Materials Science Forum Vols. 408-412 (2002) pp 1347-1352
© (2002) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/MSF.408-412.1347

Texture Evolution during High Temperature Gas Nitriding of Duplex Stainless Steels

A.P. Tschiptschin[1], C.M. Garzón[1] and N.B. De Lima[2]

[1] Metallurgical and Materials Engineering Department, University of São Paulo,
Av. Prof. Mello Moraes 2463, BR-05508-900 São Paulo SP, Brazil

[2] Nuclear and Energy Research Institute, IPEN , BR-05508-900 São Paulo, Brazil

Keywords: Duplex Stainless Steel, High Nitrogen Steel, High Temperature Gas Nitriding, Microtexture, Texture

Abstract. High temperature gas nitriding of stainless steels allows obtaining austenitic or martensitic cases with good wear and corrosion resistance. The texture evolution during high temperature nitriding of a ferritic-austenitic duplex stainless steel is studied. The microtextures were determined by Electron Back Scattering Diffraction (EBSD) analysis. UNS S31803 duplex stainless steel was gas nitrided under a 0.065 MPa N_2 partial pressure at 1423 K for times between 2.7 and 28.8 ks. At the initial stages of the nitriding treatment austenitic cases were formed, with austenite sideplates growing towards the ferritic-austenitic core, holding a Kurdjumov-Sachs (K-S) type orientation relationship with prior existing ferrite grains. The corresponding austenite texture at the steel surface is {011}<411>. For longer nitriding (28.8 ks) times the microstructure coarsens, ferrite is consumed and austenite grain growth occurs, resulting in grains 150-200 μm in diameter and a {011}<211> texture, typical of austenite in hot rolled or solution treated duplex stainless steels. The texture intensities decrease with increasing nitriding time. These results are discussed taking in account nitrogen gradients and nitrogen supersaturation built up during the nitriding treatment.

Introduction

Through High Temperature Gas Nitriding (HTGN) of stainless steels it is possible to obtain 1-2 mm cases with excellent corrosion resistance and tribological properties [1,2]. This treatment, quite different from conventional nitriding at 773 to 853 K, which entails the precipitation of a nitride layer, leads to the dissolution of nitrogen in austenite [2]. When fine-grained martensitic-ferritic dual phase stainless steels are high temperature nitrided, hard fully martensitic cases can be obtained. When austenitic-ferritic duplex stainless steels are nitrided, fully austenitic cases can be obtained. The microstructure after nitriding is basically dependent upon the steel's chemical composition and the treatment parameters: temperature, time and N_2 partial pressure [3]. In general, the HTGN treatment is made between 1323 and 1473 K under 0.05 to 0.3 MPa N_2 partial pressure for times varying between 3.6 and 86.4 ks, resulting in case depths up to 3 mm.

It is a well-accepted fact that the corrosion resistance and the tribological and mechanical properties of metals depend on the grain structure and orientation [4]. The texture of duplex stainless steels has been studied for several researchers along the two last decades [5-8]. Hutchinson and co-workers [5] studied the recrystallization texture of a duplex stainless steels DIN 1.4462, and found the texture {100}<011> for ferrite grains and {110}<112> for austenite grains. Ul-Haq and co-workers [6] studied the hot rolling texture in a similar steel, detecting two fiber textures for the ferrite grains, <110> // DN and <110> + 30° // DN. For austenitic grains, they found a texture between brass and copper type textures, {112}<111>+5° or {4 4 11}<11 11 8>. De Lima and co-workers [7] found that the recrystallised texture of the same type of stainless steels is {011}<110> and {112}<110> for ferrite grains and {110}<112> and {011}<100> for austenite grains. Padilha and co-workers [8] determined the recrystallised texture in a similar steel, reporting the components $(100)[0\bar{1}1]$, $(110)[\bar{1}11]$ and $(110)[111]$ for ferrite grains, and the components $(110)[\bar{1}11]$ and $(110)[112]$ for austenite grains.

It is worth noting that there is a good agreement between austenite grain textures reported by different authors. The same cannot be stated for the several reported ferrite grains texture, probably due to rolling and heat treatment differences.

Padilha and co-workers [8] studying a 0.2 MPa N_2 partial pressure, 1473 K nitrided duplex stainless steels during 18.0 ks, observed strengthening of the $(110)[1\bar{1}1]$ and $(110)[\bar{1}11]$ texture components and weakening of the $(001)[110]$ component.

The aim of the present work is to study the effect of the nitriding time on the surface microtexture of precipitate free austenitic cases obtained by HTGN of a UNS S31803 duplex stainless steel.

Experimental

Rectangular specimens (20 mm x 20 mm x 9 mm) of UNS S31803 duplex stainless steel were high temperature gas nitrided and direct quenched in water. The chemical composition of the studied steel is shown in Table 1. The specimens were heated up to 1423 K under 0.13 Pa in a vacuum chamber and then exposed to a high purity Ar + N_2 atmosphere under 0.065MPa N_2 partial pressure for 2.7, 10.8 and 28.8 ks.

The microstructure of the samples was examined by optical microscopy, and the chemical composition was determined by optical spectrometry and wavelength dispersive spectroscopy. The microtexture at the surface of the samples was determined using a TSL-EBSD instrument interfaced to a Philips XL30TM scanning electron microscope.

Table 1 – Chemical composition of the UNS S31803 duplex stainless steel (wt-%)

%Cr	%Ni	%Mn	%Mo	%C	%Cu	N	S	%Si
22.5	5.4	1.9	3.0	0.019	0.14	0.16	<0.001	--

Results

Figure 1 shows the micrographs of the UNS S31803 steel surface as received (hot rolled), solution treated at 1423 K, and nitrided for 2.7 ks at 1423 K. The microstructures suggest that, during the earliest stages of the nitriding treatment, precipitation of austenite at the surface occurs mainly as needle shaped grains, like the precipitation of austenite from delta ferrite in duplex stainless steels during cooling after solidification.

Figure 2 shows the micrographs of the transversal section of the studied steel, nitrided for 2.7, 10.8 and 28.8 ks. All nitriding conditions led to precipitate free austenitic cases. From the surface to the core of the specimens, three regions with different microstructures are observed: an austenitic case, a transition region and a ferritic-austenitic duplex core. The nitrogen content at the surface of the sample nitrided for 28.8 ks is approximately 1.2 wt-%. The transition region is formed by an austenitic matrix with non-transformed isolated ferrite grains. The non-nitrided core is formed by a 60/40 ferritic-austenitic duplex structure with grain sizes of 25 – 30 μm (ferrite) and 20 – 25 μm (austenite).

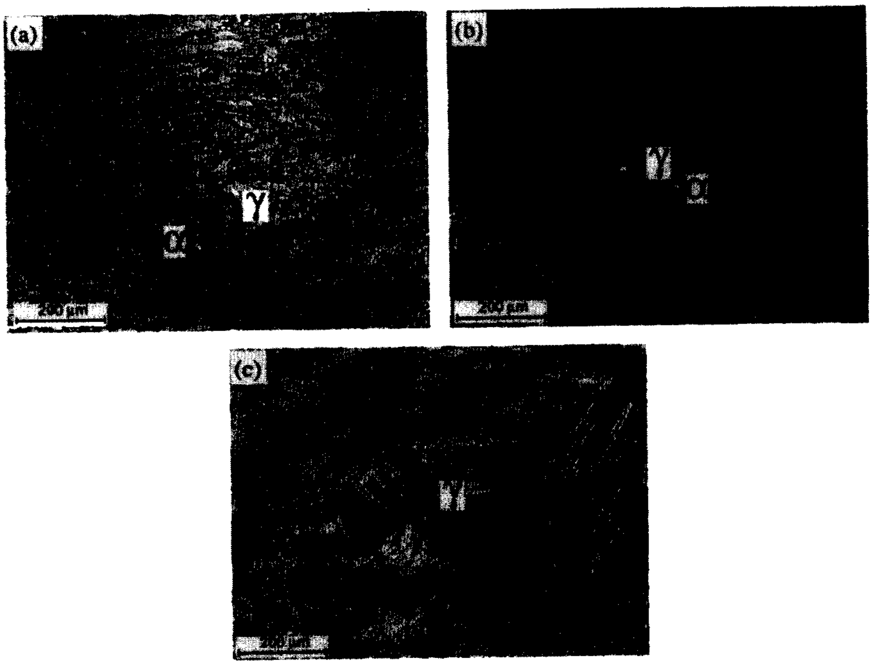


Fig 1 –Micrographs taken on top of the surface of UNS S31803 steel (a) hot rolled, (b) solution treated at 1423 K and (c) nitrided during 2.7 ks at 1423 K. Austenite (γ) and ferrite (α). O.M, etching: water regia.

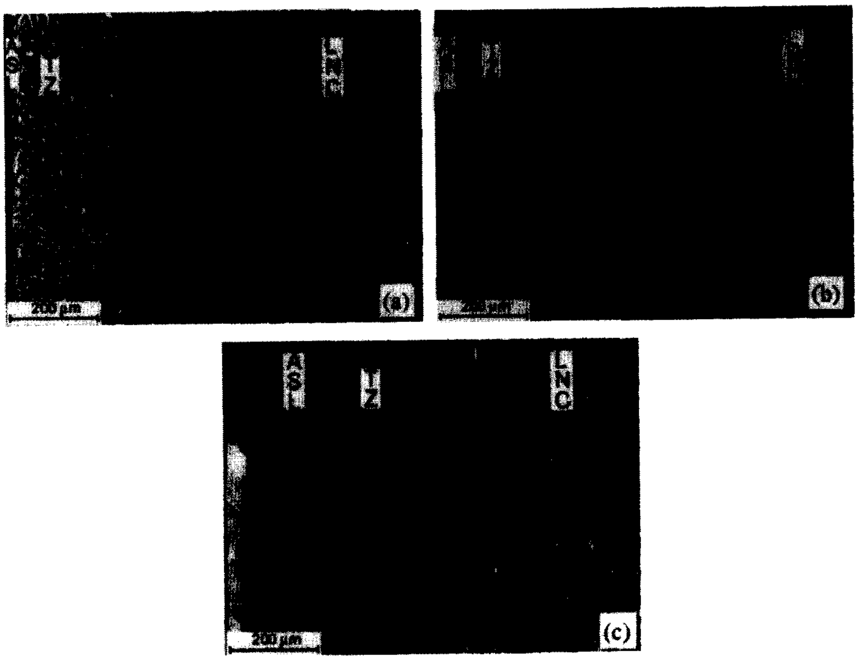
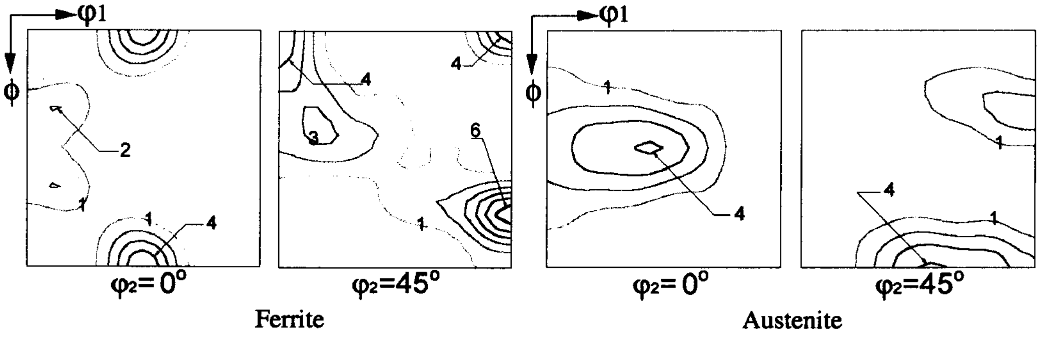


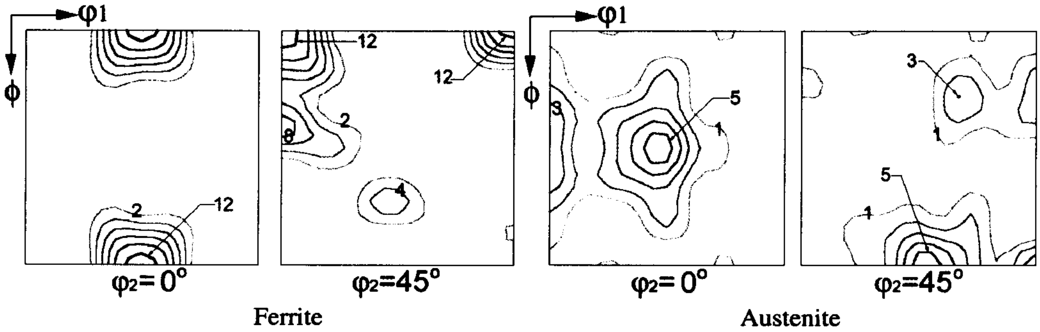
Fig 2 –Micrographs of the transverse section of UNS S31803 steel nitrided at 1423 K under 0.065 Mpa N_2 partial pressure for: (a) 2.7 ks, (b) 10.8 ks and (c) 28.8 ks. Micrographs show the high nitrogen austenitic surface layer (ASL), the transition zone (TZ) and the low nitrogen core (LNC). O.M, etching: water regia.

The microstructures observed in Fig. 2 suggest that, at the initial stages of the nitriding treatment (2.7 ks), the growth of austenitic grains into the duplex structure is similar to the growth of columnar grains usually observed in ingots. Only those grains favorably oriented can grow into the ferritic-austenitic core, due to high chemical gradients, resulting in an acicular microstructure. For longer nitriding times (28.8 ks), the chemical gradient at the interface between the austenitic case and the transition region is very small [3], not sufficient to allow the growth of the acicular microstructures. Hence, equiaxed grains are formed and an austenite/(austenite+ferrite) planar interface develops. For intermediate times (10.8 ks), coarsening of acicular ferrite grains occurs, although the acicular morphology still remains.

Figures 3 and 4 show the ODF sections at $\varphi_2 = 0^\circ$ and $\varphi_2 = 45^\circ$ (Bunge notation), determined trough EBSD at the surface of the steel as received (hot rolled), solution treated, and nitrided for 2.7, 10.8 and 28.8 ks. Table 2 shows the microtexture components for these different treatment conditions. It is observed that the nitriding treatment changes the microtexture depending on the nitriding time. The orientation relationship, at the surface, between austenite obtained after the 2.7 ks nitriding treatment and ferrite of the solution treated samples was calculated to be $\{111\}\gamma + 15^\circ // \{110\}\alpha$, close to the Kurdjumov-Sachs relationship leading to semicoherent, low energy and low mobility interfaces which require high chemical gradients to move. The microtexture intensities decrease with increasing nitriding time. The microtexture of the steel nitrided for 28.8 ks {011}<211> is the same as that observed for austenite grains in solution treated duplex stainless steels reported in literature.

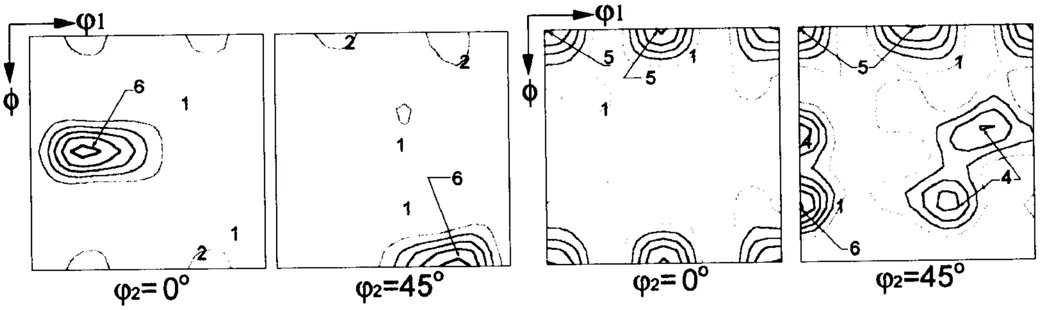


(a) Hot rolled



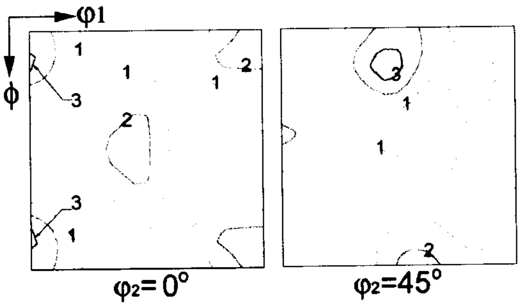
(b) Solution treated to 1423 K

Fig 3 – ODF's sections at φ2 = 0° and φ2 = 45°, for ferrite and austenite grains of UNS S31803 steel as received (hot rolled) and solution treated. Euler angles (Bunge), φ1 and ϕ vary between 0° to 90°.



(a) Nitrided for 2.7 ks

(b) Nitrided for 10.8 ks



(c) Nitrided for 28.8 ks

Fig 4 – ODF's sections at $\varphi2 = 0^\circ$ and $\varphi2 = 45^\circ$, for austenite grains of the UNS S31803 steel nitrided at 1423 K under 0.065 MPa N_2 partial pressure for 2.7, 10.8 and 28.8 ks. Euler angles (Bunge), $\varphi1$ and ϕ vary between 0° to 90°.

Table 2 – Microtexture components for UNS S31803 steel treated at different conditions.

Treatment condition		**Strongest textures** (times random intensity)	**Weaker textures** (times random intensity)
Hot rolled	α	{221}<114>, 6.7	{100}<110>, 4.7
	γ		{011}<211>, 4.2
Solution treated at 1423 K	α	{001}<110>, 13.0 {112}<110>, 8.9	
	γ	{110}<112>, 5.7	
Nitrided at 1423 K, under 0.065 MPa for 2.7 ks		{011}<411>, 6.5	
Nitrided at 1423 K, under 0.065 MPa for 10.8 ks		{221}<110>, 6.2	{001}<110>, 5.1
Nitrided at 1423 K, under 0.065 MPa for 28.8 ks			{011}<211>, 3.6 {001}<100>, 2.2

Discussion

The results showed that during the first stages of the HTGN treatment of duplex stainless steels austenite side plates precipitate from the prior ferrite grains with an orientation relationship, developing a strong texture (though smaller than that existing in the solution treated specimens). The mechanism of austenite precipitation at the surface is probably the selective growth of pre-existing austenite grains by migration of low energy austenite/ferrite interfaces. The high driving force necessary for this migration is supplied by the high nitrogen gradient existing at the surface for the lowest nitriding times.

The 15° observed deviation from the KS relationship is probably due to:

1) The texture of the austenite measured for 2.7 ks nitriding time, may not be the one developed just after the precipitation of the first austenite grains.

2) Differences between the texture of ferrite grains in the solution treated specimen and ferrite grains in the nitrided specimen may exist.

3) Differences between microtextures and macrotextures due to orientation heterogeneities inside the population of grains in the as received plate.

Conclusions

High temperature nitriding treatment of duplex stainless steels causes strong texture, which changes and weakens with the increase in nitriding time. As a consequence of the high nitrogen gradients at the austenite / (austenite+ferrite) interface, the texture developed for short nitriding times is determined by the growth of austenite sideplates, with an orientation relationship (with the pre-existing ferrite grains) close to Kurdjumov-Sachs. For the higher nitriding times the microstructure coarsens, adopting the same texture reported for austenite grains in solution treated or hot rolled duplex stainless steels.

Acknowledgements

The authors would like acknowledge the support of the Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), Brazil.

References

[1] V.G. Gavriljuk and H. Berns: *High Nitrogen Steels* (Springer-Verlag, Berlin 1999).

[2] H. Berns, R.L. Juse, J.W. Bouwman and B. Edenhofer: Heat Treatment of Metals (2000), p. 39

[3] C.M. Garzón, A. Toro and A.P. Tschiptschin: 6^{ta} Int. Conf. On High Nitrogen Steels, HNS India 2002.

[4] E.M. Lehockey, D. Limoges, G. Palumbo, J. Sklarchuk, K. Tomantschger and A. Vincze: J. of Power Sources, Vol. 78 (1999), p. 79

[5] B. Hutchinson, K. Ushida and G. Runnsyo: Mat. Sci. and Tech. Vol. 1 (1985), p. 39

[6] A. Ul-Haq, H. Weiland and H.J. Bunge: Mat. Sci. and Tech. Vol. 10 (1994), p. 289

[7] N.B. De Lima, L. Mitteregger, W. Reick and A.F. Padilha: Proc. Int. Cong. on Metallurgical technology and materials, Brazilian Metals and Materials Society (ABM), Brazil, Vol. 8 (1994), p. 405

[8] A.F. Padilha, V. Randle and I. Machado: Mat. Sci. and Tech. Vol. 15 (1999), p. 1015

Textures of Materials - ICOTOM 13
doi:10.4028/www.scientific.net/MSF.408-412

Texture Evolution during High Temperature Gas Nitriding of Duplex Stainless Steels
doi:10.4028/www.scientific.net/MSF.408-412.1347