

Analysis on Conventional Nitriding Behaviour of the AISI 316 Stainless Steel

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Abstract

The paper analyses the behaviour of the AISI 316 stainless steel during conventional nitriding in a gaseous environment, a material widely used in industrial and biomedical applications due to its corrosion resistance. The aim of the research is to evaluate the influence of process parameters on microstructural changes and surface properties. In the case of the AISI 316 steel, conventional gas nitriding is strongly limited by the passive layer of chromium oxide, which reduces the diffusion of nitrogen. For this reason, the formation of a continuous nitrided layer is difficult, the resulting layer usually being very thin, uneven or discontinuous. The changes are mainly associated with a slightly nitrogen-enriched superficial area and the local appearance of nitrides, depending on the treatment. Even under these conditions, a moderate increase in surface hardness can be observed depending on the process parameters. The conclusions highlight the limited efficiency of conventional gas nitriding for AISI 316, which require strict optimization of parameters or the use of alternative processes in order to obtain high-performance nitrided layers.

Keywords

AISI 316, conventional nitriding, process parameters

1. Introduction

Austenitic stainless steels are one of the most widely used classes of metallic materials due to their favourable combination of high corrosion resistance, good mechanical properties and excellent machinability. Among them, the AISI 31 stainless steel is widely used in the chemical, food, energy and biomedical industries, due to its molybdenum content, which gives it superior resistance to localized corrosion in aggressive environments [1, 2].

However, one of the major limitations of the AISI 316 steel is its low surface hardness and modest wear resistance, which restrict its use in applications subjected to intense tribological stress. In such conditions, surface modification treatments represent an effective solution to improve functional performance without affecting the volumetric properties of the material [3].

Conventional nitriding is one of the best thermochemical treatments for increasing surface hardness, wear resistance and, under certain conditions, fatigue resistance. The process consists of the diffusion of nitrogen atoms into the surface layer of the material, followed by the formation of nitrogen-rich phases and a diffusion layer with superior mechanical properties [4]. In the case of carbon steels and low-alloyed steels, nitriding leads to the obtaining of hard and stable layers with excellent performance in service.

The application of conventional nitriding to austenitic stainless steels is, however, much more complex. The main obstacle is the passive chromium oxide (Cr_2O_3) film, spontaneously formed on the surface of the material, which prevents the adsorption and diffusion of nitrogen into the substrate. Consequently, without the use of appropriate technological regimes, the nitrided layer can be very thin or even absent [5-7]. Moreover, at high temperatures, nitrogen reacts with chromium in the matrix, favouring the precipitation of chromium nitrides (CrN and Cr_2N), a phenomenon that leads to the depletion of the adjacent areas in chromium and can reduce the corrosion resistance of the material [8, 9].

The analysis of the nitriding behaviour of the AISI 316 stainless steel is of particular interest from both a scientific and industrial point of view. Characterization of nitriding behaviour involves the investigation of the microstructural and structural changes produced in the superficial layer [10].

The objective of this work is to analyse the conventional nitriding behaviour of the AISI 316 stainless steel, by evaluating the influence of technological parameters on the development of the nitrided layer and on the induced microstructural and mechanical changes.

2. Methods

The AISI 316 is a chromium-nickel-molybdenum stainless steel, known for its superior corrosion resistance. The addition of molybdenum (2–2.5%) gives the material increased resistance to pitting and crevice corrosion, being widely used in the chemical, pharmaceutical, food and maritime industries. To analyse the conventional nitriding behaviour of these steels, treatments such as quenching, tempering and nitriding at different temperatures were applied, according to the plan in Table 1.

Table 1. Treatments applied to the AISI316 steel

No.	Quenching			Tempering		Nitriding		
	Temp. [°C]	Time [h]	Cooling	Temp. [°C]	Time [h]	Temp. [°C]	Time [h]	Cooling
1	980	2	Recirculated nitrogen	150	2	-	-	Air
2	980	2	Recirculated nitrogen	150	2	520	20	With nitrogen until 400°C then forced cooling with air cooler
3	980	2	Recirculated nitrogen	510	2	-	-	Air
4	980	2	Recirculated nitrogen	510	2	520	20	With nitrogen until 400°C then forced cooling with air cooler
5	1120	2	Recirculated nitrogen	150	2	-	-	Air
6	1120	2	Recirculated nitrogen	150	2	520	20	With nitrogen until 400°C then forced cooling with air cooler
7	1120	2	Recirculated nitrogen	510	2	-	-	Air
8	1120	2	Recirculated nitrogen	510	2	520	20	With nitrogen until 400°C then forced cooling with air cooler

For a complete characterization of the samples thus treated, their microscopic analysis was performed. Figures 1....9 present the structures of the samples treated according to Table 1.

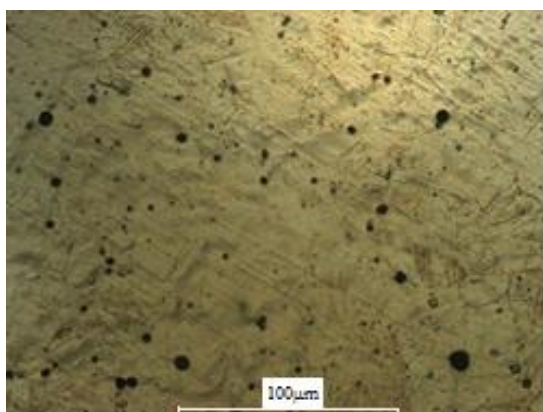


Fig. 1. Sample in its initial state. Aqua regia attack. 50X

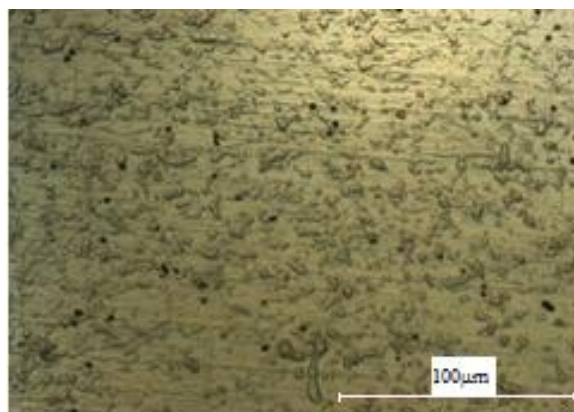


Fig. 2. Sample quenched at 980 °C and low tempering at 150 °C. Aqua Regia attack. 50X

It is observed that after tempering to 150°C the microstructure is similar to that after quenching, but with a tendency of appearance of fine precipitates arranged in rows. By increasing the temperature to 510 °C this tendency is accentuated. On the samples thus treated the hardness of the material was measured, the values being presented in Table 2

Given the fact that nitriding could not be performed on a continuous layer and to a reasonable depth, measuring the hardness in the nitrided layer was almost impossible and we considered that the values obtained were not conclusive, which is why they were not included in the table as stand-alone values on the nitrided layer.

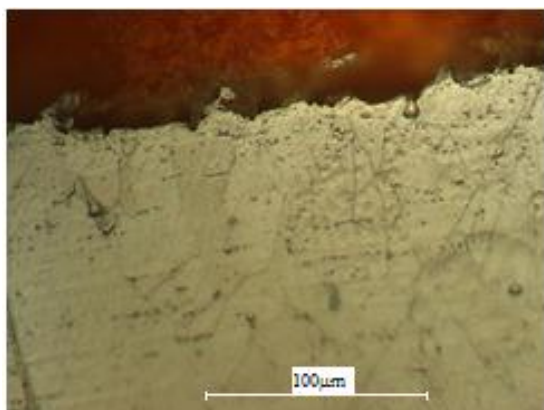


Fig. 3. Sample - layer (quenching at 980 °C, low tempering at 150 °C and nitriding at 520 °C). Aqua Regia Attack. 50X

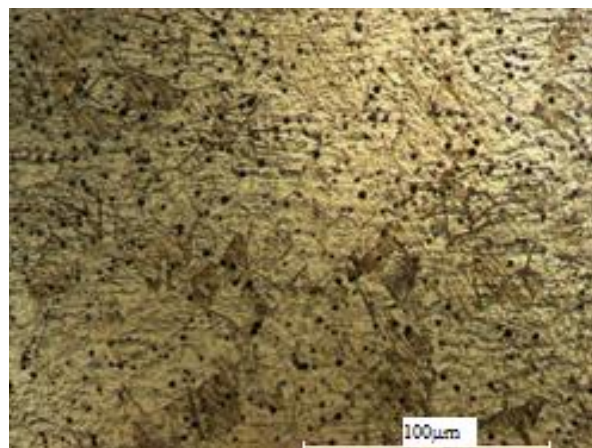


Fig. 4. Sample (quenching at 980 °C, high tempering at 510 °C). Aqua Regia Attack. 50X

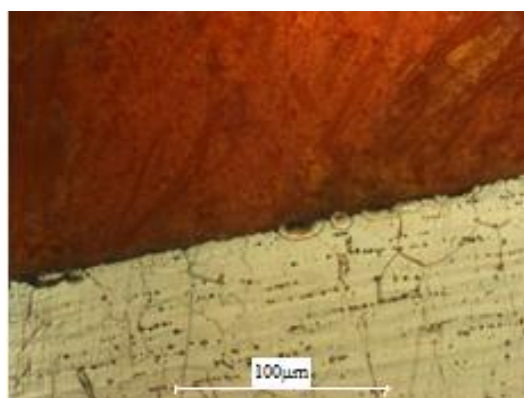


Fig. 5. Sample - layer (quenching at 980 °C, high tempering at 510 °C and nitriding at 520 °C). Aqua Regia attack. 50X

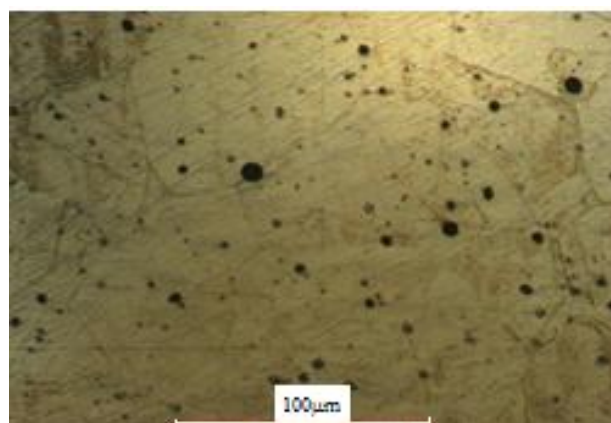


Fig. 6. Sample (quenching at 1120 °C and low tempering at 150 °C). Aqua Regia attack. 50X

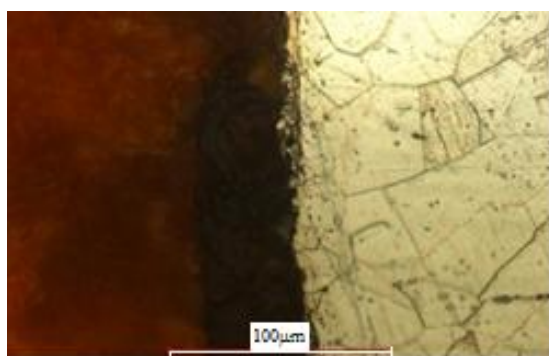


Fig. 7. Sample - layer (quenching at 1120 °C, low tempering at 150 °C and nitriding at 520 °C). Aqua Regia attack. 50X

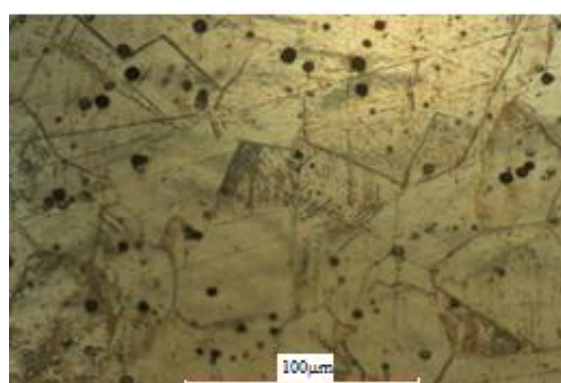


Fig. 8. Sample (quenching at 1120 °C and high tempering at 510 °C). Aqua Regia attack. 50X

3. Discussions

The heat treatments applied to the AISI316 stainless steel (quenching at 980 °C and 1120 °C, followed by low tempering at 150 °C and high tempering at 510 °C, respectively, followed by nitriding at 520 °C), led to slightly higher hardness values than if nitriding was not applied. In the initial state, the material has a hardness of approximately 232HV.

Quenching at both 980 °C and 1020 °C + high tempering at 510 °C in combination or not with nitriding, generated lower hardness values compared to those that were subjected to low tempering at

150 °C. The lower hardness values obtained after high tempering indicate a softer structure due to intense tempering and a decrease in the content of internal stresses.

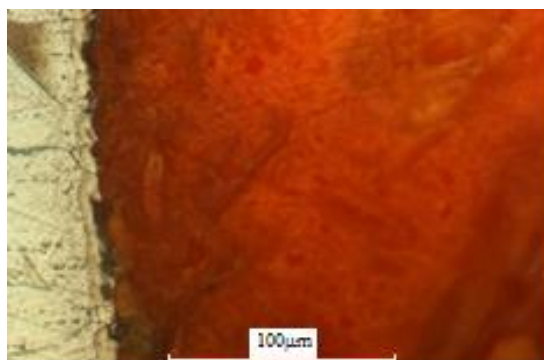


Fig. 9. Sample - layer (quenching at 1120 °C, high tempering at 510 °C and nitriding at 520 °C). Aqua Regia Attack. 50X

Table 2. Obtained hardness values

No.	Vacuum quenching			Vacuum tempering			Nitriding			Hardness [HV1]
	T [°C]	Holding time [h]	Cooling	T [°C]	Time [h]	Cooling	T [°C]	Holding time [h]	Cooling	
1	-	-	-	-	-	-	-	-	-	232
2	980	2	Recirculated nitrogen	150	2	Recirculated nitrogen	-	-	-	272
3	980	2	Recirculated nitrogen	150	2	Recirculated nitrogen	520	20	With nitrogen until 400 °C then forced cooling with air cooler	282
4	980	2	Recirculated nitrogen	510	2	Recirculated nitrogen	-	-	-	245
5	980	2	Recirculated nitrogen	510	2	Recirculated nitrogen	520	20	With nitrogen until 400°C then forced cooling with air cooler	254
6	1120	2	Recirculated nitrogen	150	2	Recirculated nitrogen	-	-	-	312
7	1120	2	Recirculated nitrogen	150	2	Recirculated nitrogen	520	20	With nitrogen until 400°C then forced cooling with air cooler	325
8	1120	2	Recirculated nitrogen	510	2	Recirculated nitrogen	-	-	-	308
9	1120	2	Recirculated nitrogen	510	2	Recirculated nitrogen	520	20	With nitrogen until 400°C then forced cooling with air cooler	320

By analysing the structure under a microscope, a very thin and discontinuous nitride layer was revealed on the surface of the steel. The results obtained confirm that the parameters chosen for the heat and thermochemical treatments and the results obtained are adequate for the AISI316 stainless steel.

High-temperature quenching treatments, followed by low and high tempering, respectively, and/or nitriding, led to higher hardness values compared to the case of quenching at a lower temperature.

Optical microscopy analyses performed on AISI3160 samples treated according to different technological parameters for quenching and tempering revealed that the nitrided layer has increasingly larger dimensions as the temperature from which the quenching was performed increases and the temperature at which the tempering is performed is lower, but the non-uniformity and thickness obtained were still not conclusive for a pertinent assessment.

The metallographic structure of the base material was not modified by heating to 520 °C; possibly minor precipitations of phases could have occurred that add to those already existing.

By choosing the appropriate heat and thermochemical treatment parameters and equipment, one can certainly benefit from these technologies even in cases where the usual processes do not meet expectations.

4. Conclusions

- Even though the layer is almost invisible, there is a small increase in microhardness
- The metallographic analysis indicates that the nitrided layer is discontinuous, very thin and in places difficult to see because: the passive chromium oxide film (Cr₂O₃) blocks nitrogen diffusion, chromium reacts rapidly with nitrogen forming local nitrides which practically represent diffusion barriers for nitrogen (nitrogen diffusion taking place more slowly) and last but not least, there is continuous repassivation of the surface which produces its non-uniformity.
- Experimental results confirm the limitations of conventional nitriding applied to the AISI 316 stainless steel determined by the stability of the passive Cr oxide film and the reduced mobility of nitrogen in the structure which leads to the limitation of the nitrided layer thickness, its lack of continuity and in the most frequent cases to an increased porosity of the nitrided layer.

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