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RESEARCH ON THE TREATMENTS AT SUB-ZERO TEMPERATURES OF THE SINTERED STEELS AND HARDENED IN OIL

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Abstract. The treatments of the hardened parts at low temperatures have like main goal the reducing of the amount of retained austenite through its transformation to martensite under the action of the strong contractions which occur in cryogenic environments. In the case of sintered steels such researches were achieved on a small scale. The paper presents results of research regarding the cooling in medium of dry ice and respectively of liquid nitrogen, for the three groups of sintered steels with structural carbon content of 0.54%, 0.75% and 0.92% sintered at 1150^0C for 60 min and oil quenched.

1. INTRODUCTION

The heat treatments at low temperature are practiced because it's necessary like the steels to be brought into a state as close as possible to the entropic minimum so that at the maintaining for a long period it is producing the finishing and the leveling of the microstructures, simultaneously with structural transformation through which are realized the reducing of the retained austenite amount, and the formation of martensite. [1]

This ensures steels greater dimensional stability, increased hardness and thus increase the wear resistance. Depending on the thermal range in which these hardened parts were cooled this treatments are divided into three categories: cold treatment (223 ÷ 193 K), shallow cryogenic treatment (193 ÷ 113 K) and deep cryogenic treatment (113 ÷ 77 K). [2, 3, 4]

In the Powder Metallurgy such researches has been limited to studies on the effects of cold treatments applied to speed steel and cutting carbide plates. [5, 6, 7, 8, 9, 10, 11]

In this framework was considered appropriate the study of heat treatments to cold (cold treatment and cryogenic treatment) applied to carbon sintered steels with carbon content of 0.54 %C, 0.75 %C and 0.92 %C after their quenching into oil.

Comparative analyses were made concerning the content of martensite phase in these steels respectively regarding the microhardnesses evolution.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

The sintered steels have been made by homogenizing a powder mixture consisting of iron powder type DWP 200, graphite and zinc stearate (denoted St-Zn in what follows, used as a binder and lubricant) in the following amounts:

Fe+0.6% Gr+2% St Zn
Fe+0.8% Gr+2% St Zn
Fe+1.0% Gr+2% St Zn

The homogeneous mixtures were compacted in mold by pressing one-sided with the pressure of 650 MPa in the form of rectangular sample with the dimensions of 10x10x55 mm.

The green compacts were sintered in the conditions specified in Table 1. After sintering (denoted S) the sintered samples were oil-quenched (denoted OQ) according to the process parameters specified in Table 2, to the cold treatment (denoted Z) in the conditions specified in Table 3, and finally subjected to the shallow cryogenic treatment (denoted N) in the conditions specified in Table 4. [12]

Table 1. Applied Sintering Conditions

	Parameters	Designation
Sintering Temperature	1150^{o} C	
Time at Temperature	60 min	S
Atmosphere	Argon	
Cooling Rate	5.8 ^{o}C/min	

Table 2. Applied Quenching Parameters

	Parameters	Designation
Austeniting Temperature	860^{o} C	
Time at Austeniting Temperature	15 min	
Atmosphere	Argon	
Quenching	Oil	OQ

Table 3. Cold Treatment Conditions

	Parameters	Designation
Cold treatment - Temperature	197 K	
cooling medium	dry ice	
Time at Temperature	60 min	Z1
	120 min	Z2
Cooling Rate	25^{o} C/min	

Table 4. Shallow Cryogenic Treatment Conditions

	Parameters	Designation
Shallow cryogenic treatment Temperature	77 K	
cooling medium	liquid nitrogen	
Time at Temperature	60 min	N1
	120 min	N2
Cooling Rate	36^{o} C/min	

The procedure applied for the below-zero treatment was as follows:

- oil quenched sintered steel samples were degreased by successive immersion in cold and hot benzene and then dried at 110 ^{o}C for the elimination of the infiltrated oil in pores during the oil-quenching operation;
- cold treatment was carried out in a mixture of dry ice (solidified CO_2) and technical alcohol;
- shallow cryogenic treatment was carried out in liquid nitrogen.
- oil quenched sintered steel samples were held in below-zero cooling mediums for 60, respectively 120 min.

3. RESULTS AND DISCUSSIONS

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

S - as sintered at t = 1150 ^{0}C

S+OQ - sintered and oil quenched

S+OQ+Z - sintered, oil quenched and cooled in dry ice (Z1-60 min; Z2-120 min)
S+OQ+N - sintered, oil quenched and cooled in liquid nitrogen (N1-60 min; N2-120 min)

For all samples the following characteristics have been determined:

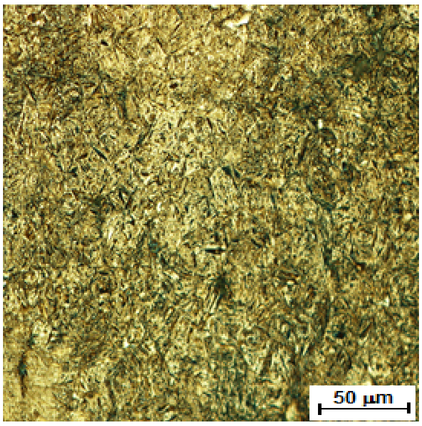
- apparent density measured according to ISO 2738:1999;
- optical microscopy analysis according to ISO/TS 14321:1997;
- structural carbon C_s in %, (calculated by means of quantitative image analysis);
- content of constituents after cooling under zero;
- μHRV 0,3 micro hardness measured according to ISO 4498:2010.

The series of micrographs in Fig.1 depict the microscopic structure for the sintered samples with various carbon content and treated in the three above mentioned structural conditions (S - as sintered; S+OQ - sintered and oil quenched; S+OQ+Z1 - sintered, oil quenched and cooled in dry ice for 60 min; S+OQ+Z2 - sintered, oil quenched and cooled in dry ice for 120 min; S+OQ+N1- sintered, oil quenched and cooled in liquid nitrogen for 60 min; S+OQ+N2 - sintered, oil quenched and cooled in liquid nitrogen for 120 min.

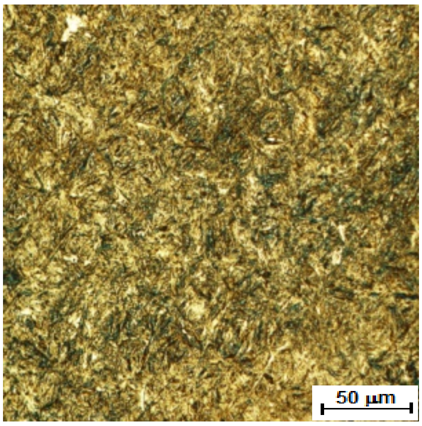
Table 5 gives the results of the quantitative image analysis applied in order to determine the amount of the various structural microconstituents identified in Fig.1 in the sintered steels in various structural conditions (F = ferrite, P = perlite, Cem = cementite, B = bainite, M = martensite, A_{ret} = retained austenite). Error determination is ~ 3%.

Table 5. Results of the quantitative image analysis in the investigated sintered steels

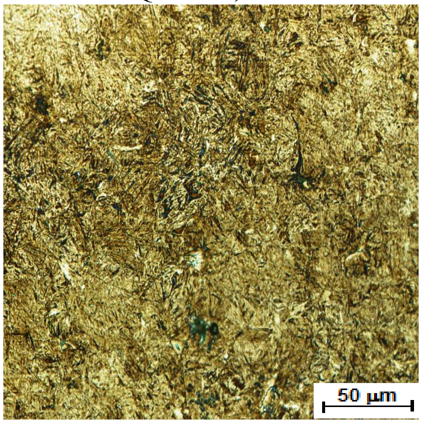
In-going Graphite [%]	**Cs** [%]	**Structural condition**	**F** [%]	**P** [%]	**Cem** [%]	**B** [%]	**Martensite**		**Retained austenite**	
							M [%]	(+)Δ**M** [%]	$\mathbf{A_{ret}}$ [%]	(-)Δ$\mathbf{A_{ret}}$ [%]
0.6	0.54	S	25.21	73.12	-	-	-	-	-	-
		S+OQ	21.65	8.91	-	14.34	43.62	-	9.64	-
		S+OQ+Z1	20.52	8.74	-	14.08	48.76	11.78	5.93	16.86
		S+OQ+Z2	20.67	3.91	-	15.21	56.38	29.25	2.78	35.97
		S+OQ+N1	17.50	3.16	-	14.23	63.12	44.70	1.98	79.46
		S+OQ+N2	18.80	2.89	-	14.92	61.15	40.18	2.16	77.59
0.8	0.75	S	2.24	97.48	-	-	-	-	-	-
		S+OQ	2.08	11.16	-	18.19	55.31	-	11.54	-
		S+OQ+Z1	2.13	4.48	-	17.25	67.16	21.42	8.81	23.63
		S+OQ+Z2	2.12	1.74	-	17.12	75.51	36.52	2.41	79.16
		S+OQ+N1	2.21	2.13	-	11.48	82.12	53.26	1.15	90.03
		S+OQ+N2	2.16	1.81	-	14.91	78.92	42.68	1.83	84.14
1	0.92	S	-	96.12	3.74	-	-	-	-	-
		S+OQ	-	4.58	3.92	8.15	65.88	-	16.38	-
		S+OQ+Z1	-	4.12	3.87	8.41	74.14	12.53	7.92	51.64
		S+OQ+Z2	-	4.15	3.65	9.12	79.57	20.78	1.51	90.78
		S+OQ+N1	-	1.11	3.63	4.15	86.63	31.49	1.48	90.96
		S+OQ+N2	-	3.38	3.72	6.47	83.71	27.06	1.72	89.49



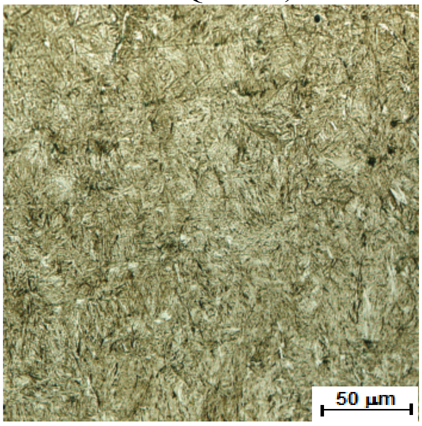
S+OQ+Z1-0,75%Cs



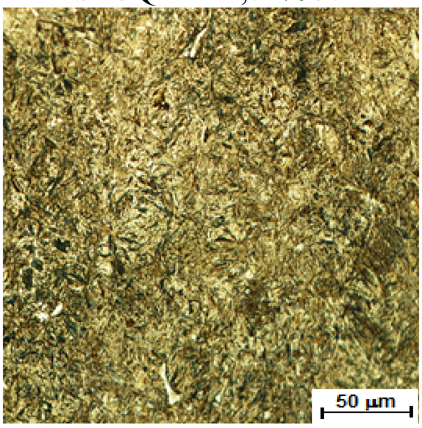
S+OQ+Z1-0,92%Cs



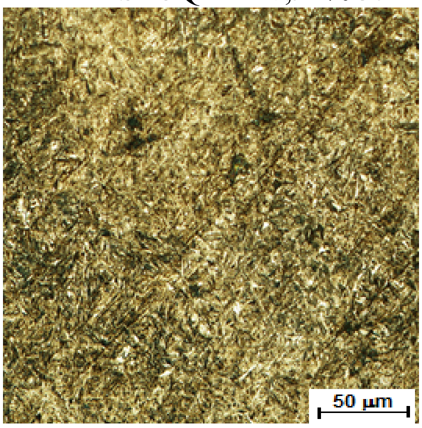
S+OQ+N1-0,75%Cs



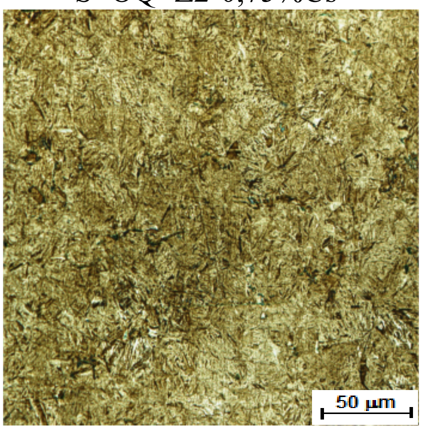
S+OQ+N1-0,92%Cs



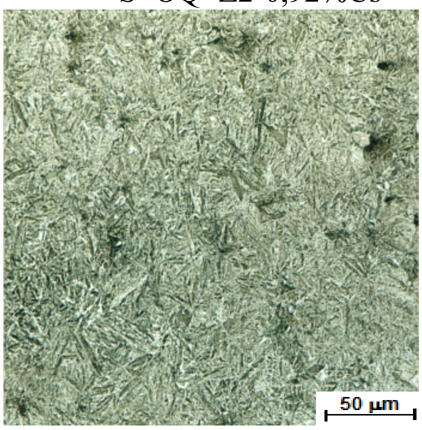
S+OQ+Z2-0,75%Cs



S+OQ+Z2-0,92%Cs



S+OQ+N2-0,75%Cs



S+OQ+N2-0,92%Cs

Fig.1. Microstructures of sintered steels samples etched with NITAL 2% and PICRAL 2%

In Fig.2 are shown comparative martensite phase and residual austenite phase in various stages of treatment of the steels, and in Fig. 3 are shown comparative the hardness values obtained in different stages of treatment of steels.

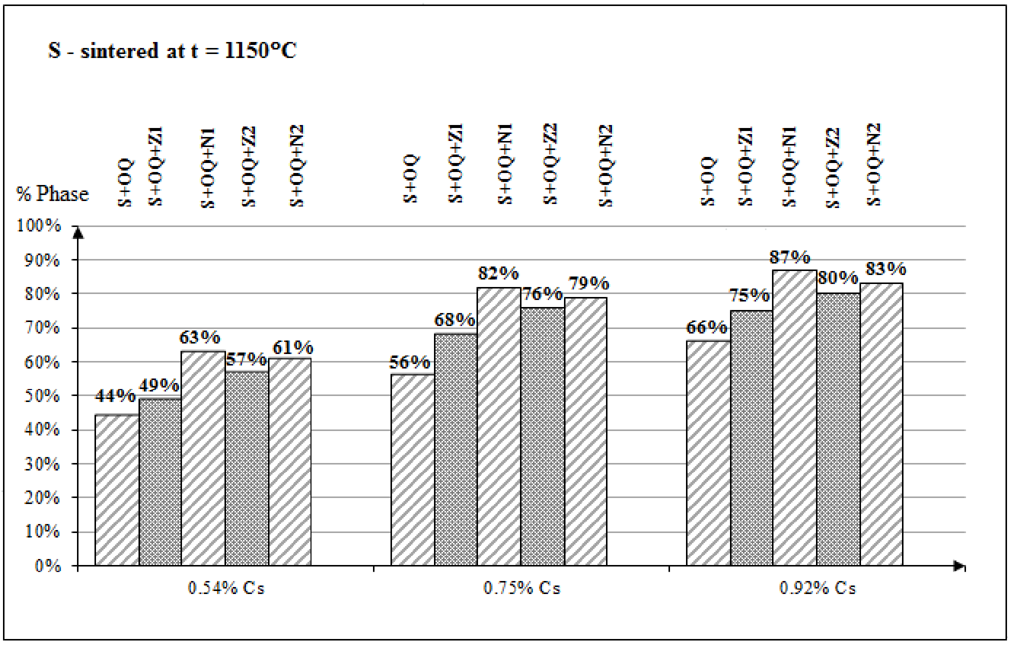


Fig.2. Percentages of hardening phases in sintered at 1150°C, hardened and sub-zero cooled steels

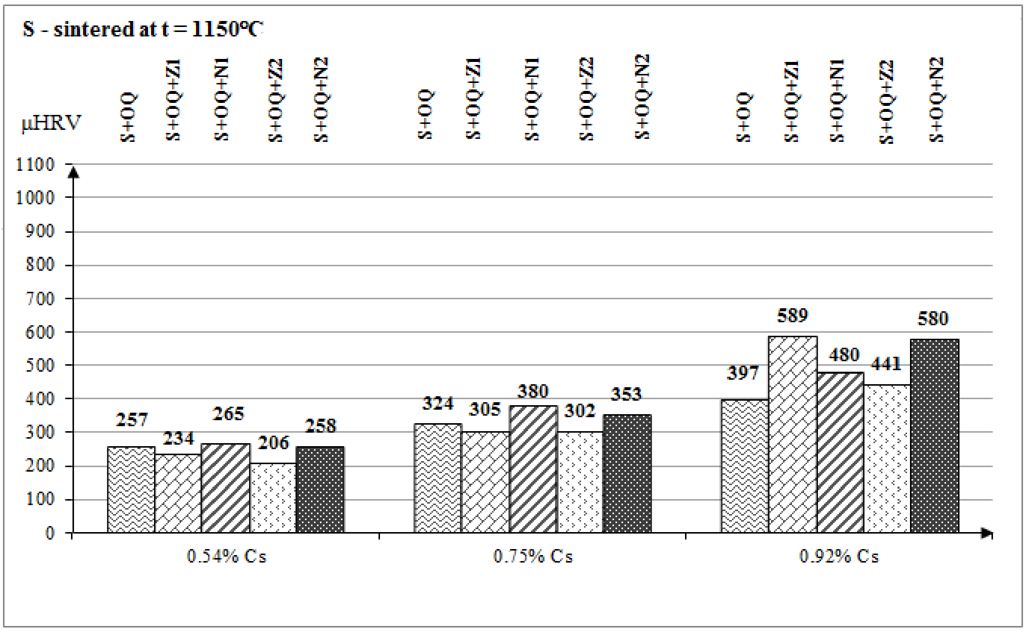


Fig.3. Micro hardness for the sintered at 1150 °C, hardened and sub-zero cooled steels

The behavior at the sub-zero treatment of the sintered steels depends on the content of the structural carbon (Cs). Analysing the content of martensite after treatment at 197 K (cooled in dry ice) and 77 K (deep cryogenic treatment) it is found the following:

- In the case of steels hipoeutectoide (C = 0.54% C) the treatment at high temperatures after quenching in oil do not have significant effects from the point of view of increase hardness although in case of this group of steels are produced the largest quantities of martensite through the transformation of residual austenite, but which has a low tetragonal martensite crystal structure.

- In the case of hipoeutectoid sintered steels the cooling at low temperature after quenching in oil has more noticeable effects with how is increasing content of structural carbon (Cs), as can be seen from Fig.2 and Fig.3 for steels with 0.75% Cs and 0.92% Cs.
- In terms of the influence of the sub-zero treatment parameters on the sintered steel samples and quenched in oil give the following: the best results are obtained at cooling in liquid nitrogen maintaining it for 60 min. ie in case of steels with Cs = 0.92% occurs increasing the content of martensite with 32% and respectively of hardness with 31.81%; in the case of the samples cooled in dry ice the best results are obtained for steels Cs = 0.92% maintained for 120 min and namely martensite content that increase by 25% and hardness to 21%.

4. CONCLUSIONS

In conclusion it can be said that sub-zero treatments applied to sintered steels after their tempering in oil are making felt its effects for contents of the structural carbon in the domain of the concentrations hipereutectoide.

If the cooling medium is deep cryogenic treatment is recommended a maintaining time reduced to 60 minutes, and if the cooling medium is the dry ice the maintaining times will be up to 120 min.

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