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Influence of sub-zero treatments on hardness and wear resistance applied on sintered steels

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Sub-zero treatments are supplementary process to conventional heat treatment, since it converts the retained austenite into martensite which is remained after heat treatment and due to martensite formation is an increase in wear resistance. In the work presented the results of the experimental researches regarding the wear resistance of steels with the concentration of 0.54 to 0.57%C; 0.75 to 0.78%C and 0.92 to 0.97%C, sintered at a temperature of 1150 ^{o}C and 1250^{o}C for 60 min, oil quenched and cooled sub-zero at 173K for 60 min, respectively 120 min.

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1. Introduction

Sub-zero treatments are applied after the hardening and tempering treatments with the purpose of increasing hardness and wear resistance, of reducing the residual stress and increase dimensional stability, increasing the mechanical properties as fatigue strength and toughness [1, 2, 3, 4].

This treatments don’t produce harmful missions emphasizing the idea that the sintered materials besides the economic benefits are also ecological materials [5].

Sub–zero treatments effects according to the growth of hardness and the wear resistances of the quenched steels, respectively the sintered metale carbide, mainly due to the reduction or elimination of the retained austenite and the quantitative increase of the martensite, also in the case of alloyed steels the precipitation of fine carbides and the finishing of the micrographic structure [4,6,7,8].

Sub-zero treatments are additional treatments to heat-treating and according to the practice range of the thermal cooling are divided into three categories: cold treatment (223-193)K; shallow cryogenic treatment (193-113)K and deep cryogenic treatment (113-77)K [3,9,10].

Sub-zero treatments are based on the third principle of thermodynamics according to which the entropy is zero, for zero absolutely.

By submitting materials at low temperatures for a long period, it aims to be brought in a state as close to the minimum entropy when takes place finishing (crystalline grain size reduction) and microstructural uniformity and then when return to the room temperature take place processes of internal contractions that generate increasing resistance to abrasive wear, adhesive and erosion, while improving corrosion resistance, fatigue and resilience resistance [7, 11, 12, 14].

Most investigations have highlighted that improving the wear resistance of materials by application of low temperature treatments, especially deep cryogenic treatment is due to increase the amount of martensite by transforming the retained austenite, carbides formation η, the precipitation of fine carbides and also due to finishing and uniformity of the structural shape and size of the structural grains [14,15, 16,17,18].

Researches have confirmed increases of abrasive wear resistance of 193 to 289% through cold treatments, respectively 315 to 335% through deep cryogenic treatments reported on wear resistance after the steels heat treatment of hardening [19,20].

After an exhaustive analysis of the problems regarding the effects for the cryogenic treatment of different types of steels, Patil and Tated conclude that “each material needs to be separately discussed and an individual process route devised for it that will depend on the combination of hardness, toughness, and wear resistance required in service” [9].

In this framework have developed a large number of studies on the behavior of the sub-zero treatment of steels in particular for cutting tools, carbide cutting plates especially those based on WC, the ceramic cutting tool inserts and of castings in special cast iron [21, 22, 23,24, 25, 26].

Regarding the materials elaborated through powder metallurgy, excepting the sintered tools steels and sintered metal carbide plates, these are less studied in terms of the effects of sub-zero treatments [27, 28, 29, 30, 31].

Researches were also oriented on the approach of some alloying hybrid processes for example with boron and the sub-zero treatments of sintered steels to increase hardness and their wear resistance [32].

For this reason was considered opportune the research of the effects of sub-zero treatments applied at 173K on the wear resistance of steels with carbon content 0.54 to 0.57% C; 0.75 to 0.78% C and 0.92 to 0.97% C produced by the heat treatment of sintering followed by heat treatment quenching in oil.

2. Experimental procedure

For experiments were developed three sets of sintered steels from three homogeneous mixtures of powders with next compositions:

Fe + 0,6%Gr
Fe + 0,8%Gr
Fe + 1,0%Gr

The mixtures were compacted by unilateral pressing into the mold with the pressure of 650 MPa.

Further crude samples were subjected to the treatments according to Fig. 1.

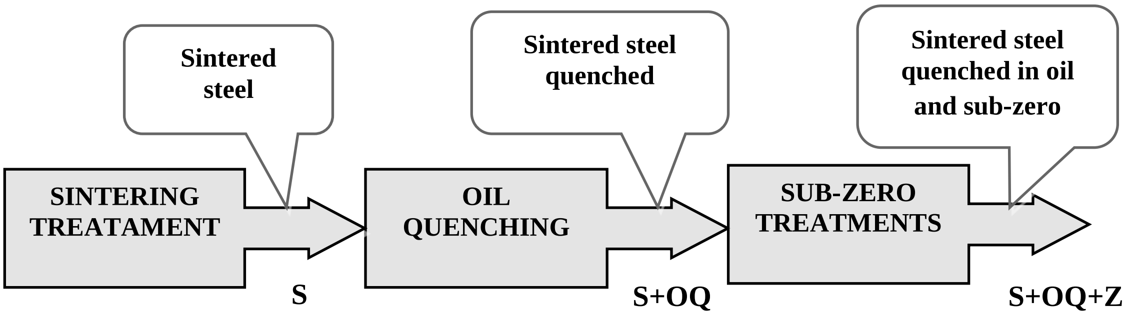


Fig. 1. Itinerary of the heat and sub-zero treatments for the crude samples.

Treatment parameters are presented in Table 1:

Table 1. Samples treatment parameters.

Treatments	Parameters	Cod
SINTERING	Sintering Temperature t_1 =1150^{o}C t_2= 1250^{o}C	S1 S2
	Time at sintering temperature τ= 60 min	
	Atmosphere Argon	
	Cooling Rate 0,8^{o}C/min	
SINTERED STEEL QUENCHED IN OIL	Austeniting Temperature 860^{o}C	
	Time at Austeniting Temperature 15 min	
	Atmosphere Argon	
	Quenching Oil	OQ
SUB-ZERO TREATMENTS	Under zero temperature 173K	
	Time at Temperature 60 min 120 min	Z1 Z2
	Cooling Rate 15^{o}C/min	

For the sub-zero thermic treatment was processed so:

- oil quenched sintered steel samples were degreased by successive immersion in cold and hot benzene and then drying at 110^{o}C for the elimination of the infiltrated oil in pores during the cooling operation for quenching;
- sub-zero medium treatment was made with an mixture of dry ice and technical ethyl alcohol;
- to provide the 173K temperature it was added dry ice on the long cooling time of samples, 60 minutes respectively 120 minutes;
- the treatment was conducted in box with special thermic isolation to avoid at maximum the thermal exchange.

3. Results and discussion

The samples which are in the three stages of treatment are symbolized as:

S - as sintered (S1- sintered at t_1 = 1150^{o}C; S2- sintered at t_2 = 1250^{o}C)

S+OQ - sintered and oil quenched (S1+OQ; S2+OQ)

S+OQ+Z - sintered, oil quenched and cooled in dry ice (S1+OQ +Z1;S1+OQ +Z2; S2+OQ +Z1; S2+ OQ+Z2)

Fig. 2 are presented the microstructures of sintered and sub-zero quenched samples, based on which were quantified by the quantitative determination the quenching constituents. For quantitative determination of constituents was used the process for the image capture and for this purpose the samples were processed metallographic according to ISO/TS14321: 1997.

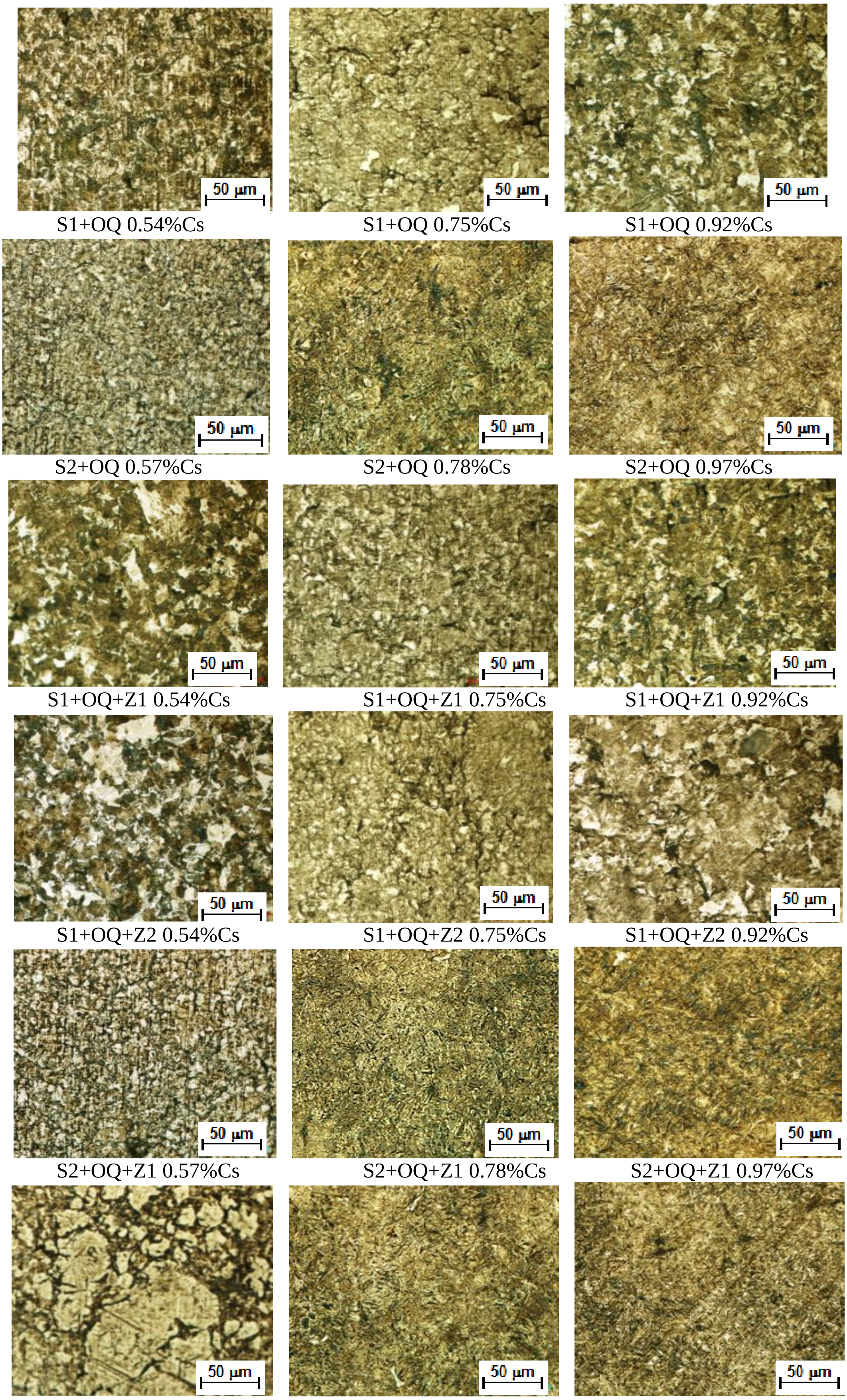
Fig. 3 are presented by compared the martensite phases and retained austenite phases in various stages of the steels treatments.

After heat treatments of sintered and hardened in oil non alloyed steels, the treatment in solution of dry ice increases their hardness. If we consider the hardness increases after cold treatment, the largest increases occurring after 120 minutes of sub-zero treatment in dry ice to steels with a high content of Cs.

It can be seen that these values are dependent on the Cs content of steels, sintering conditions and heat treatment according Fig. 2 and 3.

The micro-hardness of steels was measured according to the MPIF 51 STANDARD (2005) μHV, using for this microhardness-testing equipment NAMICON.

The results are presented in Fig. 4.



S1+OQ 0.54%Cs S1+OQ 0.75%Cs S1+OQ 0.92%Cs

S2+OQ 0.57%Cs S2+OQ 0.78%Cs S2+OQ 0.97%Cs

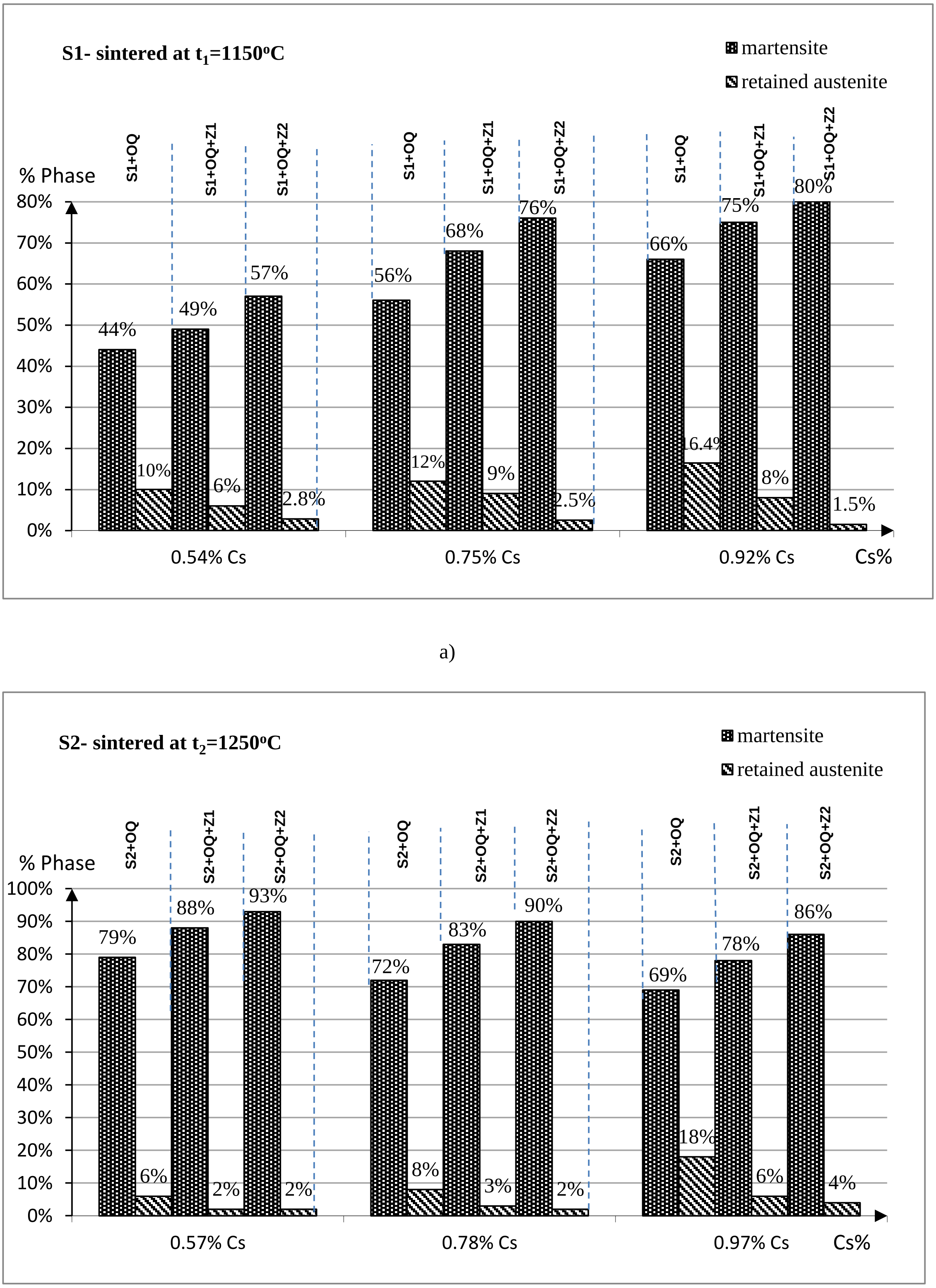
S1+OQ+Z1 0.54%Cs S1+OQ+Z1 0.75%Cs S1+OQ+Z1 0.92%Cs

S1+OQ+Z2 0.54%Cs S1+OQ+Z2 0.75%Cs S1+OQ+Z2 0.92%Cs

S2+OQ+Z1 0.57%Cs S2+OQ+Z1 0.78%Cs S2+OQ+Z1 0.97%Cs

S2+OQ+Z2 0.57%Cs S2+OQ+Z2 0.78%Cs S2+OQ+Z2 0.97%Cs

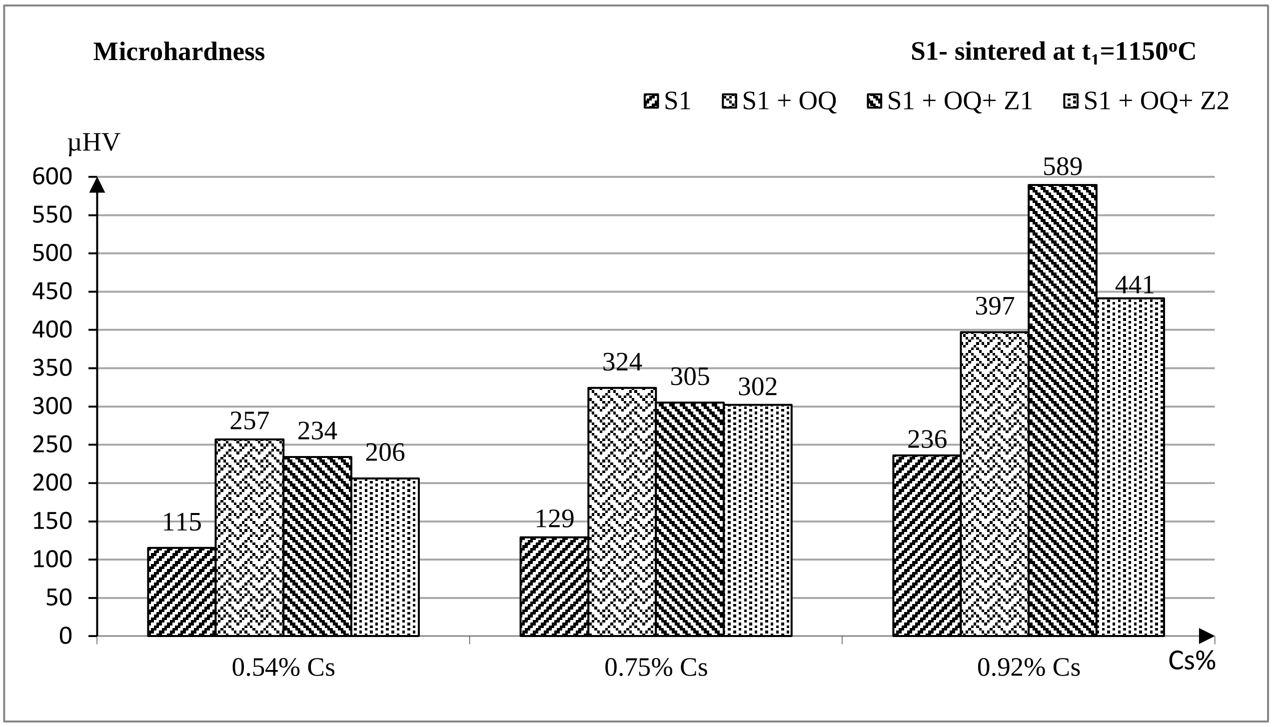
Fig. 2. Microstructures of sintered steels heat treated and sub-zero.



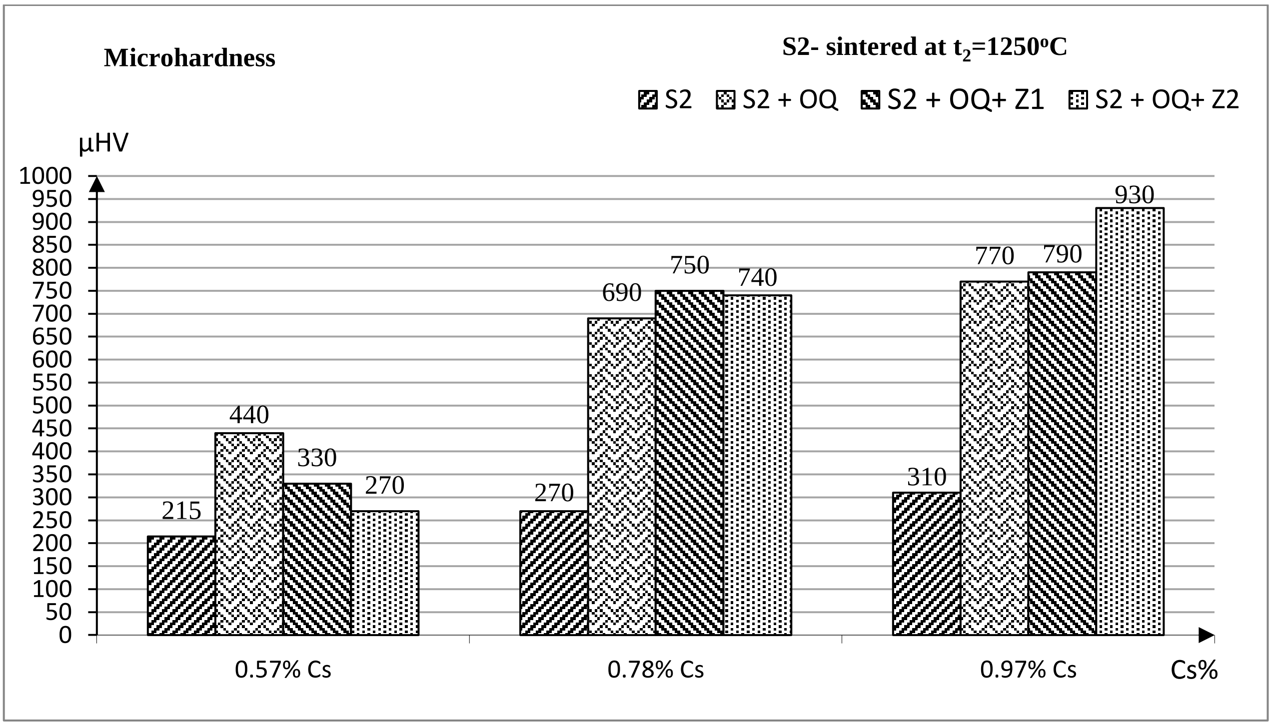
a)

b)

Fig. 3. Percentages of hardening phases in sintered at 1150°C(a) and 1250°C(b), hardened and sub-zero cooled steels.



a)



b)

Fig. 4. Micro hardness of sintered samples at 1150^{o}C(a) and 1250^{o}C, hardened and sub-zero cooled steels (b).

The characterization of the steels wear was obtained by the ball on disk procedure. For this purples the samples were subjected to tests on a tribometer TRB 01-02541 with rotary and linear oscillating movement and the trail of wear was measured by a Vickers tester with filler pin of type SURTRON 25, results are listed in Table 2 and Table 3.

4. Conclusion

The analysis of experimental research results allow the following conclusions:

-the wear behavior of sintered and hardened steels in sub-zero treated oil is influenced by sintering temperature, structural carbon content Cs (diffused carbon) and the maintaining time at sub-zero temperatures;

Table 2. Table of the wear tests of the sintered samples 60 min to 1150^{o}C.

Sample	Average friction coefficient μ	Wear section area [μm^2]	Wear ratio ω[mm^3/N/m x10^{-5}]	Depth of the wear track h[μm]
S1- 0.6%	0.491	2357	3.173	5.40
S1- 0.8%	0.345	1215	7.124	8.36
S1- 1.0%	0.311	1058	3.644	11.2
S1- 0.6% OQ	0.365	395	0.663	1.91
S1- 0.8% OQ	0.348	319	1.185	2.78
S1- 1.0% OQ	0.327	221	0.957	2.47
S1- 0.6% OQ+Z1	0.341	370	0.315	1.89
S1- 0.8% OQ+Z1	0.337	180	0.540	2.62
S1- 1.0% OQ+Z1	0.295	105	1.010	2.58
S1- 0.6% OQ+Z2	0.458	321	0.552	2.72
S1- 0.8% OQ+Z2	0.394	238	0.714	2.57
S1- 1.0% OQ+Z2	0.373	184	0.963	2.23

Table 3. Table of the wear tests of the sintered samples 60 min to 1250^{o}C.

Sample	Average friction coefficient μ	Wear section area [μm^2]	Wear ratio ω[mm^3/N/m x10^{-5}]	Depth of the wear track h[μm]
S2- 0.6%	0.542	1415	4.245	8.95
S2- 0.8%	0.396	1025	3.074	7.26
S2-1.0%	0.396	850	2.549	7.44
S2-0.6% OQ	0.468	655	1.965	4.23
S2-0.8% OQ	0.430	477	0.639	1.70
S2- 1.0% OQ	0.387	213	1.431	1.75
S2- 0.6% OQ+Z1	0.444	112	0.960	2.55
S2- 0.8% OQ+Z1	0.437	320	0.336	2.42
S2 -1.0% OQ+Z1	0.350	405	1.215	3.64
S2- 0.6% OQ+Z2	0.453	461	0.966	2.49
S2- 0.8% OQ+Z2	0.386	322	0.684	1.20
S2 -1.0% OQ+Z2	0.197	228	1.383	3.04

-the temperature of sintering ensure wear resistance features the better the more its value is greater. The experimental data highlight the fact that although the amount of martensite is lower at steels sintered at 1250^{o}C compared to steels sintered at 1150^{o}C, the hardness increases with sintering temperature and implicit resistance to wear. The explanation is that with increasing sintering temperature increases structural carbon content Cs and thus the degree of tetragonal martensite leading to hardening of the constituent;

- the maintaining time at sub-zero temperatures favors increasing of the wear resistance. The explanation is that as can be seen from the graphs, the evolution of the structural constituents, the amount of residual austenite transformed into martensite is even greater when sub-zero keeping time is longer;

Considering the research results it is found that sub-zero cooling treatment of hardened sintered steels, facilitate the wear resistance of these steels.

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