TRIBOLOGICAL PROPERTIES OF SINTERED STEEL FOLLOWING PLASMA NITRIDING

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Abstract

The object of the investigation was the resistance of sintered steel type Astaloy CrM+0.3 respectively 0.7% C to abrasive wear in dry conditions and in lubricant - after plasma nitriding. In addition to variability in the carbon contents, shot peening was used before nitriding. Measurement results showed that, when tested in dry conditions, the friction coefficient is the same for all four variants of the material and is approximately 0.48. It is associated with the movement of the indentor, still on the same surface, representing the ε - phase, emerging on the surface of the material after nitriding. Friction in the lubricant shows a lower coefficient of friction on material with 0.3% C, either without or after shot peening, in comparison with a version with 0.7% C. The actual material wear is greater at samples with higher carbon content, while for shot peened material wear is independent of the carbon content. It seems that the vital role on wear resistance is played by the presence of surface ε - phase, whose composition is the same for all variations of the material, and secondly for different surface roughness, which for the shot peened samples is incomparably greater than for the non-shot peened state. However, it is assumed that the results will change in time after the elimination of ε - phase. This requires much further testing.

Keywords: powder metallurgy, wear, microstructure, SEM studies

INTRODUCTION

The issue of wear in metal materials, whether produced by conventional metallurgical processes or more recently by powder metallurgy, is the subject of numerous studies [1-4]. By the interaction between two material surfaces, wear is the main limiting factor. As shown from results achieved in this area [5-10] it is obvious that it is the technology of powder metallurgy which is able to provide metal materials the particular chemical composition and properties of a suitable surface treatment, which dramatically improves their resistance to abrasive wear. This work is a free continuation of the project VEGA Ministry of Education and Slovak Academy of Science No.10464/08: "Tribological aspects of damage of sintered materials failures as a result of rolling contact fatigue and wear", addressing the Department of Aviation Technical Studies at the Faculty of Aeronautics at the Technical University in Košice. For the tribological tests powder type Astaloy CrM from Höganäs AB was chosen. Currently it is one of the most commonly used powders for the manufacture of highly stressed machine components. In general the material has the highest strength only at a certain level of fixed carbon in the material, thus at its optimum amount.

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EXPERIMENTAL

Prealloyed powder Astaloy CrM (Fe+3%Cr+0.5%Mo) from Höganäs AB, Sweden was used for the testing. We chose two levels of carbon amount - 0.3% C and 0.7% C. It created two sets of samples. After adding an HWC type lubricant, the mixture of powders was compacted at a pressure of 600 MPa, manufacturing samples with dimensions Ø 30 x 5 mm. These were then subjected to sintering (1120°C/60 min) in an atmosphere which consisted of 90% N_2 + 10% H_2. To prevent possible oxidation of samples the atmosphere was frozen (dew point -57°C measured at the furnace entrance). To prevent possible decarburization, the samples were also put into a dusting powder of Al_2O_3 + 5% C.

Sintering was followed by natural cooling of the samples in a retort under a protective atmosphere, outside of the furnace. The produced samples were machined to Ø 28 mm and a central bore with Ø 10 mm diameter was made on them. Following sintering and cooling, the samples were subjected to double weighing to determine the specific density. The density was around 7.0 g.cm^{-3}. Both sides of the surface were ground, mainly due to flatness, which is very important for a balanced load to eliminate the undesirable phenomena that occurred on the surface (slight cementation). To increase the effect of the final treatment – plasma nitriding – half of the samples of each material variant were treated by shot peening from both sides.

Shot peening was carried out with steel granulate Ø 0.6 mm at 7000 rpm. The angle of shot peening was 90°. For both sides 9 kg granulate was used. Surface roughness after shot peening was about Ra = 6 µm (before ~ 1 µm). After shot peening, all samples were plasma nitrided on both sides (workplace: the Academy of Defence in Brno) under the following conditions: a nitriding temperature of 500°C, hydrogen gas/nitrogen ratio of 24:8; pressure 2.8 mbar, voltage 520 V; length of the pulse - pulse spacing of 100 µs; nitriding time 35 hours.

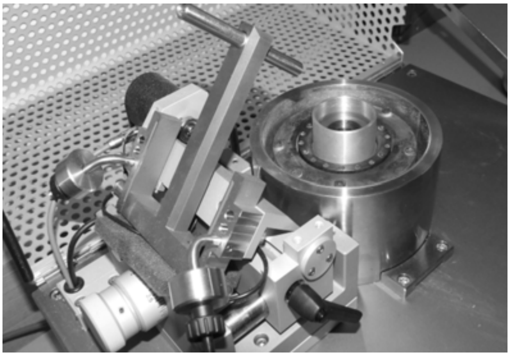
Tribological tests to obtain the necessary data were carried out on a "pin on disc" system - CSM Tribometer – Fig.1, under the following conditions: a track radius of 8 mm, speed of 0.20 m/sec, 8 N indentor load, sliding distance 360 m, temperature 20°C, indentor - hardened steel ball Ø 5 mm – DIN 100Cr6 - 62 HRC. The tests were conducted in dry conditions and with a lubricant (gear oil SAE 80 MOGUL). The coefficient of friction as well as wear damage during the entire test were measured and recorded.

Ultimately, four material variants were used for tests to determine the basic characteristics and then to determine the tribological properties of materials.

RESULTS

Microscopic analysis showed that the structure of the materials in question, following sintering and cooling, had bainitic structure. For samples with 0.3% C it is upper bainite and for samples with 0.7% C mostly lower bainite. The microstructures of materials are shown in Figs.2-3.

The figure shows the impact of shot peening, which in addition to being brought into the structure of compressive stress, is compacting the surface layer and thus reducing its porosity. Consequently, the density in a thin surface layer increased from the original 7.0 g·cm^{-3} up to 7.4 - 7.5 g.cm^{-3}. The depth of the shot peened layer was determined by observation of the metallographic sample porosity (pore absence) - Figs.2 and 3. In addition to the impact of shot peening, it can be seen to which depth the material is influenced by plasma nitriding - Figs.5 and 6.



(a) (b)

Fig.1. Principle of operation for 'pin on disc" and real Tribometer HTT).

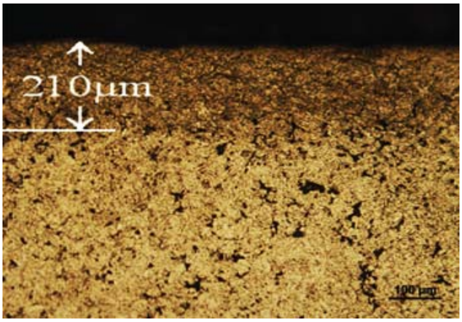


Fig.2. Microstructure of Ast CrM + 0.3% C. Shot peened + nitrided.

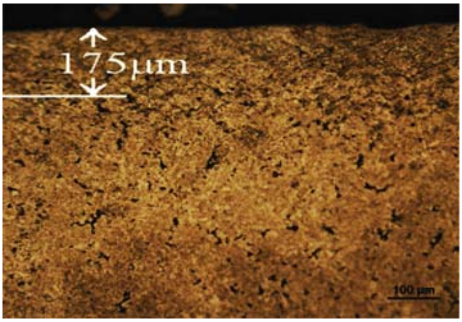


Fig.3. Microstructure of Ast CrM + 0.7% C – Sp. Shot peened + nitrided.

Tab.1. Sets of examined materials.

Mat.	1	2	3	4
All nitrided	Ast CrM + 0.3C	Ast CrM + 0.3C Sp	Ast CrM + 0.7C	Ast CrM + 0.7C Sp

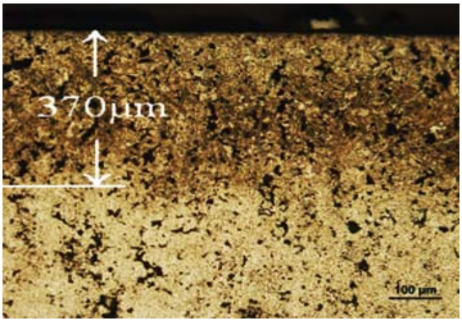


Fig.4. Microstructure of Ast CrM + 0.3% C.

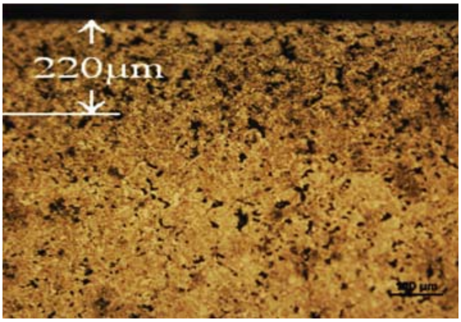


Fig.5. Microstructure of Ast CrM + 0.7% C.

It is assumed that the hard nitrided surface layer, under shock loads, will behave as a fragile layer. By observing the fracture surface one can determine the type of factual features applicable to this form of infringement. Using a scanning electron microscope, samples subjected to bending impact test showed that the subsurface layer had a typical brittle fracture, exhibiting cleavage facets, Fig.6. From their course on a cross-sectional sample it is possible to determine the depth of material affected by plasma nitriding. Finally, it should be noted that the surface of the material contained a compound layer with a thickness of about 3-4 μm. This layer, called in classical gas nitriding the ε - layer, seems to have similar properties when using plasma technology. One can at least assume that, unless removed, it will affect the tribological properties in the same way regardless of the base material. Its appearance is shown in Fig.7.

Furthermore, these variables were controlled by measuring the cross-section microhardness HV 0.05. The results are evident from Figs.8-9, confirming the value of the effect of surface layers after nitriding and after shot peening.

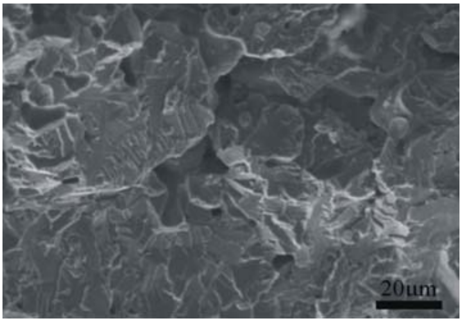


Fig.6. The area below the surface and fracture cleavage facets of brittle fracture.

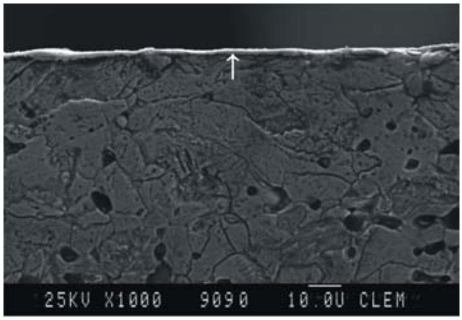


Fig.7. ε - layer on the surface of plasma nitrided material.

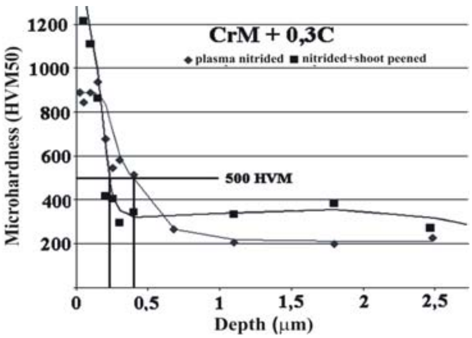


Fig.8. Behaviour of the cross-sectional microhardness of Ast CrM + 0.3% C material.

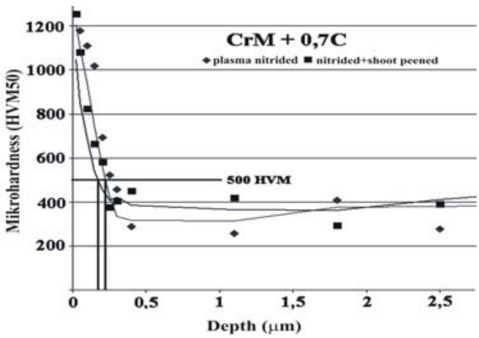


Fig.9. Behaviour of the cross-sectional microhardness of Ast CrM + 0.7% C material.

From the values measured the following facts are obvious: Material with 0.3% C has a lower strength and higher plasticity than the material with 0.7% C, which means the influence of shot peening (cold deformation), in terms of the depth, will be greater than the effect of material with less plastic. For the shot peened material the depth of the affected material is lower because the subsequent nitriding runs through a virtually denser material (subsurface porosity is substantially reduced). Logically, this shot peened layer generates more resistance to solid state diffusion than a material without this surface treatment. Shot peening and nitriding are processes which exert a similar effect in terms of etching capability. So the exact resolution, which effect is caused by surface hardening and which by nitriding, is only possible by comparing the hardness separately following nitriding and specifically following shot peening.

The following Table 2 shows the exact figures of measuring hardness by the Rockwell and Vickers methods (on the material surface) for each material variant.

Tab.2. The values of measured hardness HRC and HV10.

Samples	HRC	HV 10
Ast CrM + 0.3% C	29.0	561
Ast CrM + 0.3% C + Sp	29.7	678
Ast CrM + 0.7% C	29.4	609
Ast CrM + 0.7% C + Sp	30.6	755

Wear tests were carried out dry as well as using a lubricant (gear oil SAE MOGUL 80). The coefficient of sliding friction μ was measured, which depending on the track travelled, gives the results shown in Figs.10 and 11.

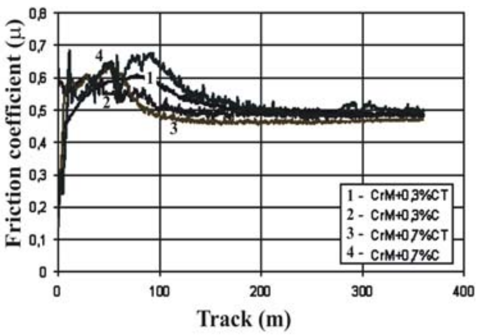


Fig.10. Friction coefficient - dry conditions.

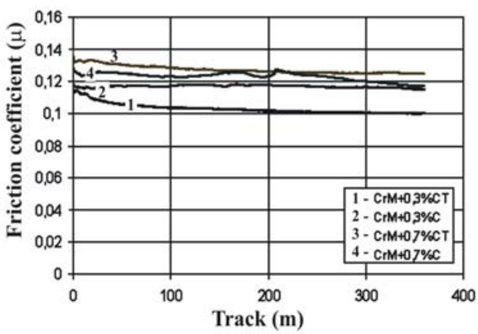


Fig.11. Friction coefficient – in lubricant.

The results showed that the friction coefficient under dry test conditions, having run about half the tracks, had stabilized at a level of about 0.48 ± 0.01, which essentially means that all four variants of the materials have a constant friction coefficient. It is given by the presence of ε - layers. This thin layer (3-5 μm) is essentially the same for all versions of the materials. This assumption of the constant coefficient of friction has been confirmed by the measurements shown in the profilograph - Fig.12, which indicates that the depth of the worn material in the track does not exceed the thickness of the ε - layer. In all the four variants it was the same friction pair of materials, the indentor material versus the ε - layer. Friction coefficients in lubricant are logically much lower than when tested dry. They range from 0.10 to 0.12. The lowest coefficient of friction material is for Ast CrM + 0.3% C and the highest for Ast CrM + 0.7% C in the state after shot peening. It is due to the fact that the microhardness on the surface in the first case is about 800 and for the second material about 1200 units. Therefore, it is necessary to mention the fact that material without shot peening has an average surface roughness Ra about 1.04 μm, the shot peened one has its Ra about 5.5.

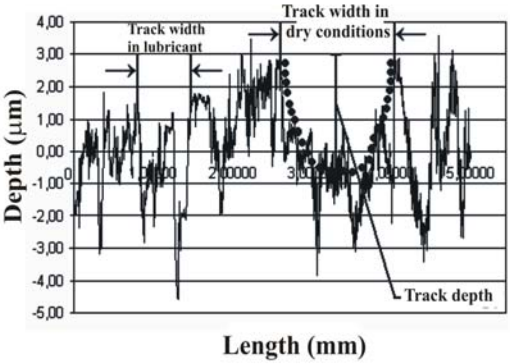
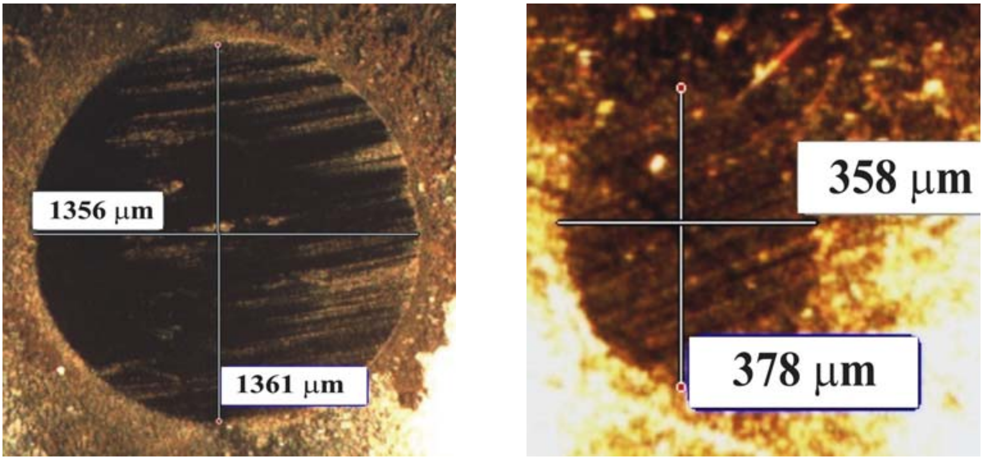


Fig.12. Example of the profilograph running along the track following the wear of Ast CrM + 0.3% C material with lubricant and without lubricant.

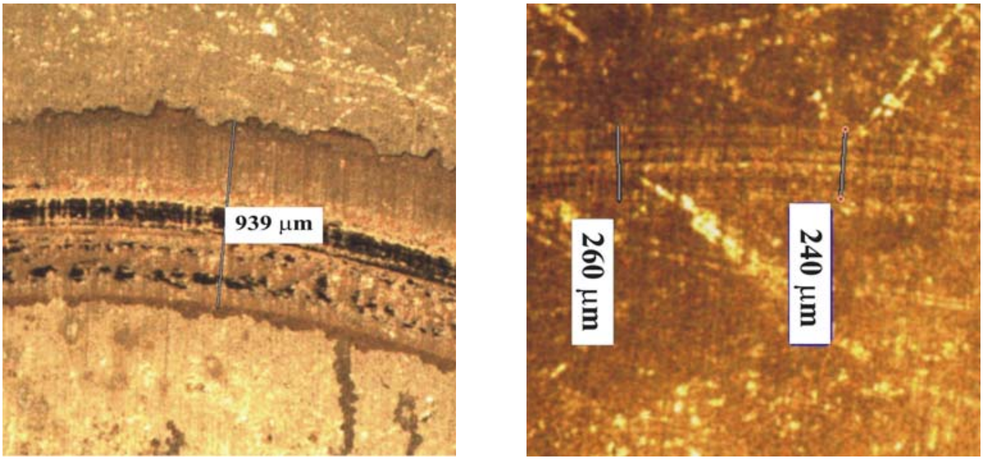
Wear recorded by test equipment actually consists of wear of the indentor and wear of the material. The magnitudes of the first and the second one can be calculated based on simple geometric formulae.

The amount of wear of the indentor was calculated on the basis of geometrical procedures of cross section; ball and wear depth of the material was measured by a profilograph. The total wear of the material was evaluated from the area which was defined by the width and depth of the track. These values are read from the given profilograph - see Fig.12, and we checked them by measuring the width of the sample directly using light microscopy, see for example Fig.14 a, b. Examples of the indentor wear in the lubricant/without lubricant are shown in Fig.13 a, b.



(a) (b)

Fig.13. The appearance of the indentor wear after riding 360 m without and with lubricant - material Ast CrM + 0.3% C.



(a) (b)

Fig.14. The appearance of wear after riding on a sample of 360 m without and with lubricant - material Ast CrM + 0.7% C.

The results thus obtained were processed into bar graphs shown in Figs.15 and 16.

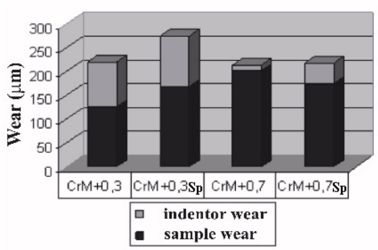


Fig.15. Wear materials and indentor test in dry conditions.

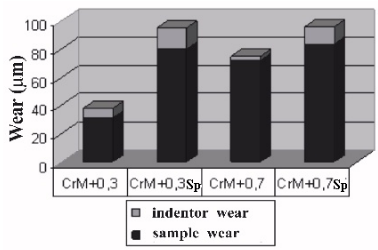


Fig.16. Indentor and material wear test in lubricant.

As shown by the results in Fig.15, wear values obtained for materials are different for those shot peened and a surface without shot peening. The wear of a surface without shot peening is worn higher for material with 0.7% C. For shot peened surfaces, wear is the same regardless of carbon content and is greater than for 0.3% C. It is due to the fact that the surface of such material has a significantly higher surface roughness and by friction the pressure of the indentor is transferred to the tip of the peaks and thus the pressure is much higher and there is more wear and tear. The wear in lubricant shows that this increases as the carbon content (i.e. material hardness) increases as for the version with shot peening and the surface without shot peening. Values for a surface with shot peening are always greater. Probably the same reasoning is valid as for dry wear, of course at a substantially lower level of values.

Finally, it is necessary to state a fact which has not been exactly solved by us. It is concerned with the wear of the indenter which does not correspond with the material wear - see Fig.13 a,b and 14 a,b. The diameter of the indenter wear, under dry testing, is 1361 µm and on the material - the width of track is only 939 µm. The indentor wear is larger than that of the material, although it is caused by the indentor itself. Logically, the values of the width of wear at the indentor and that of the track on the material should be identical. It is also valid for tests performed in oil, where the ratio is 378 versus 260 μm. This fact cannot be, by any means, attributed to the account of inaccurate measurement. It has been shown by other tests, the results of which have been published [11]. There we voice a certain presumption that for dry tests, a powder layer is generated on the surface of the material as result of wear of both the material and the indentor. New surfaces arise due to friction, and along with increased temperature, oxides are generated as a result. These hard powder oxide particles are laying freely on the surface. The indentor moving on these freely-located powder particles is preferably abrased, and it is the possibility of powder moving outside the wear track that can cause abrasion of the indentor.

CONCLUSIONS

Tribological tests of Fe-3Cr-0.5Mo + (0.3% C respectively 0.7% C) for the plasma nitriding of variants with and without shot peening showed that:

1. The coefficient of friction by tests in dry conditions for all tested materials demonstrated about the same values, expressed as $\mu = 0.48$. This phenomenon is owing to the fact that during this period the indentor still moves on the ε-layer regardless of base material. As shown by the results of measuring the cross-over track wear, this was in no way deeper than the thickness of the ε-layer.
2. Coefficients of friction in lubricants for the pair without shot peening are almost identical, so one can state the same for dry testing. The indentor is sliding on an identical nitrided layer. The lowest coefficient of friction is for the shot peened material with 0.3% C, and the highest is for the shot peened with 0.7% C. The difference is given by the hardness of the surface.
3. Wear of materials during both dry and lubricant testing have shown that material with lower carbon content demonstrates lower wear, whereas as a result of shot peening the wear increases. For material with 0.7% C this difference is not so evident.
4. For shot peened materials, either at a dry or lubricant test, the carbon content made no influence on the wear.
5. The influence of carbon and that of the shot peening appears to be negative in view of these selected tribological characteristics. This fact is becoming more interesting for the reason that both factors, i.e. shot peening and carbon content, are generally increasing the resistance of material against wear for compact materials and some tribological characteristics do it quite evidently for PM materials.
6. The tribological characteristics such as friction coefficient and abrasive wear of sintered Astaloy CrM type steel, the carbon content, following an additional treatment by shot peening and plasma nitriding, have a negative influence. On the one hand, it is an unquestionably positive influence in terms of increasing the hardness and decreasing surface porosity, but on the other hand, in view of the surface roughness, it increases wear. However, one can assume that this effect will diminish following long term exploitation.

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