MICROSTRUCTURAL AND FUNCTIONAL CHANGES INDUCED BY HEAT AND THERMOCHEMICAL TREATMENTS OF X5CrNi18-10

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Abstract: *This paper analyses the influence of heat and thermochemical treatments on the structure and mechanical properties of X5CrNi18-10 (AISI 304) austenitic stainless steel. In particular, there are assessed the effects of treatment parameters – temperature, time, cooling rate and working atmosphere – on internal structure and hardness. There are compared processes such as annealing, hardening, ageing and plasma nitriding. The results indicate that heat treatments applied in a controlled manner can improve properties, and thermochemical treatments can significantly increase surface hardness without compromising the anti-corrosion integrity if the parameters are well chosen. The paper provides a solid basis for the optimal selection of treatments in demanding industrial applications.*

Keywords: *X5CrNi18-10, heat treatment, nitriding, hardness, structure*

1. INTRODUCTION

In the context of modern industrial development, materials must meet increasingly stringent requirements with respect to mechanical performance, corrosion resistance, and durability in extreme environments. Austenitic stainless steels, especially X5CrNi18-10 (AISI 304), are extensively used in industries such as food, pharmaceutical, chemical and nuclear ones due to their chemical and structural stability [1, 2].

However, X5CrNi18-10 features relatively modest mechanical strength and low hardness in its standard state, while its sensitivity to intergranular corrosion following improper heat treatment constitutes a risk for critical applications [3, 4].

X5CrNi18-10 (AISI 304) is chosen when a combination of aesthetic appearance, corrosion resistance, and ease of processing/welding is required, in environments that are not extremely aggressive.

It is recommended for his properties: good corrosion resistance in atmospheric environments, drinking water, and mildly oxidizing solutions, excellent toughness even at low temperatures, very good weldability without the need for post-weld heat treatments, working temperature range: up to about 300–350 °C in continuous service.

Therefore, research focuses on optimizing heat and thermochemical treatments to improve the performance of this material, without compromising its essential properties.

The purpose of this paper is to identify and analyze the correlations between the specific parameters of the applied treatments and the resulting properties of X5CrNi18-10. Both conventional heat treatments and advanced thermochemical treatments – nitriding – are being investigated, as these can significantly influence the internal structure and functional properties of this material [4, 5].

2. EXPERIMENTAL RESEARCH

The experimental tests were performed on samples of X5CrNi18-10 austenitic stainless steel with the chemical composition presented in Table 1.

Table 1. Chemical composition of X5CrNi18 10 austenitic stainless steel, according to STAS and determined chemical analysis

Steel grade			Chemical composition [%]							
Numerical symbols	Alphanumerical symbols	STAS 3583-87	C	Cr	Mn max	Si max	Ni max	S max	Mo max	Cu max
1.4301	X5CrNi18 10	X5NiCr180	0.03	17-19	2.00	1.00	8.00-10.5	-	-	-
determined chemical analysis			0.02	18.3	1.80	1.00	9.5	-	-	-

After determining the chemical composition, the samples underwent heat and thermochemical treatments, hardness determinations and structural analysis. There should be noted that the hardness values presented below are the average of 5 measurements.

Table 2 shows the secondary heat treatment parameters and the hardness values obtained.

Table 2. Heat treatments of X5CrNi18-10

Item	Heat treatment					
	Homogenisation annealing [°C]	Solution immersion quenching		Artificial ageing		Hardness [HB]
		Temp [°C]	Cooling			
1	1050	-	-	-		282
		1050	water	510	air	235

It follows that, in delivery state, the material is characterized by greater hardness than after preliminary treatments; this implies that it was in a slightly out-of-equilibrium state due to cold drawing, a normalizing treatment, etc.

After the homogenization annealing and approaching the equilibrium state, there were observed slight decreases in hardness, which was to be expected.

The purpose of quenching from 1050°C is to resolve precipitated phases and improve chemical properties.

Heating and cooling were carried out in air. The rapid cooling, necessary to prevent the precipitation process after quenching, was carried out in water and the ageing was carried out in air.

There was selected an artificial ageing temperature of 510°C in order to avoid the 350....450°C range, where intergranular precipitation phenomena and tempering embrittlement may occur.

Figures 1-4 show the structures after primary and secondary heat treatments.

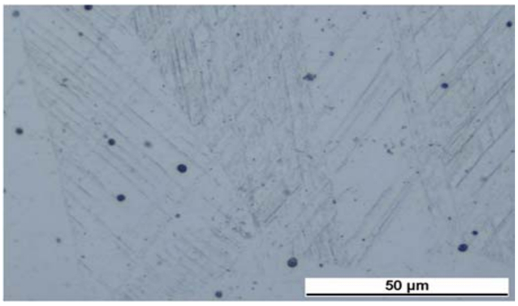

FIG. 1 X5CrNi1810 in delivery state. Aqua regia etching. 200:1

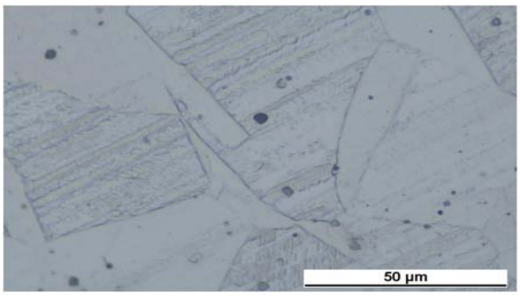

FIG. 2 X5CrNi1810 after homogenisation annealing at 1050 °C. Aqua regia etching. 200:1

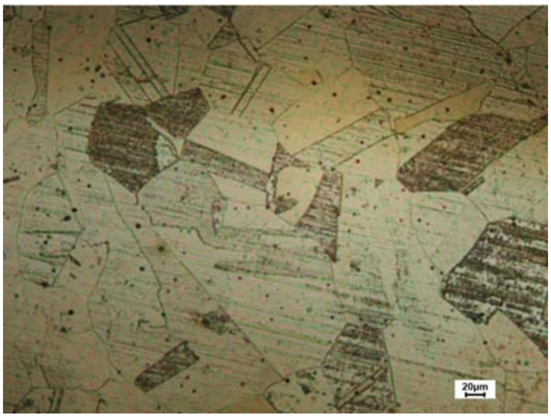

FIG.3 X5CrNi18-10. After quenching from 1050°C Aqua regia etching. 1000:1

FIG.4 X5CrNi18-10. Metallographic structure after solution immersion quenching from 1050°C and artificial ageing at 510°C. Aqua regia etching. 1000:1

In the three states, the X5CrNi18-10 structure consists of polyhedral γ solid solution crystals, with twinning bands. Compared to the delivery state, after the heat treatment, the polyhedral crystals are better outlined. To some extent, an increase in granulation is also observed. The different color shades are due to the different orientation of the crystals in the polycrystalline assembly.

In the next stage of the experimental research, there were monitored the changes in the steel performance as a result of applying a quenching + ageing sequence followed by nitriding. In this context, the aim was to modify the superficial hardness, respectively the generalized corrosion resistance of these steels. Table 3 presents the parameters of the ion nitriding treatment.

Table 3. Ion nitriding treatment parameters

Gas composition	NH_3 - Ammonia cracked at 750 °C
Holding at temperature	~20 h
Pressure	2.5-3.5mbar
Holding temperature	420+/-10°C
Thickness of the deposited layer	30-60 µm

The nitriding process is more efficient in the γ phase and slower in the α phase. Usually, the α phase is stable at lower temperatures, while γ at higher temperatures. For this reason, nitriding is carried out at lower temperatures, when they are generally encountered in BCC matrices.

In this case, the temperature selected was of 420°C, for 20 hours, not the 520°C temperature used for nitriding α structure steels. This is because the 520°C temperature can lead to a continuous desorption process.

From a practical point of view, a higher efficiency was found at 420°C compared to 520°C for γ structure steels; this is because the nitride layer of very small thickness and high hardness deposited on a relatively soft γ phase layer leads to the cracking of this layer and of the transition zone.

The metallographic structures of the thermochemically treated samples were analyzed next.

The structures were studied in parallel with microhardness measurements (Table 4 and FIG. 5).

Table 4. Hardnesses values of X5CrNi18-10 samples

Steel grade	Heat treatment				Plasma nitriding			Hardness	
	Solution immersion quenching			Artificial ageing					
	Temp. [°C]	Holding [h]	Cooling	Temp. [°C]	Temp. [°C]	Time [h]	Layer thickness [µm]	Core HV	Layer $HV_{0.1}$
X5CrNi 1810	1050	2	water	510	420	20	0.3..0.6	60.4-60.9	708-719

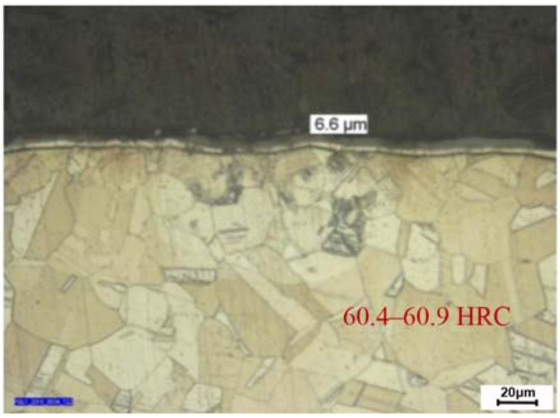

FIG. 5 X5CrNi18-10. Solution immersion quenching at 1050°C, artificial ageing at 510°C, nitriding at 420°C. layer thickness 6.6 µm. Etching reagent: aqua regia. 200:1

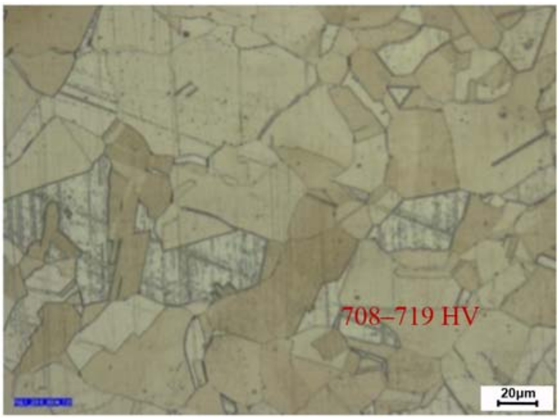

FIG. 6 X5CrNi18 10. Solution immersion quenching at 1050°C, artificial ageing at 510°C, ion nitriding at 420°C. Core – polygonal austenite crystals. Etching reagent: aqua regia. 200:1

No structural transformations can be observed for prolonged holding at 420°C. There may have also occurred, however, minor intercrystalline precipitations.

Nitrogen diffuses with difficulty into the γ crystal lattice of X5CrNi1810. Very thin surface layers are obtained as a result thereof. These layers generally consist of two layers: a white one made up of chemical compounds and a dark one of nitrogen-enriched solid solution underneath. The surface hardness is relatively high, but decreases sharply towards the interior.

No visible transformations occur in the basic mass upon heating to the nitriding temperature of 420°C, as the structure consists of polygonal crystals of γ substance and twinning bands.

CONCLUSIONS

Homogenisation annealing had a significant impact on restoring the structural homogeneity of X5CrNi18-10. As a result of this treatment, chromium carbides were dissolved from the grain boundaries, preventing the occurrence of intergranular corrosion.

Quenching applied conventionally did not lead to X5CrNi18-10 hardening, because it has a stable austenitic structure that does not undergo martensitic transformations.

Ageing applied after quenching had no significant effects, given that the material did not undergo a structural transformation during the quenching phase.

Nitriding determined a significant increase in the hardness of the stable superficial layer without compromising the basic austenitic structure, preserving corrosion resistance, while avoiding excessive oxidation and increasing wear resistance.

Of all the treatments analysed, plasma nitriding proved to be the most effective for increasing hardness without adversely affecting corrosion resistance. In contrast, quenching and ageing, effective for classically quenched steels, offer no significant benefits to X5CrNi18-10 considering the stability of the austenitic structure.

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