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Improving tribological properties of tool steels through combination of deep-cryogenic treatment and plasma nitriding

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ABSTRACT

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) and require well defined mechanical and especially tribological properties. The aim of the present work was to investigate the effect of deep-cryogenic treatment parameters (treatment time and temperature) in combination with plasma nitriding on the tribological performance of powder–metallurgy (P/M) high-speed steel. Special emphasis was put on abrasive wear resistance and resistance to galling under dry sliding conditions. Test results show that deep-cryogenic treatment contributes to improved abrasive wear resistance and better galling properties of P/M high-speed steel. Selection of the proper austenizing temperature is also an important factor, with higher austenizing temperature resulting in higher friction and wear. Plasma nitriding gives excellent tribological properties of P/M high-speed steel and reduces the effect of austenizing temperature. However, if combined with deep-cryogenic treatment it eliminates beneficial effect of deep-cryogenic treatment.

1. Introduction

In the 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 the tool's operating lifetime. Using different heat treatment processes and parameters, the microstructure of a tool steel and therefore its mechanical and tribological properties can be modified and optimized for a selected application [2].

Over the past few decades, extensive interest has been shown in the effect of low-temperature treatment on the performance of tool steels [3–5]. Low-temperature treatment is generally classified as either "cold treatment" at temperatures down to about $-80\,^{\circ}$C (dry ice), or "deep-cryogenic treatment" at liquid nitrogen temperature of $-196\,^{\circ}$C [6]. Cryogenic treatment is not, as often mistaken for, a substitute for good heat treatment, but is a supplemental process to conventional heat treatment before tempering [4,6]. As reported, the deep-cryogenic treatment has many benefits. It not only gives dimensional stability to the material, but also improves abrasive [3,5,6] and fatigue wear resistance [7], and increases strength and hardness of the material [5,8]. The main reason for this improvement in properties are the complete transformation of retained austenite into martensite and the precipitation of fine η-carbides into the tempered martensitic matrix [6,9]. Till now numerous practical successes of cryogenic treatment and research projects have been reported worldwide [2–9]. 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.

Another way of modifying the contact surface, already effectively used in the forming industry to improve wear resistance of the tools, is thermo-chemical surface treatment, i.e. carburizing, carbonitriding, nitriding, etc. [10,11]. Especially by plasma nitriding surface layers of exceptional tribological properties can be formed, which exhibit low friction and improved wear resistance [12–14]. Furthermore, plasma nitrided tool steels may also show reduced tendency to pick-up work material [15,16]. Tested against aluminium and titanium alloys plasma nitrided VANADIS 4

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Table 1
Vacuum heat treatment, deep-cryogenic treatment and plasma nitriding parameters.

Spec.	Austenizing		Deep-cryogenic treatment		Tempering [°C/h]	Plasma nitriding [°C/h]
	Temp. [°C]	Time [min]	Temp. [°C]	Immersion time [h]		
A1	1130	6	–	–	540/540/510/2	–
A2	1130	6	–	–	540/540/2	520/2
A3	1130	6	−196	25	540/2	–
A4	1130	6	−196	25	–	520/2
A5	1130	6	−196	40	540/2	–
A6	1130	6	−196	40	–	520/2
B1	1230	2	–	–	540/540/510/2	–
B2	1230	2	–	–	540/540/2	520/2
B3	1230	2	−196	25	540/2	–
B4	1230	2	−196	25	–	520/2
B5	1230	2	−196	40	540/2	–
B6	1230	2	−196	40	–	520/2

tool steel outperformed PVD coated one, giving lower and more stable friction and better protection against galling [17]. Therefore, by combining deep-cryogenic treatment and plasma nitriding, individual effects could be combined, resulting in greatly improved mechanical and tribological properties of forming tools.

The aim of the present work was to investigate the effect of deep-cryogenic treatment parameters (treatment time and temperature) and combination of deep-cryogenic treatment and plasma nitriding on the tribological performance of powder–metallurgy (P/M) high-speed steel with respect to wear resistance and resistance to galling under dry sliding conditions.

2. Experimental

2.1. Materials and treatments

Commercial P/M high-speed steel grade S390 Microclean from Boehler with 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 was used in the present investigation. The specimens in the shape of discs (ϕ 20 mm × 9 mm) and rods (ϕ 10 mm × 100 mm) were cut and machined from rolled and soft annealed bars, surface polished to $R_a \approx 0.1\ \mu$m, and subsequently heat treated in a horizontal vacuum furnace with uniform high-pressure gas quenching using N_2 at a pressure of 5 bar. After the last preheat (1050 °C) the specimens were heated (25 °C/min) to austenizing temperatures of 1130 °C and 1230 °C, soaked for 6 min and 2 min, and gas quenched to 80 °C, respectively (Table 1). The specimens were then either triple tempered for 2 h at 540 °C and 510 °C, double tempered for 2 h at 540 °C followed by plasma nitriding for 2 h at 520 °C, deep-cryogenic treated with subsequent single tempering, or deep-cryogenic treated and plasma nitrided. Conventional heat treatment of high-speed steels involves three tempering stages, with the first two stages used to transfer retained austenite into martensite and the last one for residual stress relaxation [18]. When combined with plasma nitriding, plasma nitriding besides forming diffusion layer will also act as a thermal stress relaxation stage. Therefore, only double tempering is required prior plasma nitriding to transfer retained austenite into martensite. However, in the case of deep cryogenic treatment almost all retained austenite is transformed into martensite [6,9] and therefore only stress relaxation, either by single tempering or plasma nitriding is required. After surface treatment all test specimens were re-polished to an average roughness value of ~0.1 μm.

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. Plasma nitriding was carried out in a Metaplas Ionon HZIW 600/1000 reactor. In order to prevent compound layer formation process temperature of 520 °C, nitriding time of 2 h, total pressure of 3.3 hPa and gas mixture of 95 vol.% H_2 : 5 vol.% N_2 was used. Austenizing temperatures and soaking times, deep-cryogenic treatment times, tempering temperatures and times, and nitriding temperatures and times are given in Table 1.

2.2. Hardness measurement

The Rockwell-C hardness (HRc) and Vickers hardness $HV_{0.1}$ were measured on the surface of heat treated disc specimens (ϕ 20 mm × 9 mm) using a Rockwell, B 2000 and Vickers, Tukon 2100 B, hardness machines. On each specimen up to 10 measurements were performed across the surface in order to obtain representative average hardness value.

2.3. Tribological testing

Wear resistance of surface treated P/M high-speed steel S390 specimens (Table 1) was determined under reciprocating sliding motion using ball-on-flat contact configuration. In order to simulate two-body abrasive wear and to concentrate all the wear on the stationary high-speed steel samples, an alumina ball (ϕ 10 mm) was used as an oscillating counter-body. Wear tests, aimed at evaluating the effect of deep cryogenic treatment and plasma nitriding on abrasive wear resistance of tool steels 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 steady-state coefficient of friction.

Galling resistance and the ability of surface treated S390 P/M high-speed steel to prevent transfer of work material was examined in a load-scanning test rig. The test configuration, where tempered austenitic stainless steel (ASS) cylinder (AISI 304, 350 HV, $R_a \approx 0.1\ \mu$m, ϕ 10 mm) was forced to slide against surface treated P/M high-speed steel cylinder (ϕ 10 mm) at a constant sliding speed, allows the normal load to gradually increase during testing, with each point along the contact path of both specimens corresponding to a specific load [19,20]. Galling tests were performed under dry sliding conditions at a sliding speed of 0.01 m/s and normal load in a range of 50–600 N (pH = 2.8–6.4 GPa). Galling properties of surface treated specimens 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 ASS 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.

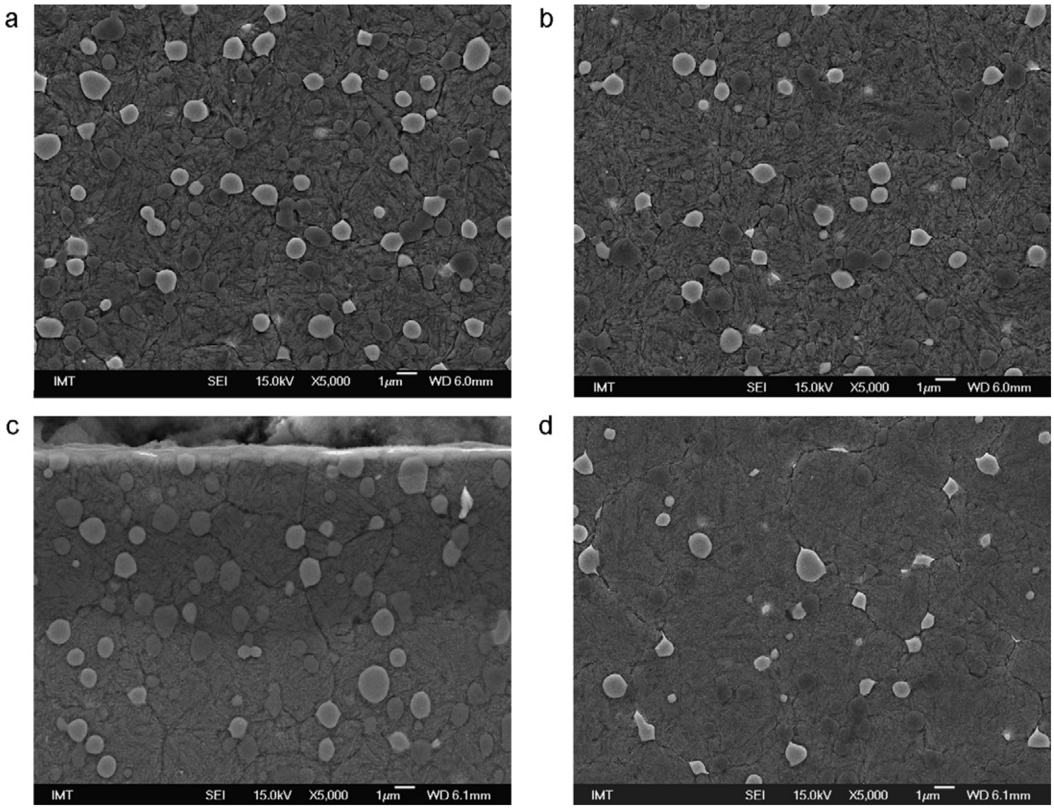

Fig. 1. Microstructure of vacuum heat treated, deep-cryogenic treated and plasma nitrided S390 P/M high-speed steel specimens: (a) A1, (b) A3, (c) A4 and (d) B1.

3. Results and discussion

3.1. Surface analysis

Microstructures of investigated P/M high-speed steel samples (Table 1) are shown in Fig. 1, and surface hardness values collected in Table 2. After vacuum heat treatment from the austenizing temperature of 1130 °C and triple tempering (Specimen A1) 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 types MC (grey or black) and M_6C (white) are uniformly distributed (Fig. 1a), showing a mean size of about 1.2 μm [21].

By performing deep-cryogenic treatment (Specimens A3 and A5), a similar microstructure with tempered martensite and fine globular undissolved eutectic carbides can be observed. However, as compared to the triple tempered specimen, cryogenic treatment of 25 h results in almost complete transformation of retained austenite into martensite, a finer needle-like martensitic microstructure (Fig. 1b) [22–24] and a surface hardness increase from 66.8 to 67.0 HRc. Longer cryogenic treatment time gives similar but even finer microstructure, and increased hardness (Table 2) [24,25].

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

Specimen	Hardness	
	HRc	$HV_{0.1}$
A1	66.8	964 ± 17
A2		1338 ± 41
A3	67.0	966 ± 27
A4		1364 ± 46
A5	67.1	972 ± 19
A6		1342 ± 12
B1	66.7	952 ± 22
B2		1383 ± 25
B3	68.4	984 ± 23
B4		1428 ± 44
B5	68.5	1005 ± 23
B6		1401 ± 38

After plasma nitriding, using parameters typically found in forming tool applications, microstructure of the investigated steel consists of the diffusion layer to a depth of ~65 μm, without any compound layer or cracks being observed on the surface. However, as a result of an increased etching effect due to large residual stresses introduced by nitriding distinct prior austenite grain boundaries can be seen in the diffusion layer (Fig. 1c). Furthermore, nitriding increased surface hardness from 965 $HV_{0.1}$ to 1340 $HV_{0.1}$. Combining deep-cryogenic treatment and plasma nitriding had no effect on the material microstructure below the diffusion zone or surface hardness (Table 2).

For specimens vacuum heat treated from the austenizing temperature of 1230 °C (Specimens B1–B3) 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 an austenizing temperature of 1130 °C, a higher austenizing temperature leads to a smaller amount of undissolved eutectic carbides with the mean size less than 1 μm (Fig. 1d) [24]. After triple tempering surface hardness is slightly lower compared to an austenizing temperature of 1130 °C, but a combination of higher austenizing temperature, deep-cryogenic treatment and/or plasma nitriding results in increased surface hardness, as shown in Table 2.

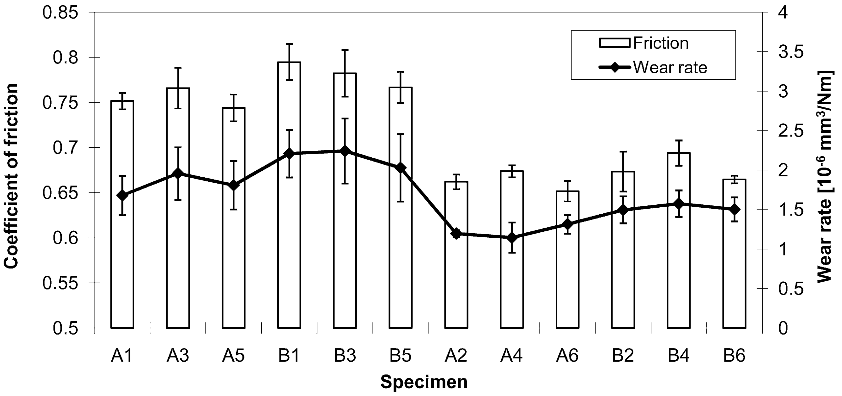

Fig. 2. Average coefficient of friction and wear rate for investigated S390 P/M high-speed steel specimens tested against alumina ball.

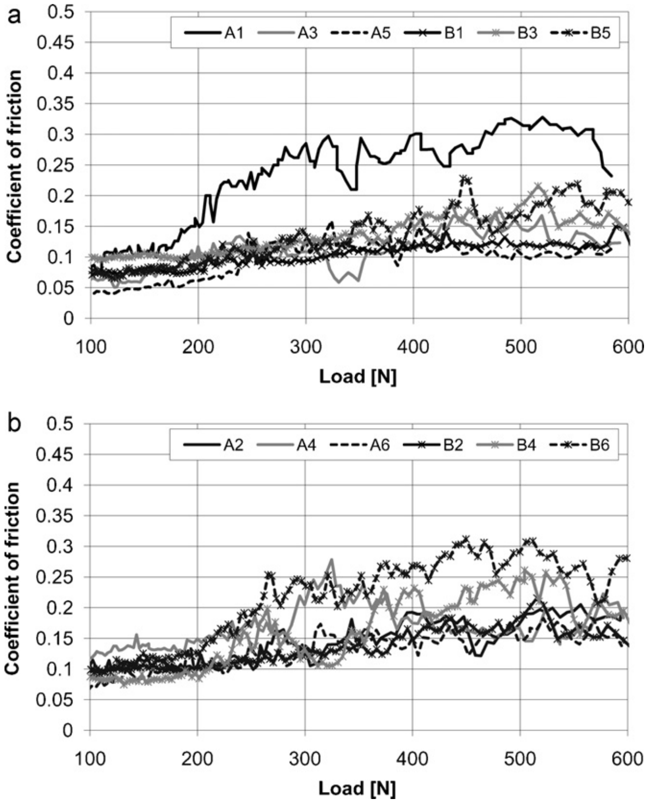

Fig. 3. Coefficient of friction vs. normal load for (a) vacuum heat treated and (b) plasma nitride S390 P/M high-speed steel, recorded during sliding against ASS.

3.2. Wear resistance

Wear test results for heat treated and plasma nitrided S390 P/M high-speed steel are shown in Fig. 2. In the case of vacuum heat treated and triple tempered S390 P/M high-speed steel (A1 and B1) average coefficient of friction against alumina ball was in the range of 0.75–0.80 and wear rate between 1.7 and $2.2 \times 10^{-6}\,\mathrm{mm^3/Nm}$. Higher austenizing temperature resulted in a microstructure with a smaller amount and size of undissolved eutectic carbides, thus reducing hard phase volume fraction in the softer matrix and leading to higher friction and higher wear rates. On the other hand deep-cryogenic treatment (A3, A5, B3, B5) with similar microstructure but almost complete transformation of retained austenite into martensite does not change friction and abrasive wear resistance of S390 P/M high-speed steel considerably. However, through the formation of finer needle-like martensitic microstructure with higher bulk hardness longer cryogenic treatment time (40 h) tends to give lower average coefficient of friction and lower wear rate. However, the difference was less than 10% as compared to vacuum heat treated and triple tempered specimens (Fig. 2). On the other hand plasma nitriding of S390 P/M high-speed steel markedly reduced its friction and wear. Through nitrogen diffusion layer formation and surface hardness increase [26] plasma nitriding reduced coefficient of friction of investigated steel to ~0.65 and wear rate even down to $1.1 \times 10^{-6}\,\mathrm{mm^3/Nm}$ (Fig. 2). Compared to non-nitrided specimens, plasma nitriding reduced the negative effect of higher austenizing temperature on the tribological properties of S390 P/M high-speed steel. However, although the difference is smaller higher austenizing temperature with reduced undissolved carbides volume fraction still results in higher friction and wear. On the other hand combination of deep-cryogenic treatment and plasma nitriding did not show any further improvement, as indicated in Fig. 2.

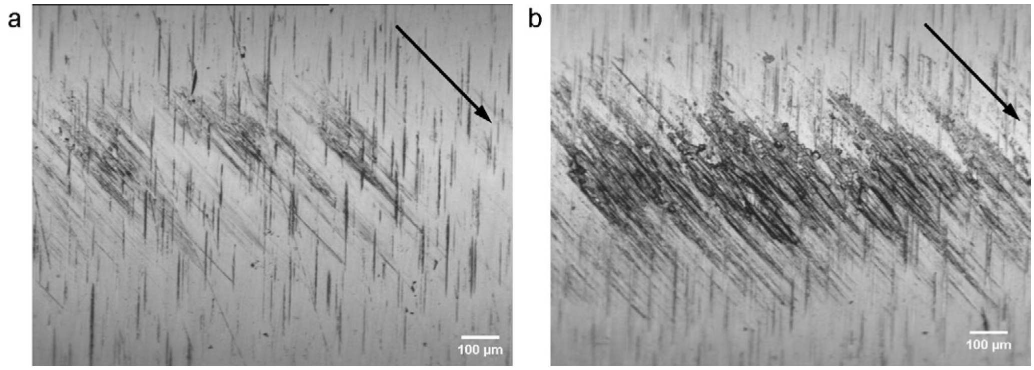

Fig. 4. Example of (a) galling initiation and (b) ASS layer build up on vacuum heat treated S390 P/M high-speed steel surface; arrows indicate direction of sliding.

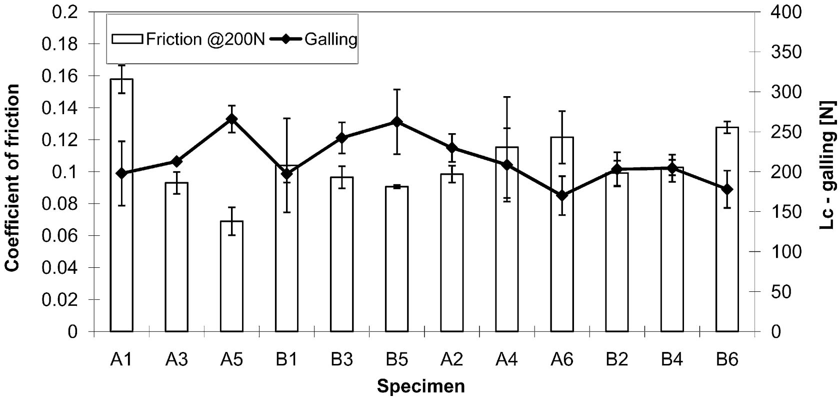

Fig. 5. Initial coefficient of friction and critical load for galling initiation for investigated S390 P/M high-speed steel specimens tested against austenitic stainless steel.

3.3. *Galling resistance*

Galling resistance and ability of vacuum heat treated, deep-cryogenic treated and/or plasma nitrided S390 P/M high-speed steel to prevent transfer of ASS, determined by monitoring the coefficient of friction as a function of load is shown in Fig. 3. In the case of polished vacuum heat treated and triple tempered specimen A1 the initial coefficient of friction varies between 0.10 and 0.15. The first sign of adhesion of ASS to the P/M high-speed steel surface, as indicated by sudden increase in friction and confirmed by post-test microscopic observation (Fig. 4a) was detected at about 200 N load, and a building-up of a layer of transferred ASS material (Fig. 4b) above 350 N load.

Through almost complete transformation of retained austenite into martensite, fine needle-like martensitic microstructure and formation of η-carbides [25,27] deep-cryogenic treatment (Specimens A3 and A5) leads to lower initial friction (<0.1) and improved galling resistance. Critical loads for galling initiation and transfer layer build-up have been increased to about 210 N and 390 N for specimen A3 and even up to 260 N and 470 N for longer deep-cryogenic treatment time of 40 h (A5), as shown in Figs. 5 and 6. Longer deep-cryogenic treatment times lead to increased density of lattice defects (dislocations) which act as nuclei for the formation of very fine η-carbides [25,27] and thus promote their formation. Improved galling properties of specimens deep-cryogenic treated for 40 h could then be related to increased density of very fine η-carbide clusters, with fine carbides normally showing better galling resistance than a metallic matrix [28]. By increasing austenizing temperature from 1130 °C to 1230 °C initial friction against ASS has been reduced to about 0.10, however, critical load for the beginning of ASS transfer remained the same (~200 N) for the triple tempered specimen (B1). As for the lower austenizing temperature, subsequent deep-cryogenic treatment led to reduced initial friction and better galling resistance, with longer deep-cryogenic treatment times again giving better results, which can be related to the finer needle-like martensitic microstructure. However, when combined with deep-cryogenic treatment higher austenizing temperature will in general result in higher hardness of the matrix and higher friction against ASS.

As expected, plasma nitriding of vacuum heat treated S390 P/M high-speed steel, if polished after nitriding, gave lower friction and better protection against galling. As compared to non-nitrided specimen A1, plasma nitriding (A2) increased critical load for galling initiation to 230 N and critical load for transfer layer build up to 370 N. However, combination of deep-cryogenic treatment and plasma nitriding (A4, A6, B4, B6) resulted in increased friction and reduced galling resistance, with the reasons behind not being clear at the moment. Furthermore, longer deep-cryogenic treatment times lead to less favourable tribological behaviour, as shown in Figs. 5 and 6.

Increase in austenizing temperature (A1 → B1) leads to lower surface hardness due to a reduced volume fraction of globular undissolved eutectic carbides and therefore to reduced abrasive wear resistance and higher friction of S390 P/M high-speed steel (Fig. 2), but still similar galling resistance (Fig. 6). However, as shown in [22] increase in austenizing temperature also causes increase in surface roughness, which if not re-polished will result in reduced galling resistance. On the other hand, vacuum

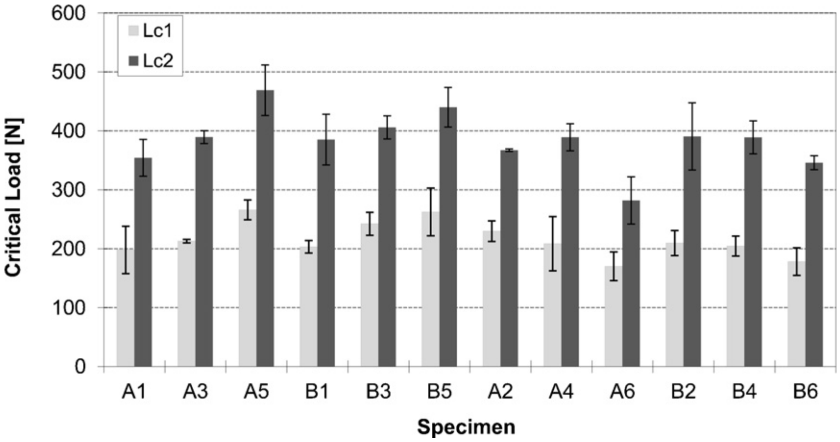

Fig. 6. Critical loads for the beginning of stainless steel transfer (Lc_1) and building-up of a stainless steel layer (Lc_2) on vacuum heat treated, deep-cryogenic treated and/or plasma nitride S390 P/M high-speed steel.

heat treatment followed by deep-cryogenic treatment results in almost complete transformation of retained austenite into martensite and in a finer microstructure with higher surface hardness and consequently slightly better wear resistance but much better galling protection. Increase in cryogenic treatment time (A3 → A5, B3 → B5), although giving higher hardness has practically no effect on S390 P/M high-speed steel wear resistance but due to even finer microstructure with higher density of η-carbide clusters it further improves its galling resistance. Through the formation of a low friction diffusion zone with high hardness plasma nitriding greatly improves friction, galling and wear resistance of vacuum heat treated S390 P/M high-speed steel (A1 → A2). However, if combined with deep-cryogenic treatment, benefits in galling resistance obtained through deep-cryogenic treatment will be jeopardized by the formation of diffusion zone, even resulting in markedly lower critical loads for galling initiation (Figs. 5 and 6).

4. Conclusions

Deep-cryogenic treatment improves microstructure of P/M high-speed steel by producing finer needle-like martensitic structure. Finer martensitic microstructure results in higher surface hardness and better tribological properties, especially in terms of friction and galling resistance against stainless steel. Longer cryogenic treatment times will result in even finer microstructure and higher surface hardness, which has no effect on abrasive wear resistance but it considerably improves galling properties.

Selection of the proper austenizing temperature is also an important factor. Increasing the austenizing temperature will lead to reduced volume fraction of carbides and consequently to increased friction and wear. On the other hand, it shows no effect in terms of galling resistance if surfaces are re-polished after treatment.

Plasma nitriding improves tribological properties of P/M high-speed steel and reduces the effect of austenizing temperature. However, if combined with deep-cryogenic treatment it might eliminate the beneficial effect of deep-cryogenic treatment, even leading to reduced galling resistance, with the reasons behind not being clear at the moment.

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