Surface mechanical properties of expanded austenite and its characters resulting from low temperature hybrid nitriding-carburizing treatment on AISI 316L

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Abstract

This paper investigates the surface properties of expanded austenite layers resulting from low temperature thermochemical treatment using conventional tube furnace. The characterization methods for this resulting layers were measured by using Microhardness, Nanoindentation, SEM, XRD and USPM. Based on indentation measurements, these hybrid-treated samples have shown the improvement of hardness and elastic modulus compared to the untreated sample. The hybrid treatment also produces a thicker layer and unique nitrogen and carbon profile across the layer with shows the absence of nitride and carbide at temperature 450°. The treatment offering improved surface hardness of ductile austenitic stainless steel on successful thermochemical treating.

Keywords : Hybrid Nitriding-carburizing, Characterization, Expanded austenite, Hardness improvement.

Introduction

It is well known that resistance to corrosion is a crucial factor that has to take into account in selecting a material especially for highly corrosive environments. In oil and gas industry, corrosion of equipment, pipeline and pipes become the most common issues facing by offshore as well as onshore infrastructure. This problem leads to numerous consequences such as maintenance and operating cost, plant shutdowns, contamination of products, loss of product and effect of safety and reliability.

Austenitic stainless steels are the most widely used in the chemical and petrochemical industry, cryogenic vessels, heat exchangers, machinery for paper, pulp, textile, pharmaceutical, implant biomaterial, bipolar plate for fuel cell, electronic consuming devices like Iphohe4 case and other domestic equipment. This type of steels has excellent corrosion resistance and forming characteristics. However, due to its inherent

austenitic structure this material has relatively low in hardness as well as poor wear resistance. This also related to the basic problem of stainless steel is that it suffers from extensive wear which prohibits wider industrial applications [1-2].

Austenitic stainless steel (ASS) especially AISI 316L is widely used for surface piping, vessel cladding and clad line pipe, are must be taken to ensure the application is completely deaerated. In the presence of oxygen, 316L will pit, for example, if exposed to even cold seawater. In conditions containing H_2S, the

performance of AISI 316L is very sensitive to the presence of chloride ions. In chloride-free environments (<50ppm chloride), 316L has given reliable service in sour gas handling facilities, but pitting is readily initiated when chloride ions are present.
Nitriding and Carburizing are surface engineering with thermochemical treatments which applied to the surface of machine tools, parts and other metallic objects to improve their surface hardness and enhance the mechanical properties. Many investigations have been performed to improve surface hardness of austenitic stainless steel and therefore enlarging their possibility of wider application, but led significant loss of its corrosion resistance. Beside that, traditional Surface hardening is a bad practice to increase surface hardness due to Sensitivity effect [3-4].

Earlier works in surface engineering for increasing the surface hardness and the wear resistance of the austenitic stainless steels, such as conventional nitriding and nitrocarburizing, have led to the deterioration in the corrosion resistance arising from depletion of chromium in the hardened layer. So far, low temperature nitriding and carburizing of austenitic stainless steels have been successfully conducted by innovative techniques, including plasma nitriding which is conducted by K. Ichii, et. al [5] and D.B Lewis, et. al [6], for the use of ion beam nitriding and ion implantation respectively [7, 8]. Hardening is due to the incorporation of nitrogen and carbon respectively in the austenite lattice, forming a structure which is supersaturated with nitrogen and carbon respectively at low temperatre to form an expanded austenite (γx) (X=N,C) layer without formation precipitation of nitride/carbide.

However, only few investigations have been made commercially by using conventional processes such as gaseous and fluidized bed processes. Previous work has shown that low temperature nitriding of austenitic stainless steel is possible to be conducted by using a fluidized bed furnace [9].

For this investigation, the author has performed low temperature hybrid thermochemical treatment of austenitic stainless steel at different temperatures. In this process, nitrogen and carbon were diffused into the surface of a solid ferrous alloy by treated the material at three different temperatures (400^{o}C, 450^{o}C and 500^{o}C) with constant treatment time and pressure in contact with a nitrogenous gas.

Methodology

The research work investigated a systematic development of a surface harden layer on AISI 316L and its microstructure, surface topography, elemental including structural phase analysis and its near-surface mechanical properties. All the sample materials were prepared in the similar method, standard metallographical examination i.e. sectioning, grinding, polishing, cleaning, drying, followed by low temperature thermochemical treatments at the appropriate parameters**.** The hybrid process involved treating the sample in an atmosphere containing both NH_3 (for nitriding) and CH_4 (for carburizing) for a total duration of 8 h. After treatments, the surface morphology of the treated specimens were characterized using Field Emission Scanning Electron Microscope (FESEM), X-ray Diffractometer will be employed to identify the produced phases from various process conditions 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.

Nano-mechanical testing and microhardness profile of Vickers indentation was made across each resultant layers. The test was also performed to reveal the surface mechanical properties. Thus, the data from the above characterization techniques, the surface characteristics of treated specimens from various treatment conditions will be determined.

Results and Discussion

The depth profiles for thermochemically hardened stainless steel typically show a trend of increasing depth with higher temperatures and longer process variations. Microhardness test was performed to determine the characteristics of each specimen under the influence of the nitriding temperatures. Microhardness Vickers tests were conducted to the samples start from the surface and along on the cross section of the nitrided samples (400°C, 450°C and 500°C). The load used was 10gf and the dwell time was 15s.

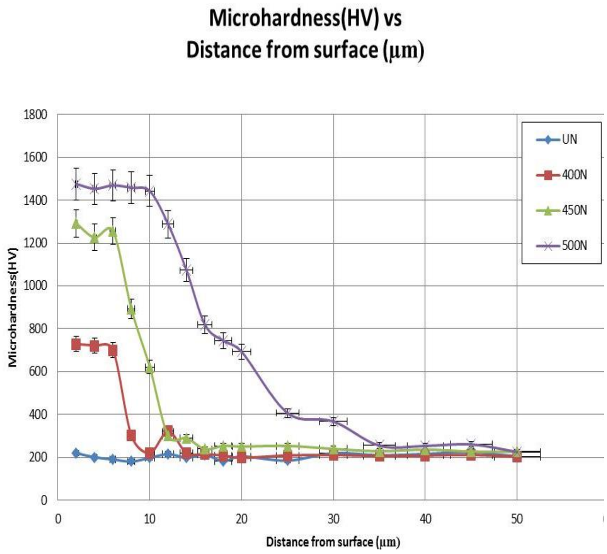
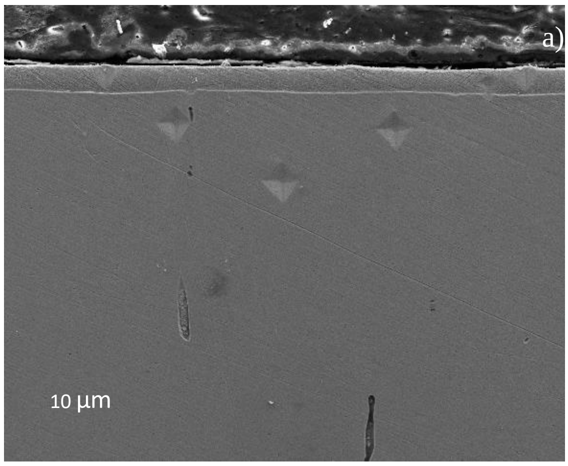


Figure 1. Microhardness depth profile

The microhardness profiles of the samples untreated and treated at 400 °C, 450 °C, and 500 °C are shown in Figure 1. All the treated samples were found to be harder that the untreated sample. A 6 μm hardened layer with hardness around 720 HV was achieved on the sample nitrided at 400 °C. As treated temperature increased, higher hardness values and thicker hardened layers were obtained. For the sample treated at 500 °C, the microhardness value (1500Hv) remained higher than the untreated sample throughout the thicker hardened layer (about 30 μm). This profile shows that the hybrid nitriding-carburizing temperature influences the hardness of the nitrided samples. The effectiveness of the treatment to increase the hardness of the steel was verified. This high surface microhardness value can be related to the presence of hard chromium nitride/carbide precipitates in the nitrided layer [10].

On the other hand, the hardness of the treated samples maintained at a high level and at a certain distance, it decreased gradually from modified layer to the metal core.

Hardness and Elastic modulus values were determined by nanoindentation test using a Nano Test 600 apparatus from Micromaterials instruments (Wrexham, UK) equipped with Berkovich indenter. The hardness and elastic modulus values have been extracted from the elastic unloading curves according to the equivalent indenter method.



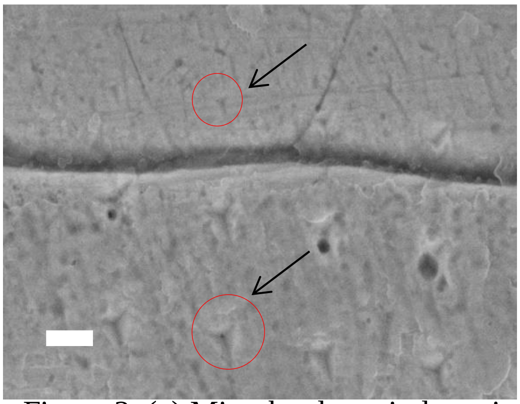


Figure 2. (a) Microhardness indentation and (b) Nanoindentation with Berkovich indenter.

The hardness and elastic modulus values have been extracted from the elastic unloading curves according to the equivalent indenter method. Performed in the same conditions, regular arrays of 6 x 6 indentations covering a 24.32 x 21.89 μm^2 area were realized in order to probe about 3 areas which consist of substrate, interface and harden layer respectively. Observation of Image: Indentation size on substate is bigger than indentation on hardened layer indicating that the hardened layer is higher than substrate hardness.

X-ray diffraction (XRD) was used to identify structural phases that formed during and at the end of the treatment process. Samples were analyzed with grains in random orientations to insure that all crystallographic directions are "sampled" by the beam. Analyses were carried out on a standard Philips diffractometer operating at 30 mA at 40 kV using CuKa radiation.

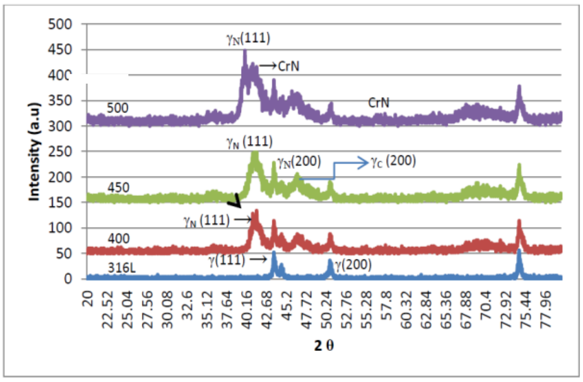


Figure 3. Comparison of XRD patterns of hybrid treated specimens from 400 ^{o}C to 500 ^{o}C as compared to untreated 316L

XRD analysis confirmed the phase composition and layer structure as shown in Figure 3 for various resultant layers. In all the resultant layers, no nitride and carbide precipitates were detectable by XRD except for treated sample at 500^{o}C. This is in close agreement with previous results [11-12] on individual plasma nitriding and carburizing which conclude that the formation of nitride and carbide precipitates requires a higher temperature, and the temperature of 450°C does not favour the formation of these precipitates.

The surface topography of hybrid treated specimens from top view and cross sectional of expanded austenite were also investigated by a scanning probe microscope.USPM observation at higher resolution of all treated surfaces shows a higher surface roughness after treatments. Meanwhile, Figure. 4 (a) and (b) shows the 3D surface topography of the treated specimens as determined by USPM.

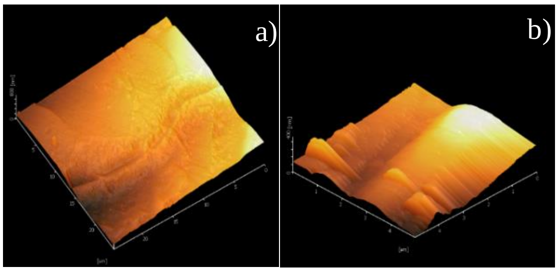


Figure 4. 3D Surface topography resulting from Universal Scanning Probe Microscopy (USPM): (a) hybrid 450^{o}C (b) 500^{o}C samples

As confirmed by cross section FESEM micrograph, it is observed that the granular structure growth of the two sublayers as a result of nitrogen and carbon diffusion and showing the results of deposition process. The densely packed columnar structure of the two expanded austenite layers in Figure 4 correlates well with the globular surface structure observed in Figure 3. The presence of CrN in treated samples 316L at 500^{o}C is confirmed by XRD as in Figure 3.

Conclusion

The thermochemical treatments of AISI 316L stainless steel in a horizontal tube process at 400 and 450^{o}C have been demonstrate the possibilities to produce hard layer of an expanded austenite phase without precipitation of chromium carbide/nitride as confirmed by FESEM images and XRD. This layer would contribute to the observed higher hardness and better corrosion resistance as compared to untreated sample. USPM observation of all treated surfaces shows a higher surface roughness after treatments. Microhardness and Nanoindentation tests reveal a higher elastic modulus for all treated samples compared to the untreated.

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