INFLUENCE OF HEAT TREATMENTS ON THE ABRASIVE WEAR RESISTANCE OF SUPERMARTENSITIC STAINLESS STEEL

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Resumo. Martensitic and supermartensitic stainless steels are corrosion resistant materials with broad application in chemical, petrochemical and power generation industrial sectors. The supermartensitic stainless steel 13 %Cr (composition: 0.0278 %C; 12.21 %Cr; 5.8 %Ni; 1.95 %Mo; 0.519 %Mn; 0.28 %Ti; 0.0112 %P; 0.0019 %S; 0.013 %N in wt%) is indicated for applications that require corrosive and abrasive wear resistance due to its higher mechanical resistance and hardness in comparison to other stainless steels. In this work the abrasive wear resistance of supermartensitic stainless steel was characterised using pin on disc abrasion tests complemented by pendular scratch tests. The microstructure and mechanical response of supermartensitic stainless steel was modified by five different heat treatments: 1) quenching from 1000°C; 2) quenching and tempering at 300°C, 1 h; 3) quenching and tempering at 500°C, 1 h; 4) quenching and tempering at 650°C, 1 h; 5) quenching and double tempering at 670°C, 2 h followed by 600°C, 2 h. Tempering treatments at 300°C and 500°C promoted a hardening effect on the steel due to fine Mo_2C precipitation. Tempering at 650°C and double tempering caused softening of the material due to the large amount of austenite as observed by magnetic measurements and metallographic analysis. The abrasive wear resistance was obtained using pin abrasion tests. In this apparatus, the pin sample slides against sandpaper making a spiral trajectory of 4,2 mm. Corundum (Al_2O_3) 220 mesh sandpaper was used. The normal load of 13.5 N was applied directly by means of dead weight. Results showed that the average abrasive wear resistance increased up to 6 % for samples tempered at 650°C in comparison with the non-tempered material due to the presence of Mo_2C precipitates. For double tempered samples, the wear resistance decreased 8.5 % due to their higher amount of austenite. Pendular scratch tests helped to understand the change in wear rate obtained by pin abrasion tests.

Palavras chave: *abrasive wear; supermartensitic stainless steel; pin abrasion test; pendular scratch test; wear mechanism*

1. INTRODUÇÃO

One of the main problems involving oil and natural gas production is related to the exposure of equipment to a mixture of fluids and of fine particles of sand. This mixture passes through several steps of processing and induces corrosive and abrasive wear in equipment such as Christmas trees, manifolds and pipelines. The supermartensitic stainless steels (SMSS) are indicated for applications that require corrosive and abrasive wear resistance due to their higher mechanical resistance and higher hardness when compared with other stainless steels.

In this work, the abrasive wear resistance of supermartensitic stainless steel was characterized using pin on disc abrasion tests complemented by pendular scratch tests. The microstructure and mechanical response to supermartensitic stainless steel was modified by five different heat treatments. The effect of the microstructural changes on the wear mechanism was analyzed.

2. EXPERIMENTAL METHODS AND MATERIALS

2.1. Samples and heat treatments

A 200 mm of diameter tube with 10 mm wall thickness was purchased as sample material. The chemical composition of the supermartensitic steel studied is presented in Table 1. This measurement was carried out by Valourec Mannesman. In detail, elements C, N and S were analysed by the combustion method, while the contents of the other elements were determined by the optical emission technique.

Table 1. Chemical composition (wt%) of the supermartensitic stainless steel 13 %Cr.

%C	%Cr	%Ni	%Mo	%Mn	%Ti	%P	%S	%N
0.0278	12.21	5.8	1.95	0.519	0.28	0.0112	0.0019	0.013

The specimens were heat treated by water quenching and tempering. The quenching was carried out after soaking at 1000°C for 1 hour. After quenching, the specimens were tempered according to conditions detailed in table 2.

Table 2. Heat treatment and specimen nomenclature.

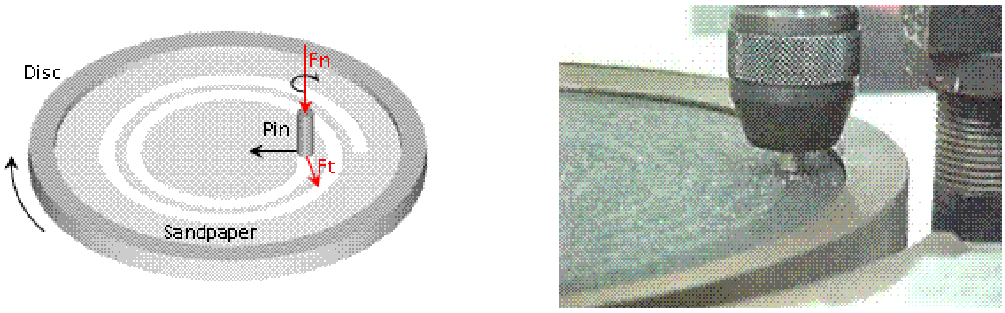
	Sample	Heat treatment
1	Q	Quenching from 1000°C;
2	QT300	Quenching from 1000ºC, tempered at 300ºC for 1h
3	QT500	Quenching from 1000ºC, tempered at 500ºC for 1h
4	QT650	Quenching from 1000ºC, tempered at 650ºC for 1h
5	DT	Quenching from 1000ºC, Double tempered (670ºC / 2h + 600ºC / 2h)

Previous work (Silva *et al*., 2011; Da Silva *et al*., 2011) presented the mechanical properties of these samples that were extensively characterized in terms of tensile test curves, Vickers hardness and Charpy impact energy tests. The austenite volume fraction ($C\gamma$) was measured using magnetometry technique and metallographic analysis (Silva *et al*., 2011; Da Silva *et al*., 2011). The strain-hardening exponent (n) was calculated using the response of tensile test curves and Hollomon's model (Silva *et al*., 2011; Dieter, 1998).

For the pin abrasion tests, samples were machined to the shape of cylinders 5 mm in diameter and 15 mm in length. Blocks with the dimensions of 10 x 10 x 35 mm were used as samples for pendular scratch tests. For scratch tests the working face of the samples was grinded, sanded and metallographically polished.

2.2. Pin abrasion test

The pin abrasion test used in this work is described in ASTM G132 Standard (ASTM, 2007) and presented in Fig. 1.



(a) (b)

Figure 1. (a) Schematic representation of the pin abrasion test; (b) Close-up view of the sample mounted in the equipment.

This equipment promotes relative motion between the abrasive surface and the pin surface. As presented in Fig. 1, the wear track of a pin describes a continuous non-overlapping spiral path. The pin also rotates on its axis during the movement. The pin specimen is pressed against the abrasive surface with a specified dead weight load. This movement induces the sliding of abrasive particle on the pin surface in random directions (Franco *et al*., 1989; Al- Rubaie *et al*., 1999).

The amount of wear was determined by weighing specimens before and after passes. Results were plotted as mass loss versus sliding distance. In each pass the sample travels the distance of 4.2 m. Tests were repeated 5 times for each condition, giving the total sliding distance of 21.0 m.

The abrasive wear mechanism was observed using scanning electron microscopy (SEM - model: Leo 940A; Carl-Zeiss).

2.3. Pendular scratch test

The test consists in producing a scratch in a sample surface with an indenter of defined geometry. The pendulum used has a mass of 0.877 Kg, set free at a 154° angle, with 4.24 J of initial energy and a percussion speed of 4.9 m.s^{-1}. The indenter is fixed at the extremity of the pendulum in the indenter holder. The pendulum is released from a defined height with the initial potential energy. Its trajectory is adjusted to scratch the sample at the position of higher velocity with a maximum of kinetic energy. After the scratch, the pendulum rises up to a certain height, that is recorded on the scale. The scale registers directly the energy consumed in the test, i.e. the difference between the initial and final potential energy of the pendulum. The maximum depth of the scratch is set by using a micrometer adapted to the sample holder. This positioning system elevates or lowers the sample in a 250 μm of range.

The specific energy, e, is calculated according equation 1.

$$e = \frac{E}{V} \quad (1)$$

The specific energy is defined by the ratio between the energy absorbed in the scratch, E, and the volume loss, V. The volume of the scratch is obtained using laser interferometry (model UBM Messtechnik; MicroFocus).

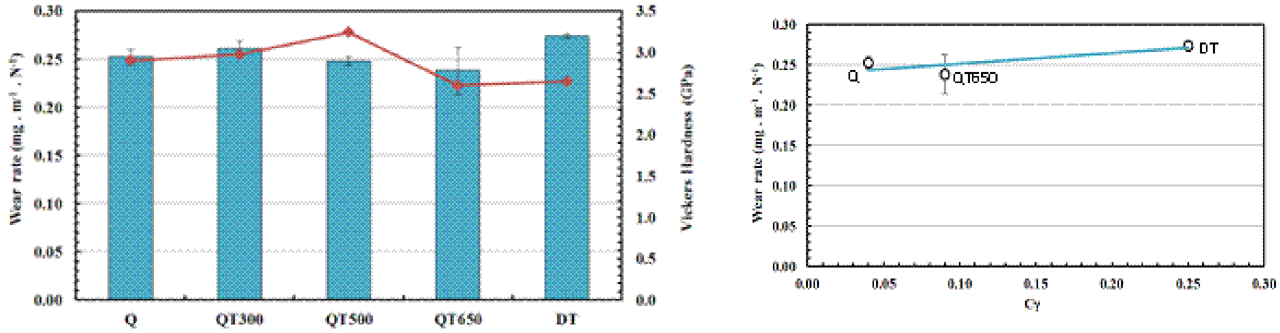
The indenter was produced from a 5 mm square bar of WC-Co that was obtained by a powder metallurgy process. The indenter was cut and ground to a square base pyramid shape. The pyramid faces received a polished finish. The indenter tip was truncated to prevent its fracture. The indenter dimension was monitored using an optical microscope.

The abrasive wear mechanism that occurred during the scratch process was observed using SEM.

3. RESULTS AND DISCUSSION

The wear mechanism was the same for all samples and test conditions. In this case the prevailing wear mechanism was microcutting and, in a small intensity, microploughing. The wear was related to the repeated interaction of scratches in the sample surface.

Figure 2 presents the results obtained using pin abrasion tests in function of the hardness and austenite volumetric fraction of the samples.



(a) (b)

Figure 2. Wear rate values obtained in pin abrasion test as function of (a) Hardness and (b) Austenite volumetric fraction (Cγ).

The wear response was statistically equivalent for all samples tested except for the DT sample. The correlation between wear rate and hardness values was not clear (Fig. 2a). Sample DT presented the highest wear value, which was related to the lowest hardness and the highest amount of austenite. In fact the wear rate presented a linear correlation when plotted in function of austenite volumetric fraction, Fig. 2b.

Analyzing only the average value, the abrasive wear rate decreased 6 % when sample Q was compared whit sample QT650. This result was related to the presence of TiC and Mo_2C precipitates (Da Silva *et al.*, 2011)

Figure 3 shows an example of a scratch topography obtained using laser interferometry technique.

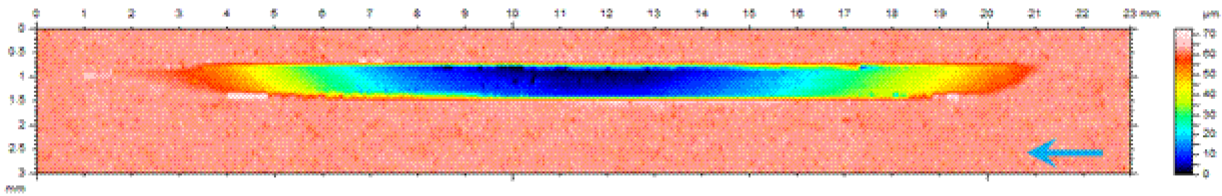
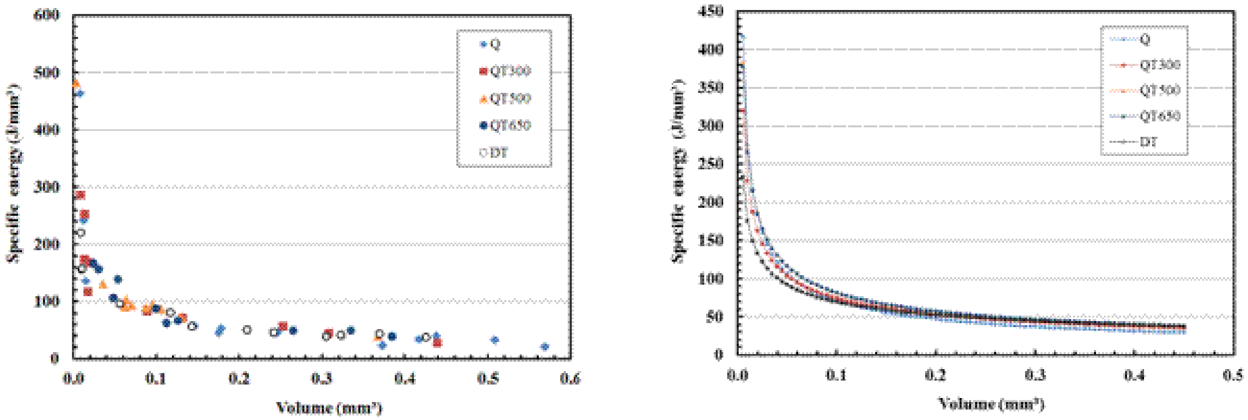


Figure 3. Scratch topography of sample Q in the top view.

For scratch volume measurements, the border of the surface topography was used as a datum, representing the surface of the sample, Fig. 3. The volume below this reference surface was the material removed in scratching.

Figure 4 presents the variation of specific energy, *e*, absorbed during the scratching process with the volume of the scratch, *V* (equation 1).



(a) (b)

Figure 4*:* (a) Specific energy data obtained by the pendular scratch tests; (b) Curves adjusted through a power model.

The relationship between the specific energy and the scratch volume is expressed by a power model as presented in equation 2 (Vingsbo and Hogmark, 1984; Franco and De Mello, 1992):

$$e = kV^{q} \qquad (2)$$

Where *k* and *q* are coefficients related to material properties and *V* is the volume of the scratch measured by laser interferometry.

Figure 4b shows the curves adjusted from Fig. 9a data using the power model presented in equation 3. For volume values lower than 0.1 mm³, the specific energy of scratching increased rapidly with the decrease of volume loss. This behavior was linked to the size effect phenomenon (Sales *et al*., 2007; Shaw 2003) and the changing of mechanisms of chip formation (Vingsbo and Hogmark, 1984; Franco and De Mello, 1992). After a scratch volume of 0.2 mm³ the specific energy remains quite constant as observed in Fig. 4b. The coefficients *k* and *q* obtained for tested samples are listed in table 3.

Table 3. Parameters of the scratch test power model according to equation 2.

	Sample	*k*	*q*	Correlation coefficient - R^2
1	**Q**	17.98	0.59	0.93
2	**QT300**	23.93	0.49	0.93
3	**QT500**	24.56	0.52	0.98
4	**QT650**	24.91	0.51	0.93
5	**DT**	27.21	0.41	0.97

The parameters *k* and q are found to be related to the strain hardening properties and localized strain respectively (Bryggman *et al*., 1986).

4. CONCLUSIONS

The main results found in this work are summarized as follows:

- Pendular scratch tests helped to understand the change in wear rate obtained by pin abrasion tests.
- The wear rate obtained by pin abrasion tests was related to the austenite volume fraction. This metallurgical parameter was also related to the scratch test parameter (*k*) and Hollomon's strain-hardening exponent (*n*). The

wear rate was the highest in samples containing austenite because they had the lowest hardness and highest capacity of plastic deformation thus offering the lowest resistance to abrasive wear.

- In pin abrasion tests, wear topography was associated with the sliding of abrasive particles in random direction. The prevailing wear mechanism was microcutting.
- The prevailing wear mechanism presented in pendular scratch tests was microcutting with a small amount of microploughing in the sides of the scratch.
- The specific energy (*e*) obtained by scratch tests was characteristic of the material response. Values of *e* followed a power model when plotted as a function of the volume of the scratches.
- Even taking into account the prevalence of the microcutting mechanism, the chip formed can differ according to the mechanical response of the material. This fact helped to understand the change in the wear rate obtained by the pin abrasion tests.

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6. ACKNOWLEDGEMENTS

The authors are grateful to the Brazilian research agencies CNPq, CAPES and FAPEMIG for financial support. Authors also acknowledge Prof. Eduardo Moreira from Dentistry Department of UFF for the SEM analysis.

7. RESPONSABILITY BY INFORMARTIONS

The authors are the only responsables by informations included in this work.