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# Influence of case hardening on wear resistance of a sintered low alloy steel

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#### ABSTRACT

PM technique has been applied for some products in the autoindustries due to unique functions and cost saving. The wear resistance of PM steel parts is one of the most significant surface properties. Nitriding and carburizing processes consist of exposing metallic materials to nitrogen and carbon to improve their surface hardness and wear resistance. In this research, the partially diffusion prealloyed powders, Ultrapac LE, containing Fe-4Ni-1.5Cu-0.5Mo with 0.2% graphite and two different densities were sintered at 1120 °C for 30 min. Depending on the applied pressures during cold pressing, two different porosities of 14.11 and 10.26 vol.% were obtained. Some specimens were carburized and some others were nitrided in cyanate liquid salt bath. The pin-on-disc wear test and hardness test were used to evaluate the surface behavior of specimens. The results showed that the wear resistance increased by nitriding and carburizing processes and the effect of nitriding is more than carburizing on wear resistance. In the case of materials studied, except for 4 h nitrided specimens, other specimens with higher porosity level showed better wear resistance. In these specimens, large pores entrapped the wear debris and created a densified layer. It prevented the formation of large abrasive agglomerates. For the carburized specimens, wear mechanisms were affected by the brittle fracture caused by abrasive wear. So, wear resistance did not increase significantly. In this investigation, abrasive, plastic deformation and oxidation wear were observed as wear mechanisms.

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### 1. Introduction

The demand for PM (Powder Metallurgy) steel components is significantly increasing and different kinds of PM steels have been applied mainly in the automotive industry for engine and transmission systems. PM method offers promising developments in view of raw material and energy saving in comparison with other metal forming processes [1].

Economic life of many structural parts is limited by wear. The demands have increased hardness and wear resistance in the surface and higher toughness in the core than those of common parts made of low carbon steel. Most of low-alloy PM steels could not be used as-sintered. Thus, heat treatments are applied after sintering in order to strengthen PM steels. In these parts heat treatment can develop monotonic properties by change in matrix and homogeneity in microstructure [2–4].

The tribological properties of nitrided parts depend on formation of nitrides of alloying elements because no trans-

formation occurs during the nitriding process  $(510-590 \,^{\circ}\text{C})$ . In these parts, the tribological properties improve by formation of white layer especially in the adhesive wear condition [4,5].

Wear properties are influenced by total porosity and pore size. The study of roles of porosity and pore size on wear showed that for the porosity levels lower than 9% and small mean pore size (10  $\mu$ m), the wear resistance increased with decreasing porosity and abrasive wear mechanism dominated. For the porosity levels higher than 21% and large mean pore size (12–18  $\mu$ m), the mechanism of plastic deformation dominated and wear resistance decreased significantly with increasing porosity and pore size [7].

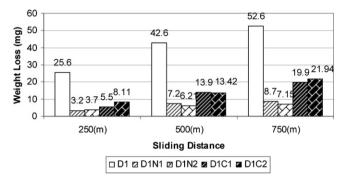
Some studies have showed that wear rate of sintered steels decreases with increasing density and hardness values [6,7]. However, Dubrujeaud et al. [8] observed that PM steels with high porosity levels display a low wear rate than steels with low porosity levels. It can be concluded that tribological behavior of sintered material is very complicated and depends on wear conditions greatly [7].

In this paper sintered low alloy steels containing Fe–4Ni–1.5Cu–0.5Mo were prepared with two different density values (6.7 and  $7\,\mathrm{g/cm^3}$ ) and then some were nitrided and some carburized in order to evaluate their wear resistance.

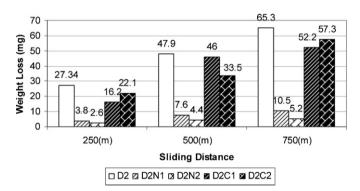
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**Table 1** Characteristics of specimens.

Code	Sintered density $(g/cm^3) \pm 0.05$	Sintered porosity $(\%) \pm 0.05$	Nitriding time (h)	Carburizing time (h)	White layer thickness (μm)	Hardness (HV20)
pi	6.7	14.11	-	-	_	82
DIN1	6.7	14.11	2	-	Not formed	214
D1N2	6.7	14.11	4	-	290	236
D1C1	6.7	14.11		4	-	290
DIC2	6.7	14.11	-	6	-	267
D2	7	10.26	-	-	-	104
D2N1	7	10.26	2		Not formed	217
D2N2	7	10.26	4	-	250	258
D2C1	7	10.26	-	4	-	343
D2C2	7	10.26	-	6	-	313



**Fig. 1.** Variation of weight loss with sliding distance for specimens with 6.7 g/cm<sup>3</sup> sintered density (14.11 vol.% porosity).



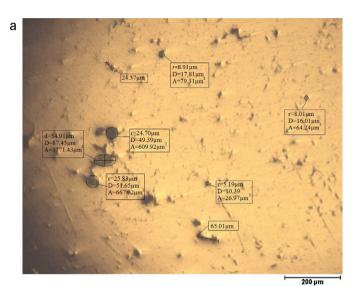
**Fig. 2.** Variation of weight loss with sliding distance for specimens with  $7 \, \text{g/cm}^3$  sintered density (10.26 vol.% porosity).

# 2. Experimental procedures

The partially diffusion prealloyed powder (with commercial name of Ultrapac LE) containing Fe-4Ni-1.5Cu-0.5Mo with 0.2 wt% C was selected for experiments. Carbon was added as fine natural graphite (UF4), and 0.75 wt% zinc stearate was admixed as lubricant. The powder was blended for 15 min in a tumbling mixture and then compacted at pressures of 480 and 600 MPa. Afterwards the specimens were ground to the size of 25 mm in diameter and 6.5 mm in length.

The compacted specimens were sintered at  $1120\,^{\circ}\text{C}$  for 30 min in furnace with  $10/90\,\text{H}_2/\text{N}_2$  atmosphere and the cooling rate was  $37\,^{\circ}\text{C/min}$ . The ASTM B328 standard was used to measure porosities of the samples. Depending on the applied pressure during cold pressing, two different porosities of 14.11 and 10.26 vol.% were obtained. Some of the specimens were carburized in cyanate liquid salt bath at  $930\,^{\circ}\text{C}$  for 240 and 360 min and tempered at 180 °C for 60 min. Some other samples were nitrided in cyanate liquid salt bath at  $570\,^{\circ}\text{C}$  for 120 and 240 min.

Wear tests were done by pin on disk test. A AISI 52100 steel pin with diameter of 5 mm was used as counter face. The dry sliding wear tests were carried out at room temperature in laboratory atmosphere (temperature:  $20-25\,^{\circ}\mathrm{C}$  and relative humidity: 30-60%) under normal load of  $60\,\mathrm{N}$  and constant sliding velocity equal to  $0.25\,\mathrm{m/s}$ .  $150\,\mathrm{m}$  sliding distance was considered as running-in and the specimens were worn for 750 m. After every 250 m, the specimens were cleaned and weighted with a balance to an accuracy of  $\pm0.1\,\mathrm{mg}$  and then were remounted in the wear tester at the same location. Each experiment for the wear test was carried out five times and the average of results was taken into account.



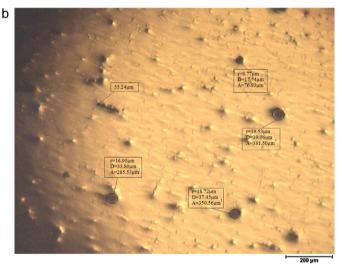


Fig. 3. Distribution of surface pores (a) D1 and (b) D2.

For evaluation of densified layer, Vickers microhardness measurements were carried out on the specimens. Hardness values do not have a fix amount in PM parts owing to the existence of pores. Thus the average amount was reported after five hardness tests. Optical electron microscope was used to investigate the distribution of porosity and thickness of white layers. The worn surface and wear debris were examined by scanning electron microscope and EDS analysis in order to identify the dominant wear mechanisms.

## 3. Results and discussion

The characteristics and hardness of examined specimens are reported in Table 1. Figs. 1 and 2 show variation of weight loss

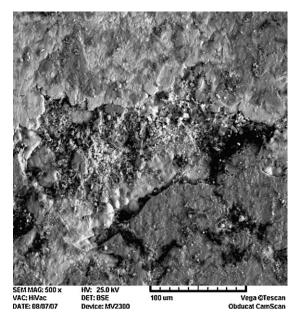


Fig. 4. Large pores filled with metallic particles and oxide debris (D1C2).

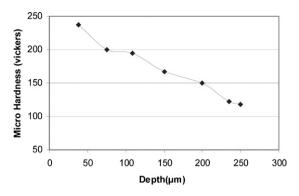
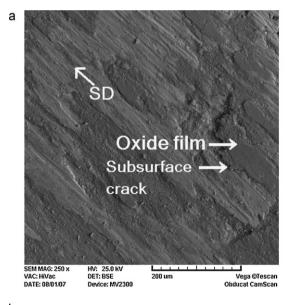


Fig. 5. Microhardness profile of densified layer D1.

with sliding distances of 250, 500 and 750 m for specimens of two different 6.7 and  $7\,\mathrm{g/cm^3}$  sintered density, respectively. According to Figs. 1 and 2, nitriding and carburizing processes improved wear resistance and the effect of nitriding on wear resistance was more significant.

In the case of nitrided specimens for 2 h, white layers were not formed and it affected the hardness and wear resistance. White layer in Nitrided specimens for 4h in comparison with 2h led to increase the hardness and decreased the adhesive wear. So it improved the wear resistance as explained by Ben Chikh Larbi et al. [9].



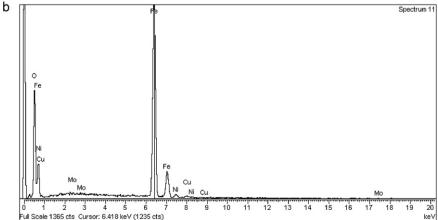
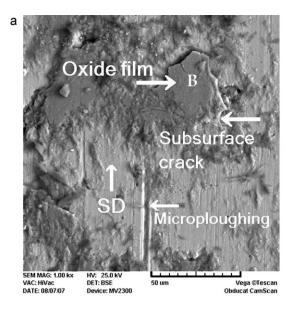
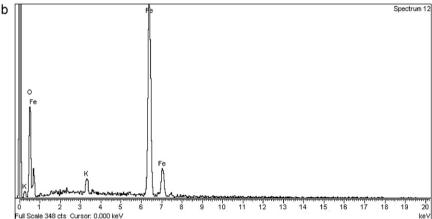


Fig. 6. (a) SEM micrograph showing the worn surface of the D1 and (b) EDS analysis at area (A).





**Fig. 7.** (a) SEM micrograph showing the worn surface of the D2N1 and (b) EDS analysis at area (*B*).

Nitrided specimens showed less hardness but higher wear resistance in comparison with carburized specimens. In the nitrided specimens, it seems that abrasive and adhesive wear were the mechanisms influenced by plastic deformation. In the carburized specimens in spite of higher values of hardness than others, wear resistance did not increase significantly. This indicates that in these specimens, wear mechanisms were influenced by the brittle fracture caused by abrasive wear [10].

According to Figs. 1 and 2, except specimens which were nitrided for 4 h, wear resistance was more affected by the porosity level. In the figures, specimens with high porosity level (14.11 vol.%) show more wear resistance than the specimens with low porosity level (10.26 vol.%).

It is well known that static properties of sintered material are dependent on the total porosity. The porosity is combination of "primary porosity" and "secondary porosity". Secondary porosity consists of residual porosity from liquid phase formation or diffusion of alloying additions such as copper at sintered temperature [4,8].

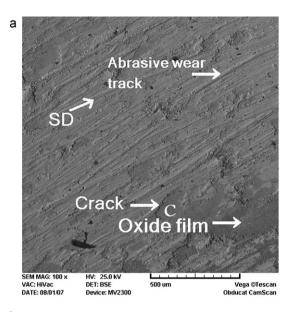
Fig. 3(a) and (b) illustrates distribution of surface pores of D1 and D2 specimens. The radius (r), diameter (D) and area (A) of some pore have been determined in the figures. As it is shown, there are some open porosities (14.11 vol.% porosity) on the surface of D1 specimen which are much larger than open porosities on the surface of D2 specimen (10.26 vol.% porosity). The pores at the surface might act

as sinks for wear debris and playing a positive role due to a decrease in the contact pressure and plastic deformation in the zones around the pores (Fig. 4).

In the specimens with low porosity level and small pore size, the capture of debris by the pores is difficult. The fact that these pores may be partially or completely closed by plastic deformation decreases the probability of filling the pores by debris. Reversely, in high porosity specimens, larger pore size favors filling the pores with metallic particles accompanied by hard oxide wear debris. In this case, the possibility of agglomeration of particles during sliding diminishes and so does the formation of large abrasive agglomerates. So, besides filling of the pores, plastic deformation around the pores creates a densified layer [11].

The microhardness profile of D1 specimen after wear test is depicted in Fig. 5. It can be seen that hardness values increase with approaching to the surface. The surface layers become harder owing to closure of the pores and work hardening and it improves wear resistance.

For specimens nitrided for 4h, another mechanism dominates by formation of white layer. In these specimens, presence of high porosity causes decreasing the real contact area between sliding surfaces and leads to increase the contact pressure. As a result it removes surface particles and generates hard wear debris and consequently wear resistance decreases.



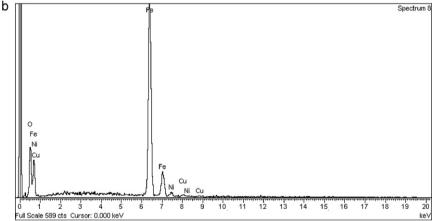


Fig. 8. (a) SEM micrograph showing the worn surface of the D2N1 and (b) EDS analysis at area (C).

**Table 2** Element contents (wt%) determined by EDS analysis at areas (A), (B) and (C) in SEM micrographs.

Element	Area (A)	Area (B)	Area (C)
0	30.91	31.71	18.24
Fe	65.90	68.29	78.28
Ni	1.73	-	2.11
Cu	0.80	-	1.36
Mo	0.66	-	

The oxidation wear mechanism is very often the predominant mechanism in dry sliding wear of ferrous materials [8]. High local temperatures attained during dry sliding allow development of oxide film. The oxide films can be seen in different specimens in Figs. 6–8. Also EDS results of the oxide films have been illustrated in the figures and Table 2. Dark regions show oxidation layers forming at high local temperatures. It leads to delamination by formation of cracks. After reaching a critical size, it breaks up and flake-like debris is generated. The wear mechanisms identified in this investigation are oxidation wear, abrasive wear and surface plastic deformation causing metallic particle detachment.

### 4. Conclusions

(1) Nitriding significantly decreased wear resistance than carburizing. Nitriding for 4 h is recommended as an appropriate process to improve wear resistance.

- (2) Inclusive of 4 h nitrided specimens, others with high porosity level (14.11 vol.%) showed better wear resistance than specimens with low porosity level (10.26 vol.%). In these specimens, large pores were filled with debris particles during sliding and created a densified layer. Debris captured by pores prevented particle agglomeration and improved wear resistance by reducing the abrasive wear.
- (3) The wear mechanisms in this investigation are abrasive, plastic deformation and oxidation wear. High local temperature attained during dry sliding, allow development of oxide film. When they reach a critical size, wear debris form.
- (4) For carburized specimens, in spite of high values of hardness, wear resistance did not increase significantly. This indicates that in these specimens, wear mechanisms were affected by the brittle fracture caused by abrasive wear.

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