



SURFACE PROPERTIES OF NITRIDED LAYER ON AISI 316L AUSTENITIC STAINLESS STEEL PRODUCED BY VARIED TIME LOW-TEMPERATURE THERMOCHEMICAL TREATMENTS

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ABSTRACT

Recently, there are several attempts to improve the surface properties of AISI 316L by the mean of thermochemical treatments. The expected result is a layer on the surface termed expanded austenite which is supersaturated with nitrogen and carbon and this structure is responsible for the higher hardness and better wear properties without impairing its corrosion resistance. In this work, AISI 316L stainless steel was gas-nitrided/carburized simultaneously at 450 °C in quartz tube furnace for 8, 24 hours of treatment time. The morphological changes, structural phase, and elemental profile were observed by an optical microscope (OM), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX). All the characterization methods confirmed the formation of nitrides and carbides on the surface layer of AISI 316L forming the expanded austenite layer (S phase), which leads to enhancement of surface properties of the nitrided samples.

Keywords: nitriding, carburizing, low temperature, AISI 316L, austenitic stainless steel.

1. INTRODUCTION

AISI 316L became the selected material in the engineering applications because of its great ductility, weldability, and better erosion-corrosion resistance. Because of the inherited austenitic structure, this material is suffering from low hardness and wear resistance, which bring nearly poor tribological properties [1][2][3][4]. Low hardness on the AISI 316L surface could lead to fast failure of operational parts and instruments. Along these lines, one of the ways to enhance surface hardness and wear resistance of AISI 316L is by thermochemical treatment [5]. Sensitization is a typical issue in austenitic steel where precipitation of chromium carbides (Cr₂₃C₆) happens at the grain boundaries at raised temperatures, normally between 450°C to 850°C; diffusional reaction in forming chromium nitride/carbide leads to the depletion of Cr in the austenitic solid solution and consequently unable to produce Cr₂O₃ passive layer to make stainless feature [6]. Therefore, it lowers the corrosion resistance property of the stainless steel [7].

A low-temperature nitriding treatment can eliminate the development of chromium nitrides but at the same time lower the reinforcement effects made by CrN precipitates [8]. The two significant low-temperature thermochemical forms produced for austenitic stainless steels exist are nitriding and carburizing [9]. The previous is regularly done at temperatures lower than 450 °C and the later beneath 500 °C [10]. The motivation behind utilizing low temperatures gas nitriding treatment is to smother the development of chromium nitrides and carbides in the alloyed layers, such that chromium is retained in solid solution for corrosion protection [11] [12]. Hardening of the nitrided layer and the carburized layer is because of the joining of nitrogen and carbon separately in the austenite cross section, framing a structure called extended austenite (S-phase), which is

supersaturated with nitrogen and carbon discretely [13] [14].

2. EXPERIMENTAL SET UP

The materials acquired from TSA manufacturing industries, with the composition of 0.02% C, 0.42% Si, 1.7% Mn, 0.03% P, 0.002% S, 17.2% Cr, 2.07% Mo, 11.55% Ni, 0.34% Co, and balance Fe. AISI 316L was cut to a measurement of 15X10X5mm. Before the nitriding treatment was done, the samples were ground and polished, grinding sequence follows a series of descending grain size from 120, 320, 600, 800 and 1200grit. After that, the sample was polished using 1,0.5,0.05 μm diamond pastes to create a scratch free surface with a mirror-like finish. To reveal the microstructure of AISI 316L the specimen was etched by Marble's Reagent (10g of CuSO₄, 50 ml HCl and 50 ml H₂O). Nitriding treatment was done in a quartz tube. Before start nitriding process, the specimens were submerged in Hydrochloric Acid HCl(2M) for 15 minutes to remove the oxide layer that can affect the nitrogen dispersion into the sample's surface. For the nitriding treatment, the nitriding temperature treatment is set to be 450 °C from the room temperature with the heating rate of 10 °C/min. The procedure was kept up for 480 minutes (8 hours) and 1440 minutes (24 hours) with an extra time 10 minutes for both samples. The flow rate for every gas is set up in view of the flow meter reading on the control board; Nitrogen gas (150ml/min), Ammonia Gas (40ml/min) and Methane Gas (10ml/min).

Characterization started with Optical microscope to reveal the microstructure, and then Scanning Electron Microscope (SEM), model Phenom ProX desktop for magnification up to 3000X with Energy-Dispersive X-ray (EDX) with line scan mode.



3. RESULTS AND ANALYSIS:

a) Microstructure and morphology

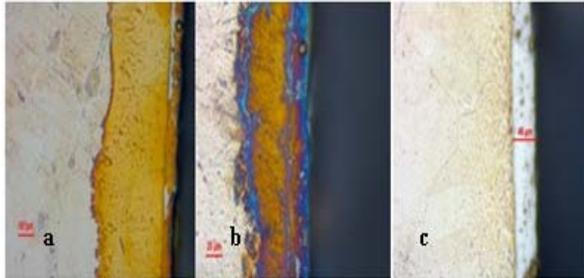


Figure-1. Microstructure of the cross-section of treated AISI 316L by optical microscope (OM).

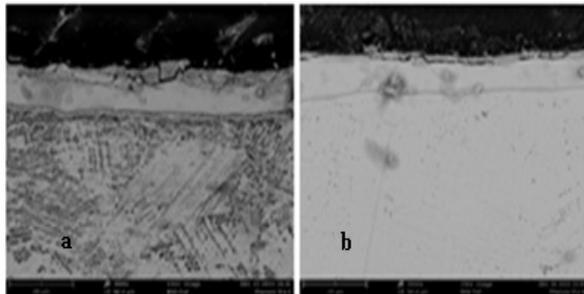


Figure-2. Scanning Electron Microscope (SEM) image of Nitrided layer morphology of sample at a)8 hours and b)24 hours for 3k X magnification.

Figure-1 shows the microstructure on the cross-section of treated 316L at 450°C by an optical microscope. Three distinct regions, a 'bright' or 'white' layer having nitrides and carbides (S-phase), a transition or interface layer below the nitrided layer and a substrate or base material which is not affected by diffusion of nitrogen/carbon atoms can be observed. It is believed that the nitrided layer partially consists of metallic nitrides/carbides (M_xN_y) depending on steel constitution elements and their percentage.

From Figure-1(a) the nitrided layers were measured to be about 28 μm for the sample treated for 8 hours, however, the nitrided/carburized layer was not uniform as shown in Figure-1(b). This might be due to the characteristic of gas nitriding technique. Theoretically, during the ammonia purged into the furnace, the gas that in contact with tempered steel will dissociate into nitrogen and diffuse into the surface [15][16]. Besides, irregular diffusion also can be occurred due to the gas that accumulating near to the surface instead of accelerating directly onto the surface for diffusion. As seen in Figure-1(c), sample nitrided for 24 hours formed the higher thickness of the nitrided layer of $\sim 40\mu\text{m}$ and the layer formed were more uniform and consistent. The nitrided layer thickness increments from 28 μm to 40 μm

as the treatment time is prolonged showing that the nitrogen flux into the metal center is bigger because of the bigger dissemination rate. At longer treatment duration. It is expected that the hardness on the top surface of the nitrided layer is higher than the hardness on the interface and substrate. This impact prompts improvement of surface properties of the nitrided AISI 316L [17].

b) Elemental analysis of the Nitrided layer (S-Phase) on the treated AISI 316L stainless steel

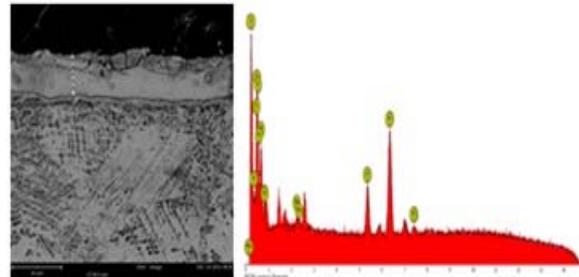


Figure-3. Elemental profile of the treated material surface for 8 hours' line scan mode.

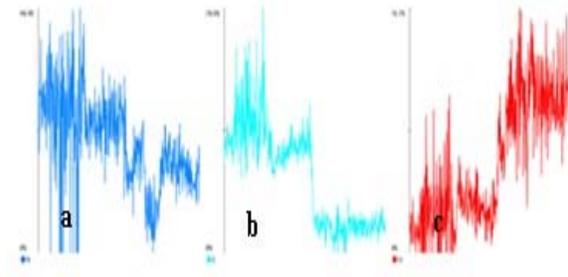


Figure-4. Elemental profile of a) carbon, b) nitrogen and c) chromium of the treated material surface for 8 hours.

The elemental profile of expanded layer was measured with EDS line scan mode. Figure-3, 5 show the typical elemental profiles produced in AISI 316L as a function of distance for 8 hours and 24 hours respectively.

Figure-3 and Figure-5 demonstrate a nearby expand nitrogen peak was identified at the surface, in this way demonstrating the push in effect essentially of broke down carbon by nitrogen amid the half-breed process for both samples [18]. The chromium profile additionally explored on both samples.

Figure-4(c) and Figure-6(c) demonstrates that higher Chromium levels on 24 hours treated samples than 8 hours treated specimen at treated layer which shows that Chromium was more responsive in the longer treatment time. From Figure-4(b), the basic examination of the 8-hour treatment example gave more carbon past the treated layer, however, some carbon was likewise seen at the surface. It can be seen from Figure-3 that the 8-hour treatment sample layer contains a high measure of nitrogen and nitrogen concentration is steadily decreasing



from surface to the center. Because of a low dispersion rate on account of tests at low temperature, nitrogen infiltration profundity on 8-hour sample is not as much as that 24-hour samples. In any case, some carbon stays in the sub-surface layer.

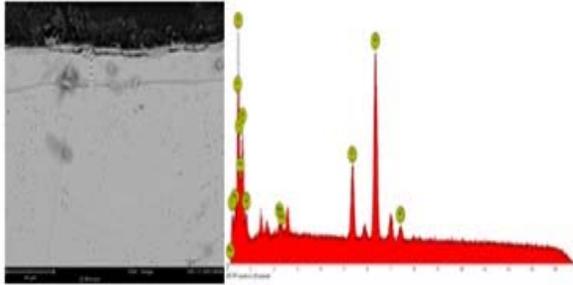


Figure-5. Elemental profile of the treated material for 24 hours' line scan mode.

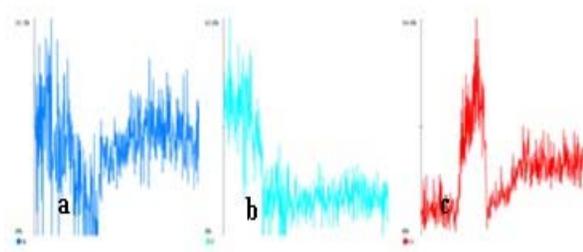


Figure-6. Elemental profile of a) carbon, b) nitrogen and c) chromium of the treated material for 24 hours.

Since both carbon and nitrogen involve the same sort of interstitial locales in the FCC lattice, the entry of the slower diffusing nitrogen particles would constrain the carbon molecules (the previous comer) to make accessible the interstitial destinations. The main simple way for carbon is to diffuse further into the substrate, along these lines shaping a push as a result of carbon by nitrogen and a different carbon extended austenite layer. [19] [20]

4. CONCLUSIONS

The thermochemical treatments of Nitriding/carburizing of AISI 316L stainless steel at 450°C for 8 and 24 hours demonstrate the possibility to produce a layer of an expanded austenite phase without precipitation of chromium carbide/nitride. The layers produced are not uniform in thickness. The layer thicknesses for 8 and 24 hours are 28 μm and 40 μm respectively for the same conditions, but the layer produced at 24 hours was more uniform than the 8 hours, which could be attributed to the high rate of nitrogen diffusion per unit area at longer treatment times. It is concluded that the nitriding of 316L stainless steel increases the nitrided layer thickness and uniformity. EDS line scan in the cross-section for treated austenitic 316L on both specimens reveal a decrease of Fe and a corresponding slight increase in N and C at the compound layer, which suggests the existence of iron

nitrides/carbides in the compound layer. Such distributions of nitrogen and carbon in the surface layer are likely to produce some beneficial influences upon the properties of surface-treated layers on AISI 316L. However, the 24-hour treatment gives higher Cr profile than the 8-hour treatment at the treated layer which indicates that Chromium was more reactive in the longer treatment time of 24 hours as compared with the 8 hours' treatment.

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