Tribological behavior and microstructural characterization of austenitic stainless steel stabilized with Nb and V

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Abstract. This study deals with the influence of the addition of vanadium and niobium with concentrations of 1.2% and 0.14%, respectively, on the variation of the microstructure, and the tribological behavior of an AISI309 austenitic stainless steel. The studied specimens were structurally characterized by optical microscopy, scanning electron microscopy (SEM) X-ray diffraction and differential scanning calorimetry (DSC). These samples were also subjected to a tribological study using the friction wear test. The results show that the addition of vanadium and niobium causes a precipitation of stable carbides (VC, NbC) which in turn causes a decrease of the chromium carbide precipitation rate. Tribological test results have also shown that the addition of these two elements improves the wear resistance of AISI309 austenitic stainless steel by decreasing the coefficient of friction from 0.824 to 0.554.

1. Introduction

Austenitic stainless steels are the most widely used metallic materials due to their cost, ease of manufacture and good corrosion resistance. They represent more than 70% of world stainless steel production. They are used in various fields due to their properties that enable them to resist in severe environments [1-3]. Due to their FCC crystal structure, these steels are considered more ductile materials. They are characterized by low hardness, low wear resistance and excellent corrosion resistance. When these steels are held at a temperature range between 823 and 1023°C, they become the seat of a precipitation of chromium-rich carbides located along the grain boundaries. This precipitation causes a simultaneous depletion of chromium in the area around these carbides [4, 5]. This phenomenon involves microstructural changes in austenitic stainless steels causing degradation of their corrosion resistance. This phenomenon can be reduced by using conventional techniques such as reducing the carbon content, addition of stabilizing elements, heat treatments, etc. The addition of stabilizing elements to austenitic stainless steels induces several useful effects such as intergranular corrosion protection and precipitation hardening [6, 7].

The objective of this work is to study the influence of the addition of vanadium and niobium on the structure and tribological behavior of AISI309 steel.
2. Materials and methods

The base material used in this study is an austenitic stainless steel type AISI309. Two chemical compositions are the subject of this work: (i) basic steel without additions (A steel) (ii) steel with vanadium and niobium additions (B steel). The chemical composition of these two steels is shown in Table 1.

|       | C     | Si    | Mn    | P      | S      | Al    | Cr    | Ni    | Cu    | V      | Nb     |
|-------|-------|-------|-------|--------|--------|-------|-------|-------|-------|--------|--------|
| Steel A | 0.4   | 1.58  | 0.74  | 0.019  | 0.012  | 0.28  | 23.94 | 14.23 | 0.096 | 0.08   | 0.022  |
| Steel B | 0.4   | 1.54  | 0.79  | 0.018  | 0.010  | 0.20  | 23.99 | 14    | 0.10  | 1.2    | 0.14   |

The samples underwent mechanical polishing with abrasive papers of different grain sizes followed by a final polishing with 3µ diamond paste and electrolytic etching with 10% oxalic acid. A Nikon ECLIPSE LV 100ND type optical microscope and a Zeiss type scanning electron microscope are used for the metallographic study. A Rikagu type diffractometer was used for the X-ray analysis on a 2theta range varying between 0 and 120° with a 0.02° step using a copper anticathode. The spectra were fitted using the Maud software.

The analysis of structural transformations during a single heating cycle from 25° to 1100°C was performed using a Thermal Analysis SDT Q600 - TA Instrument colorimeter. The study of wear properties was carried out using a ball-on-disc Tribometer (CSM instruments) with 10.05 cm/s linear speed and 5N normal force.

3. Results

3.1. Optical microscopy Observation

Figure 1 shows the optical metallographic structures corresponding to both steels A and B. The micrograph of steel A reveals a dendritic structure. It is composed of an austenitic coarse grain matrix of dendritic morphology and a continuous lattice of chromium carbides. In the structure of steel B, we note that the addition of vanadium and niobium refined the structure of AISI309 steel and consequently increased the hardness from 40 to 76 HRB. This effect is caused by the presence of niobium and vanadium carbides in the matrix [8]. Their existence gives the matrix a higher hardness. The refinement of the grains is due to the presence of hard precipitates based on the added elements, which serve as a germination center for austenite and block the progression of austenitic grain boundaries during solidification [9].

![Figure 1. Optical micrographs of the both steels A and B.](image-url)
3.2. SEM Observation

The SEM micrographs for A and B steels are given in Figure 2. The micrograph of steel A shows an increased precipitation of carbides from heterogeneous morphology (lamellar and globular) on the grain boundaries. The addition of vanadium and niobium (V, Nb) influences the refinement of carbides by modifying their morphology [10,11].

Two forms of fine precipitates characterize the microstructure of steel B. The first precipitate is localized at the grain boundaries and manifests itself as a continuous lattice, the second precipitate of globular form appears inside the grains.

![Figure 2. SEM micrographs of both steels A and B.](image)

3.3. XRD analysis

X-ray diffractograms of the two steels are shown in Figure 3. A quantitative analysis using the MAUD software based on the Rietveld method was performed. This method allowed us to define the nature and proportion of the existing phases [12]. The X-ray spectra of both steels show the presence of gamma iron, M\textsubscript{23}C\textsubscript{6} and M-C\textsubscript{3} carbides and Cr\textsubscript{2}O\textsubscript{3} oxides in steel A. In addition to the mentioned phases, an MC type carbide was detected in B steel. The content of M\textsubscript{7}C\textsubscript{3} chromium carbides in the cast steel structure is high compared to that of M\textsubscript{23}C\textsubscript{6} carbide. In terms of phase proportion, B steel shows a reduction in the content of M\textsubscript{23}C\textsubscript{6} and M-C\textsubscript{3} carbides and the appearance of peaks characteristic of vanadium and niobium (VC, NbC) carbides (Table 2). This type of carbide has been reported in several papers: [13,14].

![Figure 3. X-ray diffractogram of both steels A and B.](image)

### Table 2. Volume fraction of phases of both steels A and B.

|        | Wt % | Gamma-Fer | M\textsubscript{23}C\textsubscript{6} | M\textsubscript{7}C\textsubscript{3} | Cr\textsubscript{2}O\textsubscript{3} | NbC | VaC |
|--------|------|-----------|-----------------------------------|-----------------------------------|-----------------------------------|-----|-----|
| Acier A| 83.79| 6.04      | 10.03                             | 0.14                              | -                                 | -   | -   |
| Acier B| 86.82| 4.98      | 7.21                              | 0.09                              | 0.16                              | 0.74| -   |
3.4. DSC analysis

The figure 4 shows the results obtained by the DSC technique for both steels A and B. We note the presence of two peaks, an exothermic peak located between 627°C and 869°C for steel A and between 654°C and 832°C for steel B. The appearance of the exothermic peak is attributed to precipitation of $M_23C_6$ carbides, whereas the endothermic peak is related to dissolution of this type of carbide [15]. The addition of vanadium and niobium contributed to the shift of transformation temperature related to precipitation of the $M_23C_6$ carbides to higher temperatures with a decrease in energy flux. A slight shift in the dissolution temperature of the $M_23C_6$ carbides, related to the decrease in flow of absorbed energy is noted during the dissolution of $M_23C_6$ carbides.

![Figure 4. DSC thermograms of both steels A and B.](image)

3.5. Friction wear

The influence of the addition of vanadium and niobium on the friction wear resistance of steels A and B is shown in Figure 5. We note that the evolution of the coefficient of friction in both steels passes through two stages:

- The first stage is characterized by a sudden increase in the coefficient of friction ($\mu$) over a distance of 2m. This increase is due to shocks between the asperities of the surfaces antagonists [16];
- In the second stage, a stabilization of the coefficient of friction is noted. The asperities emerge from both surfaces and play the role of the third body. The formation of the third body affects the variation of coefficient of friction [17].

The steel B has a low coefficient of friction compared to A steel (0.824 for steel A and 0.554 for steel B). This reduction is due to the presence of stable carbides of vanadium and niobium, which increases the friction wear resistance of steel B.

![Figure 5. Variation of friction coefficient of both steels A and B.](image)

3.6. Morphology of worn surfaces

Figure 6 shows the SEM micrographs of both steels A and B after the friction wear test. The micrograph of steel A shows a slightly deformed worn surface with the presence of deep grooves parallel to the
direction of friction, these grooves result from third body particles [18, 19]. The micrograph of the surface of steel B shows the presence of wear debris, these debris are particles separated from the steel during friction test. The grooves are not deep in this steel.

Figure 6. SEM micrographs of the worn surfaces: (a) steel A, (b) steel B.

4. Conclusion

At the end of this study, the following conclusions can be drawn:

- The optical and SEM micrographs obtained from both steels showed that the microstructure reveals the presence of two principal microstructural constituents namely: an austenitic matrix and a continuous interdendritic chromium carbide network. Through these microstructures, a refining effect of the added alloying elements is detected. The grain refinement is clearly related to the formation of MC-type hard carbides which are vanadium and niobium carbides. These latter prevent the progression of the austenitic grain boundaries during solidification, which is responsible for the microstructure fineness.

- The addition of vanadium and niobium has contributed to a shift in the transformation point related to the precipitation of M23C6 carbides from 627°C to 654°C

- With the presence of vanadium and niobium, the friction coefficient decreases from 0.824 to 0.554

- The wear mechanism observed for steel A is abrasive wear.

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