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INFLUENCE OF HEAT TREATMENT AND PLASMA NITRIDING PARAMETERS ON HARDENING AN AISI 420 MARTENSITIC STAINLESS STEEL[1]

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Abstract
To increase wear response plasma nitriding have been used with some limitations for martensitic stainless steel type AISI 420. Problems are related to the combination among: hardening, corrosion resistance, wear performance and substrate hardness after the surface treatment. The present paper shows the study of a combination of different heat treatments procedures with plasma nitriding surface treatment using different parameters for MSS AISI 420. Different heat treatments procedures were studied previously to nitriding for a substrate hardness range of 38–50 HRC. Samples were nitrided using DC-Pulsed plasma process. The nitriding temperature ranged from 480 to 560°C, 4 to 16 hours. Samples were characterized by optical microscopy and hardness was evaluated by Rockwell method for substrate and Vickers microhardness for the nitride case. The results show a high hardening effect after plasma nitriding, 1400HV. For substrates treated to 50 HRC the nitriding temperature has a strong effect on the case hardening behavior. Increasing nitriding temperature from 480 to 560°C the substrate hardness decrease considerably and the expected nitriding potential hardening effect is missing. Using 38 HRC as initial substrate hardness the hardening effect is preserved over the same nitriding temperature range. As a consequence different planning between heat treatment and nitriding cycles are discussed.
Keywords: Martensitic stainless steel; Heat treatment; Plasma nitriding.

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1 INTRODUCTION

Martensitic stainless steel are widely used on components requiring high strength and corrosion resistance for oil, chemical, pumps, moulds and other critical structural applications. Plastic mould is one important application for such steels due to needs of highest strength and corrosion resistance in best combination. For such application high strength (or hardness) is required because engineered polymers are more and more abrasives due to fiber reinforced and on the other hand corrosion resistance is required because (i) some polymers are corrosive to the mould steel, as PVC (polyvinyl chlorine) or (ii) hot wall construction technique increases wet corrosion on moulds surface during operation.

Stainless steel type AISI 420 is one of the most used in plastic mould construction. Possibility for hardening on quenching and tempering heat treatment is an important characteristic of such steel. Such treatment increases hardness from 200 HV up to 500 HV (~ 50 HRC). Heat treatment practice suggests austenitizing temperature ranging from 1020 to 1030°C, followed by low temperature tempering close to 200°C.[1] Low tempering temperature is suggested by tool steel producers because of high corrosion resistance.

It is important to point out that surface treatment should not impair corrosion resistance.[2,3] To overcome wear resistance requirements, surface hardening such as nitriding, has been used for martensitic stainless steels.[2,4] An important characteristic for this high chromium steel is the high hardening effect achieved on nitriding treatment. After plasma nitriding surface hardening up to 1400 HV was previously reported.[5,6] This strong hardening effect is consequence of intensive iron and chromium nitrides precipitation on nitrided case. In this paper authors will present how different parameters for heat and nitriding treatments interact and possible solutions to best combine such treatments.

2 EXPERIMENTAL METHODS

The material used in this work was a wrought martensitic stainless steel type AISI 420, received in the annealed state. The chemical composition is presented on Table 1. Annealed microstructure is composed by ferrite matrix with spheroidal carbides dispersion.

Table 1. Chemical composition, mass %

C	Mn	Si	Cr	Ni	V	N	P	S
0.40	0.50	0.95	13.50	0.21	0.27	0.0350	0.027	0.001

Before plasma nitriding treatments all samples were previously quenched and tempered. For hardening the austenitizing temperature was fixed in 1025°C, followed by oil quenched. Tempering temperatures was tested from 200 to 650°C. Nitriding samples were previously tempered at 520 e 580°C, for 50 and 38 HRC respectively. Double tempering, 2 hours each were used. All heat treatments were conducted by vacuum.

Plasma nitriding treatments were carried out in a pulsed-DC plasma reactor with a hot wall chamber. Regarding the plasma nitriding process, one of the most important steps on the nitriding cycle for stainless steels is sputtering to remove the native passive film. For the passive film removal and surface activation the sputtering step was conducted for 1 hour using high intensity pure hydrogen plasma at 300°C for the low temperature nitriding and at 400°C for the high temperature nitriding. The nitriding step was conducted at temperatures between 480° and 550°C, during 1, 4 and 16 hours. A gas mixture composition of $3N_2:1H_2$, work pressure of 250 Pa, and voltage of 470V were used. Nitriding temperature was measured by two thermocouples embedded on the samples.

Microstructure was observed by optical microscopy after etching by Nital 4%. Phases formed on nitrided layer were characterized XRD on a Phillips diffractometer using $Cu_{K\alpha}$ radiation, λ=0.1542nm, in conventional $\theta/2\theta$ Bragg-Brentano symmetric geometry. Microhardness measurements were performed using Vickers indentations with 25 g. Substrate hardness were determined by Rockwell method.

3 RESULTS AND DISCUSSION

Figure 1 shows tempering curve obtained for the region of secondary hardening. Maximum hardening effect is achieved for 500°C, as a consequence of complex carbide precipitation.[7] The results show that 520°C should be the selected tempering temperature for achieve substrate hardness preferred for plastic mould applications, 48 – 50 HRC, and 580°C is the temperature to achieve hardness typical of structural components 36 HRC.

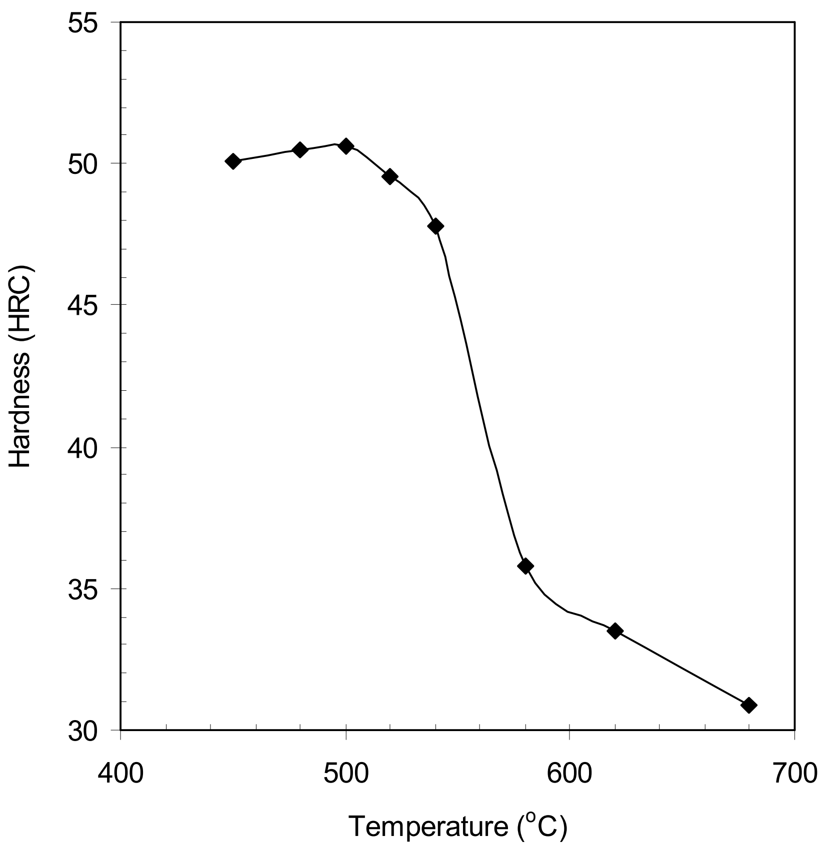

Figure 1. Tempering curve for the martensitic stainless steel type AISI 420.

Figure 2 shows the hardening curves obtained after plasma nitriding treatments for samples previously tempered at 520°C. Nitriding for 1 hour is not enough to achieve maximum hardening. Maximum hardening effect is obtained for

4 hours treatment. For nitriding time of 16 hours surface hardness is lower than that obtained for 4 hours. Hardening behaviour is that expected increasing nitriding time from 1 to 4 hours, but not increasing from 4 to 16 hours. Once achieved maximum hardening effect at 4 hours it would be expected at least maintenance for 16 hours of nitriding. Substrate hardness after nitriding, observed on Figure 2 for depths higher than 0.2 mm, show a softening for higher nitriding times. For short nitriding times substrate hardness is maintained in the tempered level, 470 HV, but for 16 hours substrate hardness down to 351 HV. In this case, surface hardening is accompanied by the substrate softening and the maximum hardening potential can not be achieved. Although nitriding depth increases with nitriding time an adversary effect from substrate softening hinders maximum surface hardening for higher times.

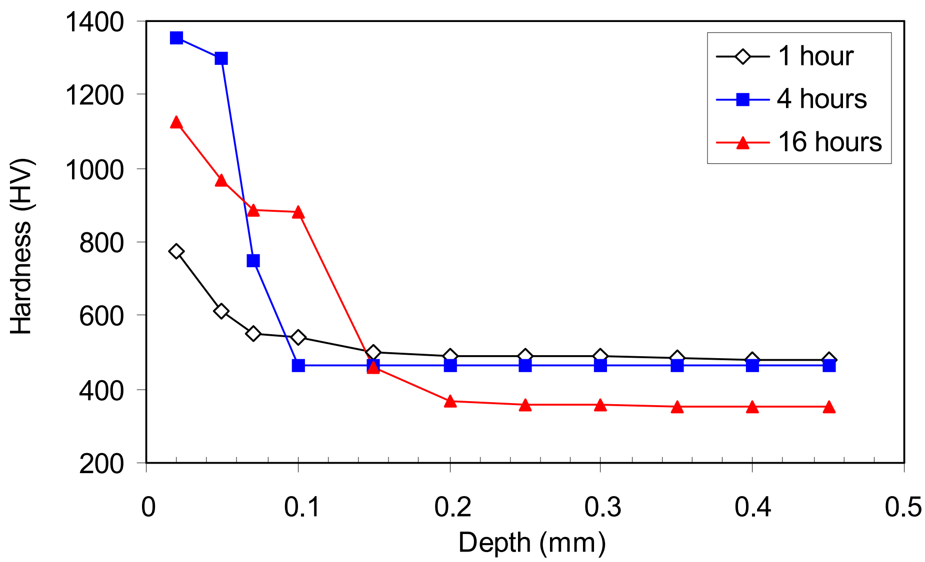

Figure 2. Hardness profiles determined after plasma nitriding at 520°C.

Such results show that substrate softening plays an important role on surface hardness for long nitriding times. To best understand hardness drop during nitriding, aging treatments were performed for samples quenched and double tempered, 2 hours each, at 520°C. Results from Figure 3 show a strong softening on aging at 540°C and even at 520°C. Therefore, when plastic mould tools require substrate hardness close to 49 HRC (470HV), nitriding depth is controlled (i) by nitriding temperature limited to values lower than 500°C, and (ii) because the need of longer times promote substrate softening and lower surface hardening.

For structural components where low hardness level is required a best combination of nitriding time and temperature can be done. Figure 4 shows aging behaviour at 520°C for samples tempered at 580, 620 and 680°C, on hardness level from 30 – 36 HRC. Results show no hardness change and allow a more flexible combination for the nitriding parameters.

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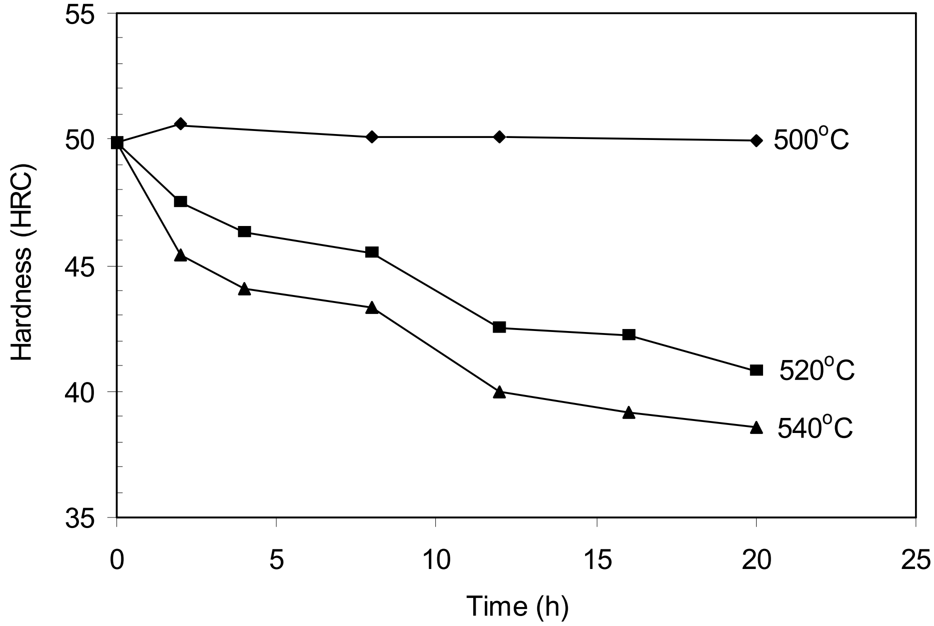

Figure 3. Aging behaviour at 500, 520 and 540°C for samples previously double tempered at 520°C.

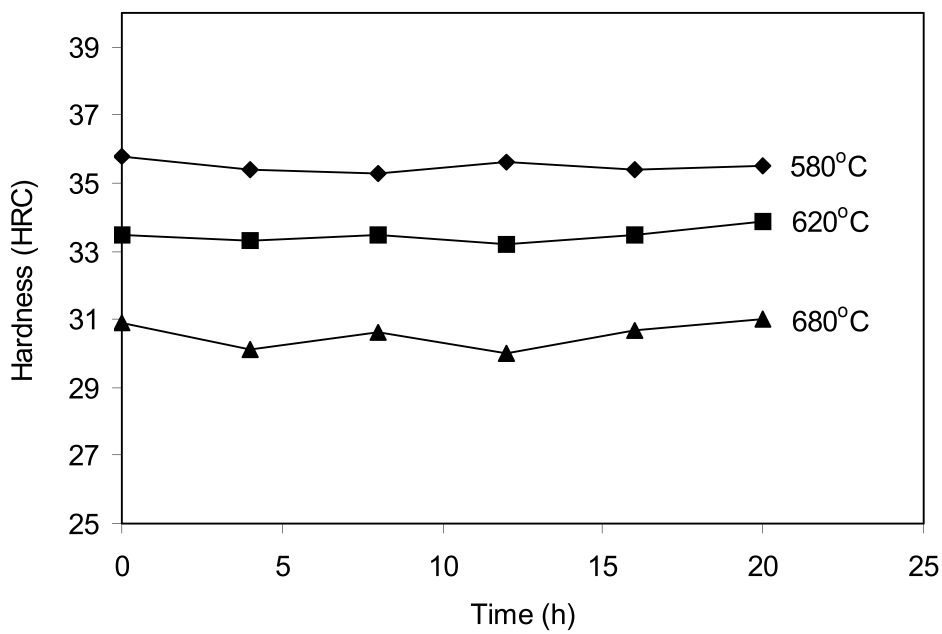

Figure 4. Aging behaviour at 520°C for samples previously double tempered at 580, 620 and 680°C.

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Plasma nitriding treatments were carried out on temperatures between 480°C and 560°C by 4 hours, on samples quenched and tempered at 580ºC with substrate hardness 35.8 HRC (360 HV). Surface microstructures are shown on Figure 5. Plasma nitriding at 480°C and 560°C forms a compound layer, insipient for the lower temperature. Diffusion zone appears as a heavily etched area due to the strong chromium and iron nitrides precipitation, as shown on XRD diffraction pattern on Figure 6. Compound layer is formed by γ'-Fe_4N, ε-$Fe_{2-3}N$, and CrN nitrides.

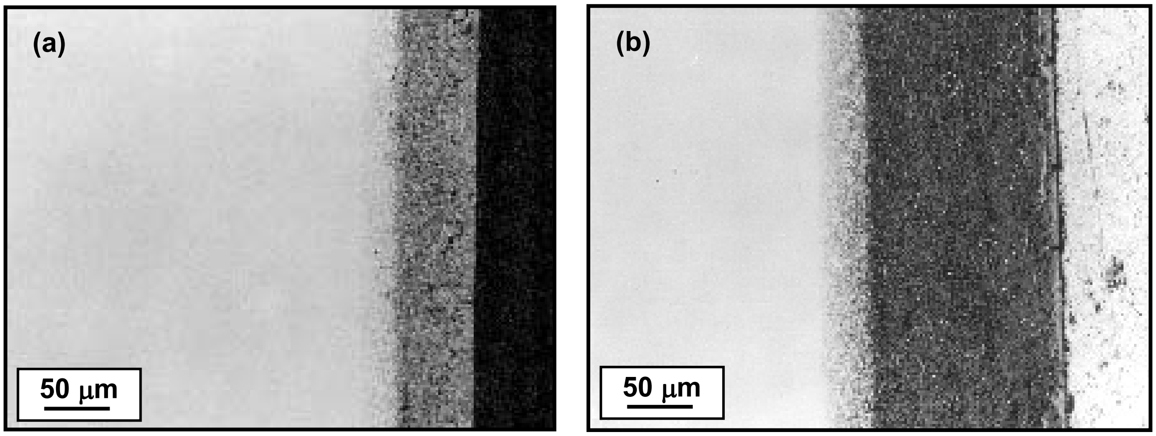

Figure 5. Surface microstructures observed on optical microscopy after nitriding at (a) 480°C and (b) 560°C. Etch: Nital 10%.

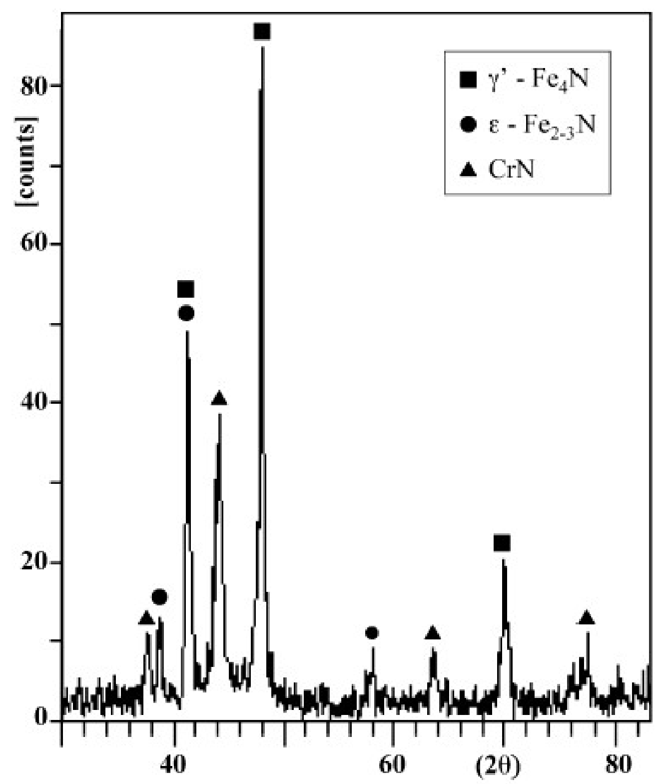

Figure 6. XRD diffraction pattern after nitriding at 560°C.

The hardness profiles, Figure 7, show a strong hardening effect by nitriding, for all tested temperatures. This hardening is due to the precipitation of a fine and homogeneous nitrides[8,9] on diffusion zone, reaching up to 1560 HV. It is important to point out that when substrate is tempered at temperatures higher than that used on nitrided not only maximum hardness is attained, but this hardness exhibit a plateau shape, increasing in depth with increasing the nitriding temperature. Another important characteristic of the hardness profiles is a sharp decrease after the maximum plateau to the substrate hardness. Such behaviour is a consequence of nitrogen profile on the nitrided case[9] this behaviour is a consequence of a strong interaction between nitrogen and carbon[10,11] responsible by complex nitriding reactions taking place at the interface, as presented in previous paper.[6]

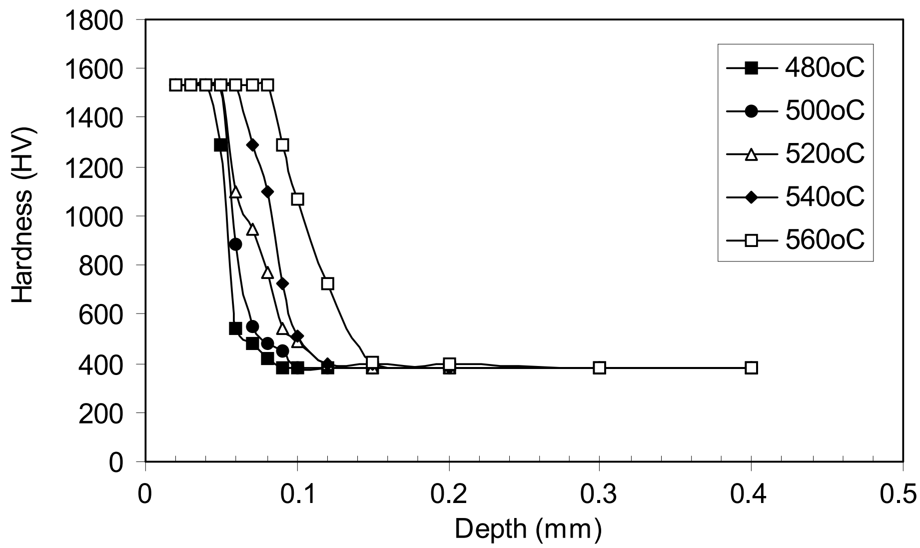

Figure 7. Hardness profiles determined after plasma nitriding, 4 hours, for a substrate tempered at 580°C.

These results show that no change on the substrate hardness is observed, for any position up to 0.2 mm hardness is equal to 390 HV, independently of the nitriding temperature. Of course, this behaviour corresponds to the highest tempering temperature used in previous substrate heat treatment.[5]

4 CONCLUSION

Heat treatment and plasma nitriding processes parameters must be optimized to get the most of surface properties. Considering plastic moulding application, requiring substrate hardness up to 50 HRC, plasma nitriding temperature must be lower than 520°C. For substrates tempered at 520°C, with initial 500HV hardness, the nitriding promotes a strong hardening effect for times up to four hours. Increasing nitriding time surface hardening potential is lost because the over-tempering during the nitriding process act contrary to nitriding hardening impairing the maximum hardening potential for the martensitic stainless steel AISI 420.

When substrate hardness is lowered up to 36 HRC, for example that used on structural parts, after tempering at 580°C, full nitriding benefits are attained. For the nitriding temperatures utilized herein surface is composed by a compound layer

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followed by the diffusion zone, even at lower temperature. The diffusion zone has a smooth interface with the substrate, resulting from growth mechanism process. Hardness profiles present a steady maximum hardness, up to 1560HV, in a depth comparable to the diffusion zone.

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