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Structural and Properties Development of Expanded Austenite Layers on AISI 316L after Low Temperature Thermochemical Treatments

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Abstract: Low temperature Thermochemical treatments in fluidized bed furnace involving nitriding, carburizing, and hybrid treating, sequential carburizing-nitriding, have been conducted in the present work with the aim to improve surface properties of AISI 316L. The resulting layer is expanded austenite which is responsible to the higher hardness and better wears properties. Characterization of this expanded austenite layers were performed including XRD analysis, SEM and SPM and microhardness indentation were used to reveal the characters of the produced thin layers. The study demonstrates that hybrid treating produced a thicker layer and higher surface hardness with gentle-drop hardness profile compared to the other treatments. This is caused by the presence of dual expanded austenite γ_C and γ_N compared to individual nitriding and carburizing which only produces a single layer. XRD patterns of treated surfaces showing the absence of nitride and carbide precipitates. SPM-c characterization reveals that expanded austenite has densely packed morphology which is responsible to the hardness improvement as well as high wear resistant surfaces.

Key words: Structural properties, expanded austenite, hardness transition, sensitization

INTRODUCTION

Austenitic stainless steel is widely used in the chemical and petrochemical industry, cryogenic vessels, heat exchangers, machineries for paper, pulp, textile, pharmaceutical equipments and internal combustion engine (Abdullah *et al.*, 2010). The type 316L is more resistant to pitting and crevice corrosion due to having more than 2 to 3% molybdenum and less than 0.04% carbon (Bazargan-Lari and Bazargan-Lari, 2009). This type of steel has excellent corrosion resistance and forming characteristics. However, due to its inherent austenitic structure this material has relatively low hardness as well as poor wear resistance.

Sensitization is a common problem in austenitic steel where precipitation of chromium carbides ($Cr_{23}C_6$) occurs at the grain boundaries at elevated temperatures. This precipitation is due to the depletion of chromium from its matrix and consequently unable to produce Cr_2O_3 passive layer to make stainless feature. As a result, it reduces the corrosion resistance property of the stainless steel. This phenomenon causes reduction in ductility, toughness and aqueous corrosion resistance (Matula *et al.*, 2001).

It is known that surface hardness of the steels can be increased by the addition of carbon and nascent nitrogen.

In carburizing hardness is increased only by sacrificing the corrosion resistance property of the material. During the carburizing process, the carbon atoms diffuse through the surface and occupy lattice sites of the stainless steels. These excess carbon atoms have the affinity to react with the chromium present in stainless steels and form chromium carbide ($Cr_{23}C_6$). So as explained the sensitization property of the stainless steels comes into the picture. But in nitriding, the problem of sensitization is not encountered. Instead, diffusion of nitrogen into surface layers of steel promotes the formation of hard nitrides. In case of stainless steels, it is expected that the complex hard Fe-Cr nitrides form on the surface, which in turn could improve the wear resistance. Nitrogen is very strong austenite stabilizer and strongly promotes an austenite structure. So the advantages of nickel addition/alloying to promote an austenite structure can also be obtained by the case hardening stainless steels by nitriding. It also increases the mechanical strength substantially. Beside that, in cold forging when the higher stress was applied, the shorter die life. In order to improve die performance, nitriding and nitrocarburizing can also be proposed to improve die life (Abdullah *et al.*, 2007).

The low temperature nitriding makes the surface layer of austenitic stainless steel hard and nitrides-free

(Ichii *et al.*, 1998). This surface layer so-called S phase can obtain the hardness around 1000 Hv and keeps good corrosion resistance of austenitic stainless steel (Thaiwatthana *et al.*, 2002; Christiansen and Somers, 2006; Yamauchi *et al.*, 2001).

On the other hand, because of the low temperature diffusion treatment the S phase has a thin layer and steep drop of hardness at the border between the hardened layer and substrate as the character of nitriding. Low temperature carburizing also forms an S phase like layer at the surface of austenitic stainless steel. These carburized layer have a gentle drop of hardness but have a lower surface hardness than that of Nitrided S phase (Sun and Bell, 2002). Furthermore, low temperature nitriding of austenitic stainless steel shows the push-in effect of dissolved carbon (Yamauchi *et al.*, 2001; Fewell *et al.*, 2001; Abd El-Rahman *et al.*, 2005; Bell, 2008). Hence the case hardening process of combination carburizing-nitriding is chosen for this study. Based on these observations, the investigations in the possibility of implementing the hybrid process of low temperature nitriding-carburizing, which involves the incorporation of nitrogen and carbon simultaneously into austenitic stainless steel surface to form an alloyed zone comprising a carbon-enriched layer on the top of nitrogen-enriched layer are still challenging. The structure, thickness and quality of the alloyed zone can be controlled by the processing parameters, such as temperature, time and gas composition in the fluidized bed. The duplex surface layer by combined carburizing and nitriding of 316 L steel should be thick and mildly dropping hardness profile. This process has similarity with the conventional nitrocarburising process for ferrous alloys. But the nature of hybrid process is apparently different to that of conventional nitrocarburising. The nitrocarburising is employed at a temperature around 570°C with the purpose to produce a carbonitride compound layer at the surface, whilst the present hybrid treating applies to austenitic stainless steels and is carried out at much lower temperatures (350-450°C) with the purpose to produce a hybrid structure without nitride and carbide formation.

The duplex surface layer produced by combined carburizing and nitriding, so called hybrid surface alloying process, on austenitic stainless steel 316 L, has demonstrated thicker layer and gentle dropping hardness profile treated in plasma chamber (Sun and Haruman, 2006). Because of push-in effect of dissolved carbon during subsequent nitriding (Yamauchi *et al.*, 2001; Fewell *et al.*, 2001; Abd El-Rahman *et al.*, 2005; Ozturka *et al.*, 2009; Tsujikawa *et al.*, 2005a) a distinct dual layer is discernable under metallographic

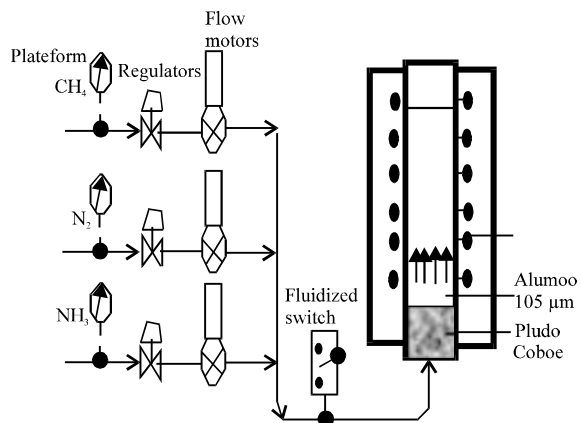


Fig. 1: Schematic picture of fluidized bed furnace

observation. Nitriding of austenitic stainless steel 316 L in a fluidized bed furnace has been successfully performed (Haruman *et al.*, 2006) but the hybrid process and the sequential carburizing-nitriding in fluidized bed furnace have not been cited elsewhere. This study describes characteristics of surface alloyed austenitic stainless steel 316 L treated by low temperature thermochemical treatment at 450 and 470°C at 8 h duration.

The schematic picture of fluidized bed furnace is shown in Fig. 1. Recent study has shown that low temperature thermochemical treatments of austenitic stainless steel is possible in a fluidized bed furnace.

MATERIALS AND METHODS

The substrate material used in this study was AISI 316L type austenitic stainless steel of following chemical compositions (in wt.%): 17.018 Cr, 10.045 Ni, 2.00 Mo, 1.53 Mn, 0.03 C, 0.048 Si, 0.084 P, 0.03 S and balance Fe. This steel was supplied in the form of 2 mm thick hot-rolled plate. Samples of 20×70 mm size rectangular coupon were cut from the plate. The sample surface was ground on 320, 600, 800, 1000, 1200 grit SiC papers and then polished using 1 μm Al₂O₃ pastes to the mirror finish. Before treating, these specimens were cleaned with acetone.

Nitriding, carburizing, nitrocarburising and hybrid treatments were performed at 450 and 470°C in a fluidized bed furnace having particulate alumina as fluidized particles which flow inside the chamber due to the flow of nitriding or carburizing gases. Electrical resistance heating heated the specimens. Prior to treating, the specimens were soaked in concentrated HCl (2 M) solution for 15 min duration with the purpose to remove the native oxide film that commonly forms on austenitic stainless steel and protects the metal matrix from corrosion. This

Table 1: Hybrid treatment conditions and their corresponding layer thicknesses

Treatment	Symbol	Time (h)	Gas (%)			Gas (%)			Temp. (°C)	Thickness (µm)
			CH ₄	N ₂	NH ₃	CH ₄	N ₂	NH ₃		
Hybrid nitrocarburizing	8 (C+N)	8	450	5	80	15	0	0	0	5.5
Hybrid nitrocarburising	8 (C+N)	8	470	5	80	15	0	0	0	12.2
			1st step (C)			2nd Step (N)				
Carburizing+Nitriding	4 C-4N	4+4	450	5	95	0	0	85	15	5.2
Carburizing+Nitriding	4 C-4N	4+4	470	5	95	0	0	85	15	8.4

oxide layer is believed to act as a barrier for diffusional nitrogen transport (ASM, 1991).

Table 1 shows details of each fluidized treatment conditions with specimen symbols. For example, 8(C+N) specimen was treated by nitrocarburising process for 8 h with a mixture of 80% nitrogen gas, 15% ammonia gas and 5% methane gas for 8 h. The 4C-4N specimen was processed sequentially, first treated by carburizing for 4 h followed by nitriding for 4 h. In such a sequential process, the gas mixture changes were carried out within 10 sec without temperature change.

After treatments, the surface morphology of the treated specimens were characterized using Field Emission Scanning Electron Microscope (FESEM) and further corroborated with Scanning Probe Microscope (SPM) at higher resolution for the hybrid alloyed surface. Prior to this metallographic examination the polished specimens were etched by Marbles reagent (4 g CuSO₄+20 mL HCl+ 20 mL H₂O). Microhardness profile with 3g load of Vickers indentation was made across each resultant layers were also performed to reveal the surface hardness of carbon autenite (γ_c) and nitrogen autenite (γ_N). X-ray diffraction (XRD) analysis using Cu-K α radiation (40 kV, 150 mA) was employed to identify the produced phases from various process conditions. Thus, conforming the data from the above characterization techniques, the surface characteristics of treated specimens from various treatment conditions are determined. The specimens were further characterized by microhardness indentation and elemental analysis by EDS.

RESULTS AND DISCUSSION

Surface morphology and topography of Hybrid treated specimens at 470°C: In t he hybrid process, both nitrogen and carbon species are simultaneously introduced into the fluidized retort at a treatment temperature sufficiently low to prevent a formation of nitride and carbide precipitates in the surface alloyed zone. This process has similarity with the conventional nitrocarburizing process for ferrous alloys. But the nature of hybrid process is apparently different to that of conventional nitrocarburizing.

Commonly, hybrid nitrocarburizing is employed at a temperature around 570°C with the purpose to produce

a carbonitride compound layer at the surface, whilst the present hybrid treating applies to austenitic stainless steels and is carried out at much lower temperatures (350-450°C) with the purpose to produce a hybrid structure without nitride and carbide formation. Table 1 shows the summary of resulting layer thicknesses. For example, the hybrid 8 (C+N) specimen at 470°C has a thickness 12.2 µm and the hybrid 4C- 4N as dual stages specimen has up to 8.4 µm and its consist actually of two separate zones with a somewhat diffused interface which clearly observed under microscope but not revealed in the micrographs. The outer zone is γ_N and the inner zone is γ_c . Conversely, the hybrid nitrocarburized sample shows a distinct separation of the γ_N and γ_c layers, with the γ_c layer closest to the austenite substrate (as indicated in the micrograph in Fig. 2b).

Figure 2 shows the SEM micrograph of hybrid processing at 450-470°C. This hybrid treated dual layer structure was distinguishable under microscope but not clearly revealed in the present micrograph. However, using the Scanning Probe Microscopy the presence of this dual structure can be clearly seen in 3D picture, as depicted in Fig. 3 which shows that the bottom layer consisting of γ_c structure is more deeply attacked by the Marble etchant as compared to γ_N structure of the top layer and thus appears darker as indicated in the picture. The surface topography of hybrid treated specimens fr om top view and cross sectional of expanded austenite were also investigated by a scanning probe microscope. The AFM was operated in the contact mode and the measured signal height. Figure 3 shows the 3D surface topography of the specimen hybrid at 450 and 470°C for 8 h. It is observed that domains of about 1-2 microns, as observed in SEM, consist of agglomerates of granular structure and showing the results of deposition process containing some pore channel.

Figure 3a and b shows the topography of hybrid transition layer after treated at 450 and 470°C, the roughness profiles measured across the grains, also showed the presence of nano-features of dimensions of about 60 nm and about 1.84 - 2.47 µm in height. As shown in Fig. 6, the existence of carbon expanded austenite and as confirmed by XRD is located below the nitrogen expanded austenite, compared to previous study, the thickness is thinner than that produced by plasma hybrid

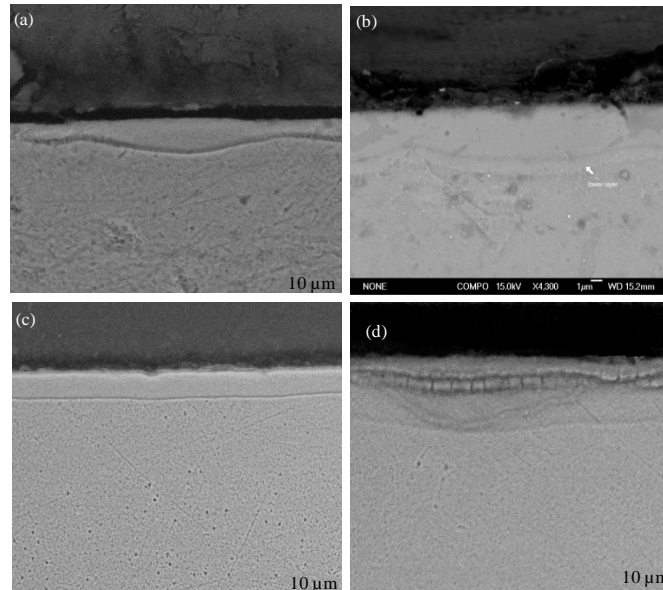


Fig. 2: SEM micrographs of Hybrid treated specimens for 8h: (a) Nitrocraburised 450°C (b) Nitrocarburiized 470°C (c) Dual stages 450°C (d) Dual stages 470°C

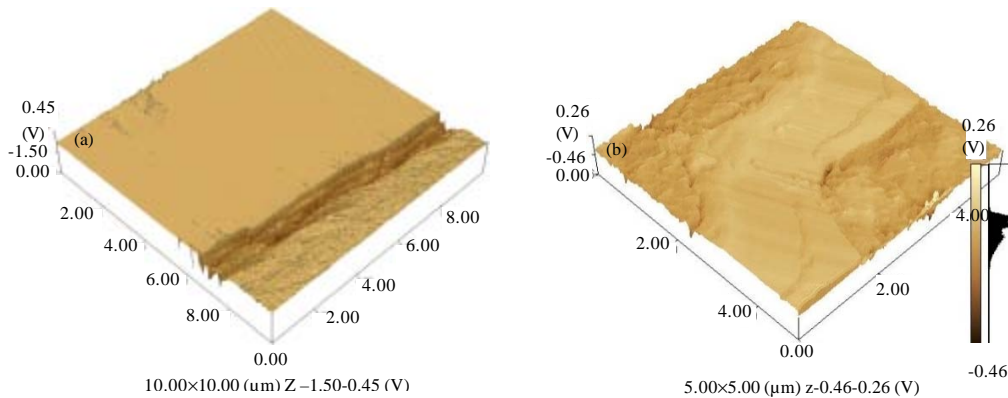


Fig. 3: The AFM topography of hybrid transition/interfacial layers hybrid dual stages specimens at (a) 450 and (b) 470°C

processing. The average roughness as well as the grain size is found to increase with increase in treatment temperature. The average grain size increases from about 87.92 nm after hybrid at 450°C for 8 h to 168.2 nm on hybrid at 470°C for 8 h and the corresponding roughness values varied from 432 to 1280 nm, respectively. Figure 4a and b shows the 3D surface topography of the specimen treated at 450 and 470°C for 8 h. It is observed that domains of about 1-2 microns as observed in FESEM consist of agglomerates of smaller grains of 2470 nm.

Hardness profile: The hybrid process results in the formation of a dual layer structure with an exceedingly hard nitrogen-enriched layer on top of a hard carbon-enriched layer and nitrogen and carbon reside in the relevant layer to form a precipitation-free and supersaturated solid solution with expanded austenite structure, thus attaining increased hardness than other specimens treated by individual carburising and nitriding. The push in effect of dissolved carbon by nitrogen species in hybrid process developed a dual-thick layer

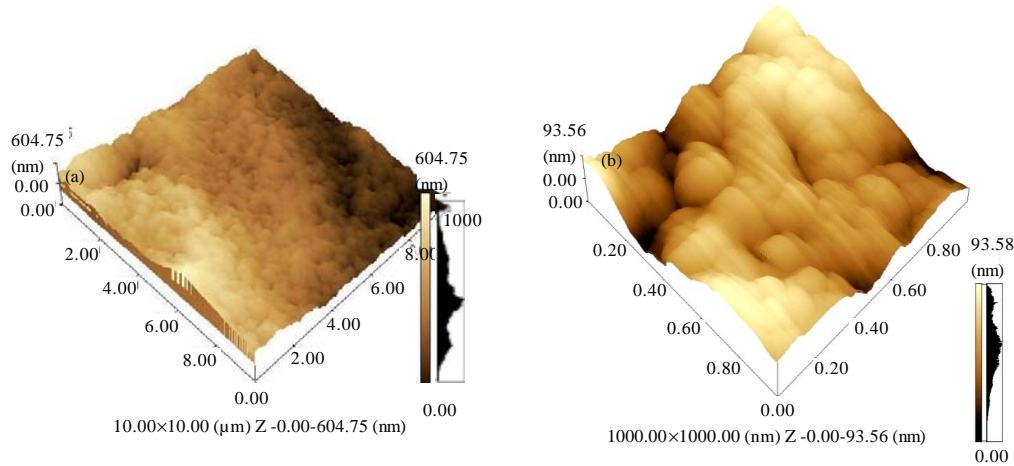


Fig. 4: The AFM surface topography of hybrid specimens at (a) 450°C (b) 470°C

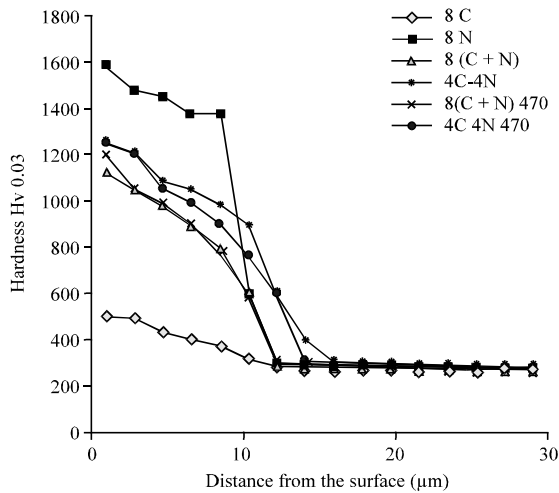


Fig. 5: Depth profiles of microhardness

and thus a gentle-drop in hardness profile was created across the layer thickness, as indicated in Fig. 2. Figure 5 shows that nitriding (8N) produces a sharp-drop in hardness profile compared to other low temperature surface alloying methods due to less nitrogen diffusion in austenitic lattices. Such a gentle hardness distribution in dual layer from hybrid process is expected to be beneficial in achieving enhanced tribological and load bearing properties.

For similar treatment duration, the plasma process is reported (Tsujikawa *et al.*, 2005b) to produce about much thicker layers compared to those of the present treatments in fluidized bed furnace. In plasma process the native oxide layer is removed mostly by bombardment of the plasma charged particles which is completely absent in conventional fluidized process. This is one of the reasons

why conventional fluidized bed treatment produced small layer thickness compared to the corresponding plasma processing.

Elemental profiles of nitrogen-carbon and XRD analysis:

The carbon profile investigated on 4C-4N specimen using EDS is depicted in Fig. 6. It is found that higher carbon at the deeper layer which indicates that carbon pushed-ahead by the incoming nitrogen atom and the dissolved carbon is accumulated at the front of the nitride layer which has also been reported by Tsujikawa *et al.* (2005a,b).

Elemental analysis of the Hybrid nitrocarburized specimen 8 (C+N) gave more carbon beyond the nitrided layer but some carbon was also observed at the surface.

The variations of chemical concentration in the hybrid nitrocarburized layer were also measured with EDS. Figure 6 shows the typical nitrogen and carbon profiles produced in treated 316L steel. It can be seen from these Figure that there are two features in the nitrogen and the carbon profiles. Firstly, the nitrogen profile on the surface of the treated layer is similar to that of nitrided 316L steel. Secondly, the maximum nitrogen concentration is on the surface and the maximum carbon concentration appears beneath as if carbon was ‘pushed’ to the middle of the layer by nitrogen.

Such distributions of nitrogen and carbon in the surface layer are likely to produce some beneficial influences upon the properties of hybrid treated 316L steel. Figure 6b shows results of nitrogen concentration on hybrid dual-stages obtained from energy dispersive X-ray (EDS) analysis. According to these curves, it can be clearly seen that the surface hybrid layer contains very high amount of nitrogen and nitrogen concentration is

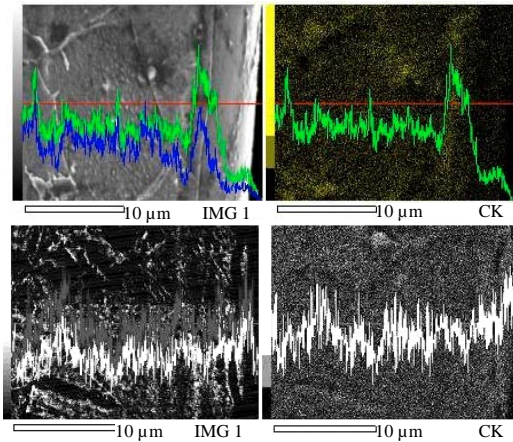


Fig. 6: Carbon profiles (a) and nitrogen profiles (b) along the depth

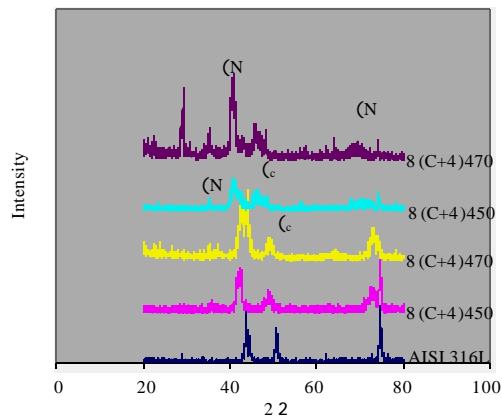


Fig. 7: Comparison of XRD patterns of hybrid treated specimens at 450 and 470 °C

gradually reducing from surface to the core with distance increasing. Due to a low diffusion rate in the case of samples at low temperature, nitrogen penetration depth at 450°C is less than that at temperatures 470°C. However, some carbon remains in the sub-surface layer.

In the present study, the nitride and carbide precipitates were not detected by XRD analysis in the whole treated surfaces as shown in Fig. 7. This result is in close agreement with the previous works (Tsujikawa *et al.*, 2005a; Triwiyanto *et al.*, 2010) on individual nitriding and carburising where the formation of nitride and carbide precipitates will develop at the higher temperature of 450°C. As confirmed by XRD analysis, the nitriding treated surface layer comprises mainly the S phase or the expanded austenite.

Thermodynamically, chromium nitrides and carbides could form under the hybrid processing conditions. On the other hand, at sufficiently low processing temperature the rate of precipitation is so slow that for a short time treatment the produced N-enriched layer is precipitation-free. Analysis shows the expanded austenite (S phase) layer formed on the nitrided and carburised surfaces but with a slight displacement of diffracted angle between (2θ) between expanded nitrogen austenite (γ_N) and expanded carbon austenite (γ_C) as recorded by XRD diffractogram in the above figure. This occurrence is predictable due to dissimilar intensity of lattice distortion created by individual nitrogen and carbon dissolved in the face centered cubic (fcc) austenite lattices. The low temperature nitride and carburised layers thus have a relatively simple structure with all the incorporated nitrogen or carbon atoms residing in the austenite lattices. The carbon content is the maximum at the surface, up to 3.0 wt.% and decreases gradually towards the layer-core interface, exhibiting a diffuse type carbon and nitrogen concentration profile. Such a significant amount of carbon or nitrogen dissolved in the layer obviously leads to the supersaturation of the austenite lattices. This causes several important structural and property modifications to the austenite in the near surface region. Firstly, the austenite lattice in the surface alloyed layer is expanded as evidenced from XRD and TEM electron diffraction analysis (Tsujikawa *et al.*, 2005b; Blawert *et al.*, 2000). The lattice expansion in carburised layers is about 3%, as compared to 9% lattice expansion in low temperature nitrided layers. The expansion and distortion of austenite lattices result in the formation of high density dislocations and stacking faults in the carburised layer as well as nitrided layer (Tsujikawa *et al.*, 2005a,b) and thus resulted in an improvement of surface hardness of autenitic stainless steel.

For the hybrid process, consisting of dual layers (Fig. 2), revealed another thin interfacial layer. This interfacial layer as shown in Fig. 3 is believed to be due to the accumulation of carbon as has also been reported in literature (Sun and Haruman, 2006). The X-ray diffraction pattern of treated AISI 316L is shown in Fig. 7. γ_C γ_N are identified as the only phase present in the surface adjacent region, i.e., within the information depth for the probing X-ray beam. The position of the peaks depend on the temperature of the treatment and a slight shift towards lower 2θ angles is observed for the samples of thermochemical treatments in comparison with untreated sample. The larger amount of nitrogen incorporated in this layer is dissolved in the austenite lattice, causing lattice expansion. The lattice constant calculated from the d-spacing for the (200) plane is higher than the lattice constant calculated by the d-spacing of the (111) plane. The lattice parameter of the γ_N phase increases with

nitrogen concentration and can be as large as 0.390 nm, as compared to 0.358 nm for face-centred cubic austenite of the substrate. This registers a lattice expansion by nearly 9% which causes significant hardening effect and induces many structural disorders in the nitrided layer, such as stacking faults, dislocations and residual stresses (Ozturka *et al.*, 2009).

CONCLUSION

The thermochemical treatments of AISI 316L stainless steel in a fluidized bed process at 450 and 470°C demonstrate that it is possible to produce hard layer of an expanded austenite phase without precipitation of chromium carbide/nitride.

Hybrid treating which simultaneously introduces carbon and nitrogen into the substrate will produce thicker layer compared to individual carburising and nitriding. The hybrid process thus successfully integrates the low temperature nitriding and carburising processes and results in the formation of an alloyed zone characteristic of these two individual processes. As a result of nitrogen pushing effect to the carbon species during the hybrid treating, dual phase layer will form consisting of nitrogen expanded austenite at the outer region and carbon expanded austenite at the inner-region. Nitrogen expanded austenite (γ_N) produced by nitriding performed higher hardness value than carbon expanded austenite (γ_C) produced by carburising, thus a combination of these will lead to a smooth hardness profile resulting in much improved load bearing capacity of the austenitic stainless steel without compromising its corrosion resistance.

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