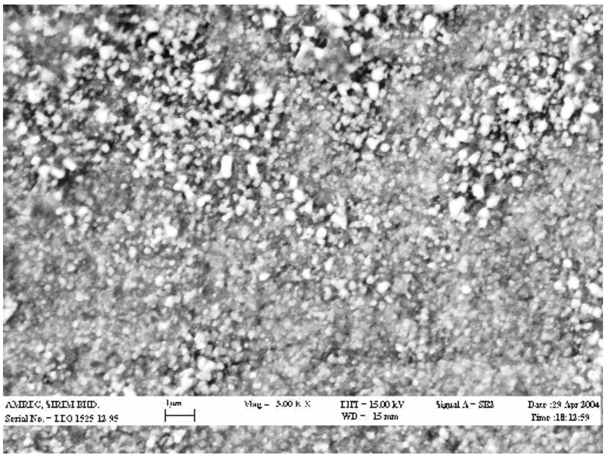


(c)

Figure 4: 3D topographic image of AFM under different nitrogen hidrogen ratio; (a) Sample A, (b) Sample B, (c) Sample D.



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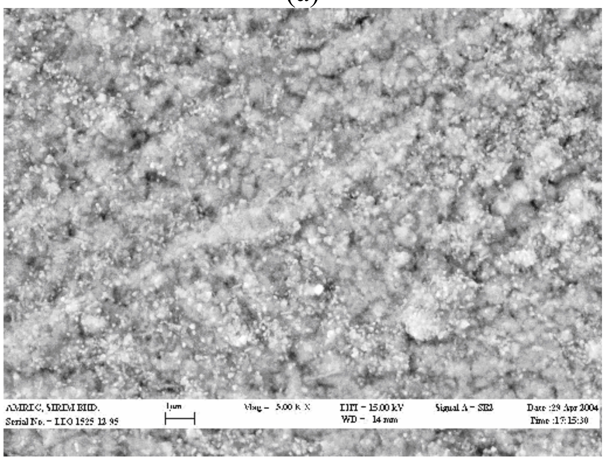

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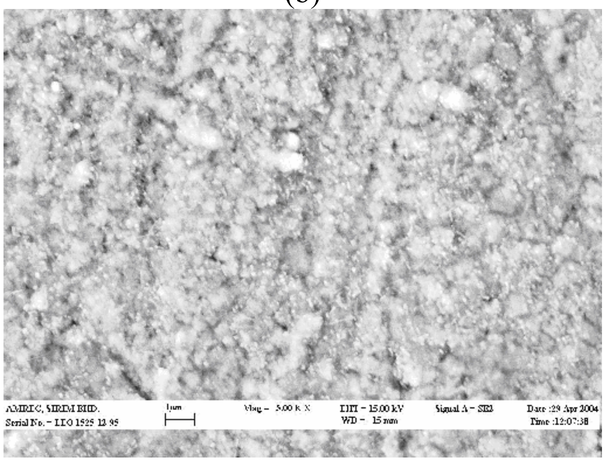

(c)

Figure 5: SEM images of plasma nitrided at different nitrogen and hydrogen ratio**;** (a) Sample A, (b) Sample B, (c) Sample D.

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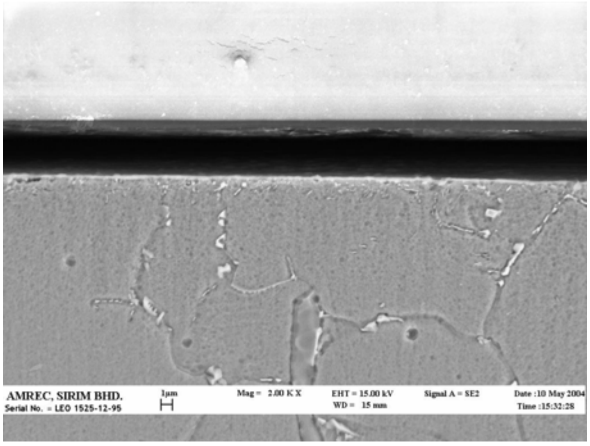

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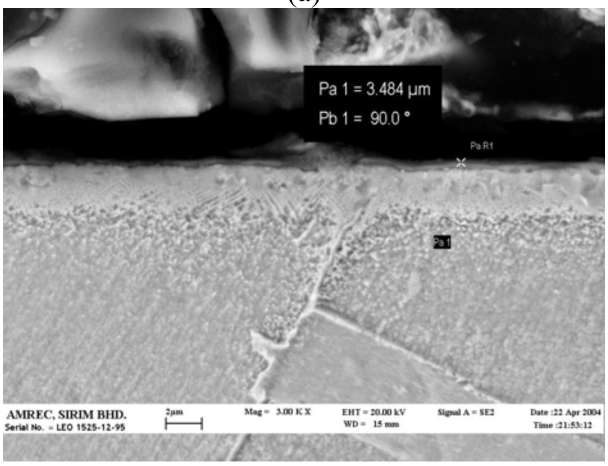

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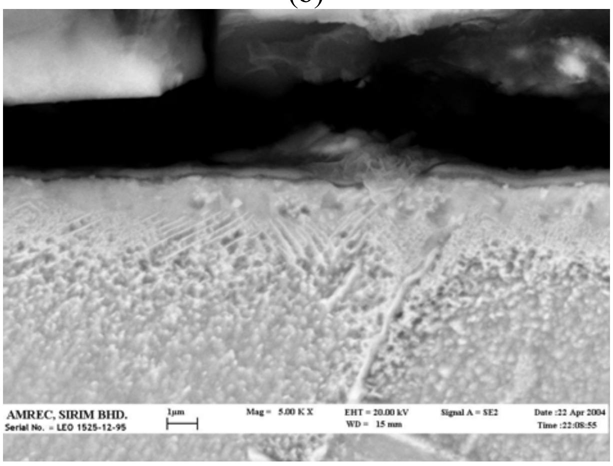

(c)

Figure 6: X-section images of plasma nitrided at different nitrogen/hydrogen ratio**;** (a) Sample A, (b) Sample C, (c) Sample D.

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CONCLUSION

The following phenomena could be concluded on the effect of deposition duration and the surface finish on the microhardness, surface finish and microstructure of the plasma nitrided stainless steels;

i) the maximum microhardness was observed under the nitrogen / hydrogen ratio of 90:10

ii) surface roughness of the nitrided sample became smoother with increasing nitrogen / hydrogen ratios due the formation of smaller grain size of nitride participates

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