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Influence of microstructure on the sliding wear behavior of nitrocarburized tool steels

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ABSTRACT

The wear mechanisms during dry sliding of two representative tool steel grades, one of lower and one of higher percentage of carbide-forming elements—AISI H13 hot work and Cr–Mo–V cold work steels respectively—were investigated. Prior to testing, both grades were properly subjected to heat treatments aiming to their hardening up to final values of 40, 45 and 50 HRC. After heat treatments, a part of the specimens from each hardness level was subsequently surface treated via liquid nitrocarburizing (Tufftriding), under typical industrial processing conditions. The sliding friction behavior of all specimens was studied on a ball-on-disc apparatus, applying normal loads in the range of 1–10 N. The continuous recording of the friction coefficient, the measurement of the wear volume, together with the post-testing microscopic observation of the wear surfaces provided for the evaluation of material removal mechanisms and of the effects of prior heat and surface treatments on the response of the material under surface loading. It was found that the dry friction coefficient remained practically constant (0.80–0.95) for all specimens. The hardening heat treatment did not affect substantially the wear coefficient, in contrast to the surface treatment that resulted in its significant reduction. Finally, in the case of the grade with the higher percentage of carbide-forming elements, it was found that both the wear coefficient and related mechanisms are strongly affected by the presence of coarse chromium carbides within the metallic matrix.

1. Introduction

Liquid nitrocarburizing is an industrial surface treatment process employed for the improvement of wear/friction and fatigue resistance of steels, since it is associated with high dimensional accuracy required in high precision tooling. Compared to conventional gas-nitriding, the liquid nitrocarburizing technique, especially Tufftriding [1] which involves treatment of metallic components in molten cyanide salt baths at 580 °C, is faster and produces nitride layers of higher wear/corrosion resistance and enhanced fatigue strength and toughness [2–5]. Although Tufftriding is less expensive and conceptually simpler than other non-conventional nitriding techniques such as plasma-, ion- and fluidized bed-nitriding [6–9], is, however, burdened by post-processing cleaning steps targeted on the one hand to the neutralization of the depleted cyanide baths and on the other hand to the removal of the bath residues from the surface of the treated components. The first target is reached by initial conversion of cyanide (CN^-) to cyanate (CNO^-) ions, and subsequent neutralization of the latter, traditionally via alkaline chlorination [10]. The second goal is achieved by either chemical cleaning via immersion in acid solutions (pickling), or mechanical removal, commonly glass-blasting.

During liquid nitrocarburizing the temperature is kept under the eutectoid point (591 °C) of the Fe–N phase diagram, allowing simultaneous diffusion of nitrogen and carbon atoms into the ferrite lattice. This results in the formation of two distinct surface layers with different nitrogen concentration, which constitute the so-called nitrocarburized case, varying in depth from several tens to hundreds of micrometers The first layer, on the top of the treated surface, is known as the compound or white layer, composed mainly of ε-carbonitride $Fe_{2-3}(C,N)$. Its higher toughness, compared to that of ε-nitride phase, is due to its hexagonal layered structure that provides excellent anti-scuffing properties under dry and lubricated friction conditions. Although, the beneficial contribution of the compound layer on mechanical applications has been established, the mechanisms of its growth and crystallographic orientation are still a matter of scientific quest [11]. The layer underneath consists the diffusion zone, mainly composed of α-(Fe,N) solid solution [5,12]. Additionally, alloy nitrides and carbonitrides such as γ'-Fe_4N and, rarely, ζ-Fe_2N can be precipitated in both the compound and/or the diffusion zone. As reported in the literature: "*The diffusion zone characteristics are*

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essentially independent of the type of nitrocarburizing media used. During the treatment of plain carbon steels with a large proportion of ferrite only nitrogen diffuses in from the carbonitride layer, since the ferrite is normally already at its equilibrium concentration with respect to carbon. However, some outward carbon diffusion from high carbon concentration regions of the matrix into the compound layer can be experienced with most grades of steel and cast iron" [13].

Liquid nitrocarburizing is applied as final chemical-heat treatment for the improvement of tribological performance of various machine parts and friction elements, such as forming/cutting tools, dies, gears, shafts, clutches, etc. which are subjected to sliding or rolling friction. In this perspective, numerous experimental studies in the literature examine the performance of nitrocarburized steels under various tribological systems, involving sliding [14–19], rolling [7], erosion [20] and/or corrosion [21], in an effort to optimize either the surface treatment process parameters or the material selection for specific operational conditions. Representative studies on improving service life of parts/ components used in the metalworking industries concern the wear behavior of H13 steel dies used in crankshaft hot forging process [22] and in hot extrusion of Al [23].

Previous research by the present authors involved the study of the microstructure of the case depth obtained by Tufftriding on three different tool steels and its effect on disc-on-disc sliding friction under severe tribological conditions [17]. In subsequent works, industrial tungsten carbide inserts were used as counterbodies to simulate the wear mechanisms taking place during hardened and nitrocarburized tool steels' machining [18,19].

The present study is focused on the investigation of the influence of prior heat treatments of two commercial tool steel grades on their tribological behavior after nitrocarburizing under industrial conditions. The non-surface-treated state of the materials examined was also evaluated as a reference to assess quantitatively the potential improvement of the wear behavior due to nitrocarburizing.

2. Experimental procedure

2.1. Materials

The two selected tool steel grades, AISI H13 and Cr–Mo–V, were provided by Uddeholm S.A. Their chemical composition and commercial names are presented in Table 1. It should be noticed that all the alloying elements, except of Si, are strongly carbide-forming ones. In the case of the hot-work AISI H13, the presence of the strongly carbide-forming Mo and V leads to microstructures of fine-dispersed, spherical carbides, while its hardness at soft-annealed condition is 180 HB [17]. In the case of the cold-work Cr–Mo–V tool steel, the higher Cr and C percentages lead, during solidification, to the formation of characteristic, eutectic, coarse chromium carbides, while its hardness at soft-annealed condition is 235 HB [18].

Table 1
Commercial names and chemical compositions (wt%) of the examined tool steels.

AISI	Commercial name	Fe	C	Si	Mn	Cr	Mo	V
H13	ORVAR	bal.	0.39	1.00	0.40	5.20	1.40	0.90
–	SLEIPNER	bal.	0.90	0.90	0.50	7.80	2.50	0.50

2.2. Heat and surface treatments

The heat and surface treatments were performed at the industrial site of Uddeholm Steel Trading Company, Greece. For both materials, all testing specimens were sectioned from cylindrical bars of 40 mm diameter to disks of 10 mm thickness and subsequently polished to 1.0 μm average surface roughness (R_a). The specimens from each steel grade were divided in three groups; each one of them was then subjected to proper heat treatment that led to a final hardness of 40, 45 and 50 HRC. In all cases, the heat treatment procedure followed was that recommended by the materials' supplier and the whole thermal cycle included two preheating stages, austenitizing and three tempering stages. During austenitizing, the polished surfaces of specimens were protected against decarburization employing salt baths that prevented contact with air. The actual conditions are presented in Table 2.

Half of the heat-treated specimens of each grade and hardness obtained were further subjected to Tufftriding surface treatment, under the same industrial-scale conditions. Tufftriding is a liquid nitrocarburizing technique employing molten cyanide salt baths. The process was carried out in three steps: (a) preheating at 250 °C, (b) immersion in the molten salt bath consisting of 60% KCN, 24% KCl and 16% K_2CO_3 by weight, at 580 °C, for 4 h and (c) oil cooling. During the second stage of the process the basic chemical reactions are the typical ones, have been described previously [1,17].

2.3. Tribological testing and examination techniques

Sliding friction tests were performed in dry air (25% RH, 20 °C) using a ball-on-disc apparatus (Centre Suiss d' Electronique et de Microtechnique, CSEM). An alumina ball of 6 mm in diameter and 15.7 GPa hardness was used as a counterbody. Specimens of all series were tested under 1, 2, 5 and 10 N normal load, 200 mm s^{-1} sliding speed and for a total sliding distance of 10000 m. During testing, the friction coefficient was recorded, as a function of sliding distance. The total wear volume was calculated by measuring with a stylus profilometer (Taylor–Hobson) the track cross-sectional

Table 2
Heat treatment conditions applied for hardening of the two steel grades.

Steel grade	**AISI H13**			**Cr–Mo–V**		
Targeted hardness (HRC)	**40**	**45**	**50**	**40**	**45**	**50**
Heat Treatment stage	**Temperature (°C)/Time ($_{min}$)**			**Temperature (°C)/Time ($_{min}$)**		
1st preheating	650/30	650/30	650/30	650/30	650/30	650/30
2nd preheating	850/30	850/30	850/30	850/30	850/30	850/30
Austenitizing	1030/40	1030/40	1030/40	1030/40	1030/40	1030/40
1st tempering	550/120	550/120	550/120	550/120	500/120	500/120
2nd tempering	640/120	630/120	610/120	640/120	600/120	590/120
3rd tempering	590/120	590/120	580/120	590/120	550/120	530/120