



DEEP CRYOGENIC TREATMENT OF P/M S390MC HIGH SPEED STEEL¹

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Abstract

Deep cryogenic treatment is claimed to be an efficient technique to improve the properties of tool and high speed steels. Sometimes the influence of subzero treatment could be directly ascribed to a specific metallurgical transformation. It is the case of the transformation of retained austenite into martensite, causing a general increase in hardness and higher wear resistance (but lower toughness!). In other cases, however, the increase in wear resistance is not supported by a higher hardness and a lot of theories were proposed to justify the observed results. However, poor experimental evidences were reported in literature for this phenomenon. Specific attention is paid to the influence of subzero treatment placed just after quenching and solubilization in the vacuum heat treatment or nitriding cycle of the P/M S390MC high speed steel, respectively. Special emphasis was put on abrasive wear resistance and resistance to galling under dry sliding conditions. Abrasive wear resistance was tested under reciprocating sliding conditions using alumina ball, while galling resistance against austenitic stainless steel was determined in a load-scanning test rig. From obtained results it can be concluded that the application of deep-cryogenic treatment results in significantly higher wear resistance of high speed steels, but no significant improvements in toughness have been noticed. **Key words:** High speed steel; Deep-cryogenic treatment; Hardness; Fracture toughness; Wear resistance; Nitriding.

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1 INTRODUCTION

In metal forming industry tools can be exposed to very complex and surface demanding conditions, which are the result of different effects (mechanical, thermal, chemical or tribological loading).^[1] Therefore tool materials need to fulfil many requirements, which, to a certain extent, are not mutually compatible, i.e. high hardness and high toughness. Beside the material's intrinsic properties, tribological properties of the tool surface, including abrasive wear resistance, coefficient of friction and resistance to galling, will also determine tool's operating lifetime. Using different treatment processes and parameters, the microstructure of a tool steel and therefore its mechanical and tribological properties can be modified and optimized for selected application.^[2] Bearing all the discussed things in mind, one easily realizes that general tool steel with the optimum properties' profile is very difficult to create. For that reason over the past few decades, extensive interest has been shown in the effect of ion nitriding and deep-cryogenic treatment on the performance of tool and high-speed steels.^[3-6] From this point of view the increase of performance, the diminishing of abrasive and adhesive wear as well as galling make the nitriding a valuable process for surface treatment.^[7-9] On the other hand, the low-temperature treatment is generally classified as either "cold treatment" at temperatures down to about -80°C (dry ice), or "deep-cryogenic treatment" at liquid nitrogen temperature of -196°C.^[10] Cryogenic treatment is not, as often mistaken for, a substitute for good heat treatment, bud supplemental process to heat treatment before tempering.^[7,10] As reported, the deep-cryogenic treatment has a lot of benefits. It not only gives dimensional stability to the material, but also improves abrasive^[10] and fatigue wear resistance.^[11] and increases strength and hardness of the material.^[9,12] The main reason for this improvement in properties are contributed to complete transformation of retained austenite into martensite and the precipitation of fine n-carbides into the tempered martensitic matrix.^[10,13] Numerous practical successes of cryogenic treatment and research projects have been reported worldwide.^[13] However, the treatment parameters including cooling rate, soaking temperature, soaking time, heating rate, tempering temperature and time need to be optimized with respect to the material and application. Furthermore, reported investigations were mainly focused on abrasive wear resistance, while resistance to galling still needs to be investigated. The aim of our work was to investigate the influence of ion nitriding and deep-cryogenic treatment parameters (treatment time and temperature) and austenizing temperature on the tribological performance of powder-metallurgy (P/M) high-speed steel with respect to abrasive wear resistance and resistance to galling under dry sliding conditions.

2 EXPERIMENTAL

2.1 Materials and Treatments

In this work, commercial P/M high-speed steel grade S390 Microclean from Böehler, delivered in the shape of rolled, soft annealed and piled bars was used. The steel had the following composition (in wt.%): 1.47% C, 0.54% Si, 0.29% Mn, 0.023% P, 0.014% S, 4.83% Cr, 1.89% Mo, 4.77% V, 10.05% W and 8.25% Co. Specimens in the shape of plates (Φ 20 mm x 9 mm) and rods (Φ 10 mm x 100 mm) were cut and machined from bars, surface polished (discs to Ra \approx 0.01 µm and cylinders to







Ra \approx 0.1 µm) and subsequently heat treated in a horizontal vacuum furnace with uniform high-pressure gas quenching using N_2 at a pressure of 5 bars. After the last preheat (1050°C) the specimens were heated (25°C/min) to the austenizing temperature of 1130°C and 1230°C, soaked for 6 min and 2 min, and gas guenched to 80°C, respectively (Table 1). The specimens were then either triple tempered for 2 h, or removed from the furnace for a subsequent deep-cryogenic treatment followed by a single tempering or ion nitriding for 2 h. The deep-cryogenic treatment of selected specimens (Table 1) was performed by a controlled immersion of the individual test specimens in liquid nitrogen for 25 and 40 h. respectively. Thermo chemical treatment of test specimens was performed in a Metaplas Ionon HZIW 600/1000 reactor equipped with a convection heating system and internal gas/water heat-exchanger for fast cooling. Pulse plasma nitriding at 520°C was applied using 3 hPa pressure and a total gas flow rate of 75 l/h. The gas atmosphere was 25%N₂-75%H₂. Heating to the process temperature took approximately 3 hours and the duration of treatment was 2 hours, Table 1.

2.2 Hardness and Fracture-toughness Tests

The Rockwell-C hardness (HRc) and Vickers hardness HV_{0.1} was measured on specimens in the shape of plates (\$\Phi20 mm x 9 mm) and rods (\$\Phi10 mm x 100 mm) using a Rockwell, B 2000 and Vickers, Tukon 2100 B, hardness machine. A semi-empirical model for estimating the fracture toughness, K_{lc} , of high-speed steels,^[14] where the fracture toughness of the high-speed steel is guantified on the basis of microstructural parameters and several other material properties was used to estimate the fracture toughness of the specimens in the shape of plates (Φ 20 mm x 9 mm):

$$\mathbf{K}_{\mathsf{lc}} = 1.363 \cdot \left(\frac{\mathsf{HRc}}{\mathsf{HRc} - 53}\right) \cdot \left[\sqrt{\mathsf{E} \cdot \mathsf{d}_{\mathsf{p}}} \cdot \left(\mathsf{f}_{\mathsf{c}}\right)^{\frac{1}{6}} \cdot \left(1 - \mathsf{f}_{\mathsf{c} \ge \mathsf{a}_{\mathsf{crit}}}\right) \cdot \left(1 + \mathsf{f}_{\mathsf{aust}}\right)\right]$$
(1)

The above correlation is a semi-empirical one, and it was derived by taking into account the critical strain criterion.^[15] the experimentally determined effects of the microstructural parameters and the hardness, and so it is necessary to take great care with the units. The constant, 1.363, was obtained by assuming that the modulus of elasticity, E, is expressed in MPa, the mean distance between undissolved eutectic carbides, d_p , in m, the Rockwell-C hardness in units of HRc, f_c and f_{aust} as volume fractions of undissolved eutectic carbides and retained austenite, and $f_{c \ge a_{max}}$ as the cumulative fraction of undissolved eutectic carbides and/or carbide clusters equal to or larger than the critical defect size ($\geq a_{crit}$). In this case the fracture toughness, K_{lc} , is obtained in units of MPa m^{1/2}. According to linear elastic fracture mechanics the critical defect size, a_{crit}, is exceeded when a decrease in the fracture strength is achieved.^[16]

Spec.	Auster	nizing	Deep-Cryogenic treatment		Tempering [°C/h]	Nitriding [°C/h]
	Temp.	Time	Temp. Immersion			
	[°C]	[min]	[°C]	time [h]		
A1	1130	6	/	/	540/540/510/2h	
A2	1130	6	/	/	540/540/2h	520/2h
A3	1130	6	-196	25	540/2h	
A4	1130	6	-196	25		520/2h
A5	1130	6	-196	40	540/2h	
A6	1130	6	-196	40		520/2h
B7	1230	2	/	/	540/540/510/2h	
B8	1230	2	/	/	540/540/h	520/2h
B9	1230	2	-196	25	540/2h	
B10	1230	2	-196	25		520/2h
B11	1230	2	-196	40	540/2h	
B12	1230	2	-196	40		520/2h

Table 1. Vacuum heat treatment and deep-cryogenic treatment parameters

2.3. Tribological Testing

Abrasive wear resistance of vacuum heat treated, deep-cryogen treated and nitrided P/M high-speed steel S390 Microclean was determined under reciprocating sliding conditions using ball-on-flat configuration (Fig.1a). In order to concentrate all the wear on the stationary high-speed steel specimens, alumina ball (ϕ 10 mm) was used as an oscillating counter-body. Abrasive wear tests were performed under dry sliding conditions at an average sliding speed of 0.02 m/s (frequency of 5 Hz and amplitude of 2.4 mm), a maximum Hertzian contact pressure of 1.3 GPa (F_N = 10 N) and total sliding distance of 30 m. Test results were evaluated in terms of P/M high-speed steel wear volume and average coefficient of friction under reciprocating sliding.

Galling resistance and ability of investigated S390 P/M high-speed steel to prevent transfer of work material was examined in a load-scanning test rig (Fig.1b), where cylindrical P/M high-speed steel specimens (ϕ 10x100 mm) were tested against quenched austenitic stainless steel (AISI 304, 150 HV₁₀, R_a \approx 0.1 µm). The test configuration, which involves two crossed cylinders that are forced to slide against each other under a constant speed, allows the normal load to gradually increase during testing.^[17] Thus, each point along the contact path of both specimens corresponds to a unique load and displays a unique tribological history after test completion. During testing, performed under dry sliding conditions at a sliding speed of 0.01 m/s, normal load was gradually increased from 100 to 1300 N. Galling properties were determined by monitoring coefficient of friction as a function of load, and by examining contact surfaces after sliding and defining critical loads for galling initiation and stainless steel transfer layer formation.

Prior to tribological testing, performed at room temperature (21±1°C) and relative humidity of 50%, all specimens were ultrasonically cleaned in ethanol and dried in air.



Figure 1. Test setup; (a) ball-on-disc and (b) load-scanner.

3. RESULTS AND DISCUSSION

3.1. Surface Analyses

Microstructures of investigated P/M high-speed steel specimens (Table 1) are shown in Fig. 2, and surface hardness and roughness values collected in Table 2.

After vacuum heat treatment from the austenizing temperature of 1130°C and triple tempering (Specimens, A1 and B7) a microstructure without proeutectoid carbides precipitated on the prior austenite grain boundaries was obtained. In the matrix of tempered martensite fine globular undissolved eutectic carbides of the types MC (grey or black) and M₆C (white) are uniformly distributed (Fig. 2a), showing the mean size of about 1.2 μ m.

By performing deep-cryogenic treatment (Specimens A3 and A5), similar microstructure with tempered martensite and fine globular undissolved eutectic carbides can be observed. However, as compared to triple tempered specimen, cryogenic treatment of 25 h and 40 h results in finer needles-like martensitic microstructure (Fig. 2b) and surface hardness increase from 66.8 to 67.1 HRc. Longer cryogenic treatment time also leads to fine needles-like microstructure and higher hardness (Table 2).

For specimens vacuum heat treated from the austenizing temperature of 1230° C (Specimens, B9 and B11) similar fine martensitic microstructure without proeutectoid carbides precipitated on the prior austenite grain boundaries and uniformly distributed fine globular undissolved eutectic carbides was obtained. However, as compared to austenizing temperature of 1130° C, higher austenizing temperature leads to smaller amount of undissolved eutectic carbides with the mean size less than 1 µm (Fig. 2d) and higher surface roughness (Table 2). After triple tempering surface hardness is slightly lower compared to austenizing temperature of 1130° C, but combination of higher austenizing temperature and deep-cryogenic treatment results in increased surface hardness, as shown in Table 2.









Figure 2. Typical microstructure of vacuum heat treated, deep-cryogenic treated and nitrided S390 P/M high-speed steel specimens: a-c) specimens A1 to A6; d-f) specimens B7 to B12.

The microstructure of the specimens, A4, A6, B10 and B12, which were deep cryogenic treated after austenitization in liquid nitrogen for 25 and 40 hours followed by simultaneous tempering and ion nitriding, consists of the diffusion layer to a depth of ~ 65 μ m. On the surface of the specimens even at higher magnifications we did not observed γ' compound layer or cracks. Crystal prior austenite grain boundaries in the diffusion layers are distinct, as a result of an increased etching effect because of the presence of large residual stresses after nitriding. The microstructure in the core of the specimens is similar with previous specimens.





3.2 Hardness and Fracture-toughness

Rockwell-C hardness of the core of the specimens and calculated fracture toughness values according to equation (1) are shown in Fig 3. Surface hardness HV_{0.1} and roughness values after polishing of vacuum heat treated, deep-cryogenic treated and nitrided specimens are shown in Table 2. As it can be seen from the diagram 3 estimated fracture toughness values for the non nitrided specimen's austenizet at 1130°C are lower in comparison to the nitrided specimens. This is due to the higher hardness of the bulk material of the first one. In the case of nitrided specimens B7 and B8 as compared to non nitrided specimens austenized from the same temperature, can be also ascribed to the lower hardness of the bulk material. The relationship between Rockwell-C hardness, and related fracture toughness of the specimens in the shape of plates (Φ 20 mm x 9 mm) is similar to those for rods (Φ 10 mm x 100 mm), Fig 3. However, the relationship between Rockwell-C hardness, microstructure and related fracture toughness will be the object of the future investigation.



Figure 3. Hardness of core of the specimens and calculated fracture toughness according to equation(1).





Table 2. Surface hardness and roughness values after vacuum heat treatment, deep-cryogenic treatment and nitriding.

Spec.	Hardness-plates		Hardness-cylinders		Roughness – plates		Roughness – cylinders	
	φ20x9 mm		φ10x100 mm		φ20x9 mm		φ10x100 mm	
	HRc	HV _{0.1}	HRc	HV _{0.1}	R _a [µm]	R _{max} [µm]	R _a [µm]	R _{max} [µm]
A1	66.4	976	66.8	981	0.025±0.001	0.22±0.02	0.10±0.009	1.07±0.28
A2	67.0	1342	64.6	1338	0.028±0.002	0.30±0.08	0.10±0.015	1.03±0.19
A3	66.8	962	67	993	0.036±0.003	0.41±0.01	0.11±0.015	1.09±0.11
A4	66.8	1371	64.1	1364	0.027±0.002	0.26±0.05	0.11±0.005	1.10±0.38
A5	67.8	967	67.1	991	0.024±0.001	0.23±0.03	0.11±0.007	1.09±0.20
A6	68.1	1317	64.6	1342	0.029±0.000	0.42±0.07	0.10±0.008	1.12±0.17
B7	63.4	945	66.7	974	0.111±0.023	0.77±0.11	0.09±0.002	1.03±0.41
B8	63.4	1387	66.6	1383	0.127±0.002	1.37±0.24	010±0.016	0.78±0.02
B9	63.3	959	68.4	1007	0.066±0.001	0.59±0.06	0.07±.,008	1.02±0.33
B10	65.9	1408	69.3	1428	0.040±0.002	0.38±0.09	0.10±0.007	1.11±0.17
B11	68.8	1009	68.5	1029	0.049±0.007	0.53±0.17	0.06±0.000	0.73±0.04
B12	68.0	1403	68.9	1401	0.064±0.022	0.74±0.40	0.10±0.019	1.01±0.08

3.3 Abrasive Wear Resistance

When tested against alumina ball, vacuum heat treated S390 P/M high-speed steel shows an average coefficient of friction in the range of 0.75 to 0.80 and wear rate between 1.8 and $2.2 \cdot 10^{-6}$ mm³/Nm, with higher austenizing temperature leading to higher friction and higher wear rates. On the other hand deep-cryogenic treatment does not change friction behaviour and abrasive wear resistance of S390 P/M high-speed steel considerably. However, longer cryogenic treatment time (40 h) tends to give lower average coefficient of friction and lower wear rate, with difference being less than 10% as compared to vacuum heat treated and triple tempered specimens (Fig. 3). In the case of nitrided specimens substantially lower average coefficient of friction and lower wear rate in comparison to non nitrided ones can be observed.



Figure 3. Average coefficient of friction and abrasive wear rate for investigated S390 P/M high-speed steel specimens tested against alumina ball.





3.4 Galling Resistance

Galling resistance and ability of vacuum heat treated, deep-cryogenic treated and nitrided S390 P/M high-speed steel to prevent transfer of stainless steel, determined by monitoring coefficient of friction as a function of load is shown in Fig. 4.



Figure 4. Coefficient of friction vs. normal load for vacuum heat treated and deep-cryogenic treated S390 P/M high-speed steel, recorded during sliding against stainless steel.

In the case of vacuum heat treated and deep-cryogenic treated and triple or single tempered specimens (ϕ 10x100 mm) A1, A3, A5, B7, B9 and B11 the initial coefficient of friction varies between 0.05 and 0.2. The first sign of adhesion of stainless steel to the P/M high-speed steel surface, as indicated by sudden increase in friction and confirmed by post-test microscopic observation was detected at about 200 N load, and a building-up of a layer of transferred stainless steel material above 350 N load.

Deep-cryogenic treatment (Specimens (ϕ 10x100 mm) A3 and A5) gives similar friction behaviour as observed for specimen A1 but improved galling resistance, with critical loads for galling initiation and transfer layer build-up being increased to about 210 N and 390 N, respectively. No mayor differences can be observed between specimen A3 and A5, as shown in Fig. 5.

By increasing austenizing temperature form 1130°C to 1230°C (Specimens B7, B9 and B11) initial friction against stainless steel has been reduced to the level of 0.1, but also critical load for the beginning of stainless steel transfer has slightly dropped for triple tempered specimen B7. As shown in Figs. 4 and 5, deep-cryogenic treatment does not give any improvement in galling resistance when austenizing temperature is 1230°C.

Increase in austenizing temperature leads to lower surface hardness due to reduces number of globular undissolved eutectic carbides and therefore to reduced abrasive wear resistance of S390 P/M high-speed steel (Fig. 3). Furthermore, it also increases surface roughness, which together with increased area of softer matrix results in reduced galling resistance against stainless steel (Fig. 5). On the other hand, vacuum heat treatment







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followed by deep-cryogenic treatment gives finer microstructure with higher surface hardness and consequently lower wear rate. Longer the cryogenic treatment time higher hardness and better wear resistance we can expect (Fig. 3). For austenizing temperature of 1130°C deep-cryogenic treatment will also result in greatly improved galling resistance against stainless steel, which can be even up to 100%. However, when using austenizing temperature of 1230°C subsequent deep-cryogenic treatment seams to have no influence on galling properties of S390 P/M high-speed steel, as shown in Fig. 5.

In the case of vacuum heat treated and deep-cryogenic treated and nitrided specimens A2, A4, A6, B8, B10 and B12 the initial coefficient of friction varies between 0.05 and 0.1. The analysis shows that on the critical load L_{c1} , more than roughness and deep-cryogenic treatment in liquid nitrogen, influence austenizing temperature, which has a significant impact on the volume fraction of undissolved eutectic carbides. In addition the plasma nitriding in both groups of cylindrical specimens lowers the coefficient of friction and gives better adhesion resistance of the surface, i.e. increases the critical load L_{c1}. The first signs of adhesion of austenitic steel on contact area of cylindrical specimens, as indicated by a sudden increase in coefficient of friction and confirmed on the contact surface by microscopy, were observed at loads between 230 and 370 N.



Figure 5. Critical loads for the beginning of stainless steel transfer (L_{c1}) and building-up of a stainless steel layer (L_{c2}) on vacuum heat treated and deep-cryogenic treated S390 P/M high-speed steel.

4 CONCLUSIONS

Deep-cryogenic treatment improves microstructure of P/M high-speed steel in terms of producing finer needles-like martensitic structure. Finer microstructure results in higher surface hardness and better wear properties, both abrasive wear resistance and galling resistance against stainless steel. Longer cryogenic treatment times will result in finer







microstructure and higher surface hardness, however, this has limited effect on tribological properties.

On the other hand, selection of the proper austenizing temperature is much more important. Increase in austenizing temperature will lead to reduced amount of carbides and increased surface roughness, resulting in reduced tribological properties. Furthermore, subsequent deep-cryogenic treatment might loose its beneficial effects if austenizing temperature is too high.

Main processing innovation is the utilization of a combined vacuum hardening and deep-cryogenic treatment followed by single tempering or nitridig (simultaneous tempering and nitrogen diffusion) to produce the desire metallurgical transformations of P/M highspeed steel. In case of nitriding, high residual stresses at the same time also promote nitrogen diffusion along the steel surface layer. On nitrided specimens, an increase of the resistance to adhesive and abrasive wear of the outer surface layer was observed. A detail study of the deep-cryogenic treatment before nitriding on the delamination behaviour and resistance to guenched austenitic stainless steel chemical attack of nitrided layer was studied and will be published in separate paper. Thereafter, important economical benefits could be derived from the reduction of maintenance and dead times of dies, decreasing of dies cost and increasing of forming speeds due to the enhancement of die performance. Rapid implementation of this crvo-based technology is possible due to the reduced cost of existing cryogenic equipments and the required liquid nitrogen consumption.

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