THE TRIBOLOGICAL PROPERTIES OF THE COATED SINTERED MATERIAL

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Abstract
The basic tribological properties were investigated – friction wear resistance and contact fatigue resistance of sintered steel materials. These samples were produced on the basis of commercial powder (Höganäs) type Astaloy CrL and Astaloy CrM containing 0.3% C. The samples were coated by PVD method with use the DLC type of coating with a thickness of about 1μm. In qualitative point of view, the coating is beneficial, for increase of resilience to wear, as well as for resistance to contact fatigue. The article described phenomena that cause a discrepancy between the friction and wear. The measurement of surface roughness and metallographic-microscopic analysis were used. It was noted that the DLC coating thickness of 1 μm for sintered steels with standard porosity is insufficient.

Keywords: Sintered steel, wear, rolling contact fatigue

1 Introduction

Research of PVD coatings and their use in industrial practice is currently in an advanced stage. Especially hard PVD coatings are used mostly as abrasion resistant barriers on cutting tools, which allow extension of service in terms of the machinability of tough and hard materials. Also machine parts are coated to increase the abrasion resistance and reduce the friction (injection mold). They fit well also as decorative layers, e.g. in furniture fittings, jewellery or china. The coatings are used also in medicine for joint replacement surgery, dentures etc. [1-5].

At present, there is a large number of different coatings in practice, which can be divided according to various criteria. From practical point of view, some of the producers divided them into the following groups:

• Group one includes mainly the TiN - the basic and the most used layer. With micro-hardness of 20-25 GPa, it can be used in almost all applications. The advantage of TiN is good elasticity and adhesion. Next comes the coating TiAlN – micro-hardness of 25-33 GPa. It has excellent resistance to high temperatures. The interesting feature is the formation of a surface layer of Al_2O_3, which during operation contributes to reduced friction, increased diffusion resistance and improved cutting properties.

DLC coatings – (Diamond-like carbon coatings) with very low friction and high hardness - up to 60 GPa. They are mainly used in the automotive industry for coating parts - pumps, locks, etc.

• Group two in addition to traditional products of some manufacturers such as TiAlSiN, incorporates also layers used in highly specific applications such as: TiCN - microhardness of

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30-40 GPa. Due to problematic achievement of uniformity, as well as the maintenance of the chamber at the end of the process, the coating fades into the background. Today it is replaced, in particular, by coatings based on TiAlN for improved cutting tools and improved TiN in the area such as tensional tools. The ZrN coating features are consistent with those of the TiN. However, it is much more expensive. It is used mainly in medicine for its biocompatibility and for the decorative coating as well. The Me: C-H carbide coatings (Me: - as an optional metal) with hardness about 14 GPa are used mostly as lubricating coatings. They are cheaper than the DLC and are primarily used as a top sliding layer on hard – abrasion resistant coating. MoS_2 is known for similar characteristics and uses.

- Group three is actually an area of research noted for four main developments of PVD applications. These include - a combination of layers (multilayers), refining their structure, doping by other elements (Hf, W, Y) and the new types of layers. Creating new types of coatings include attempts of developing technology such as PVD layer, which currently can be made only by applying the process of CVD (Chemical Vapor Deposition - preparation of layers from gases at high temperatures around 1100 °C). Here we find in particular the BN type coatings or Al_2O_3. The area of new layers research can still involve very hard DLC layers whose properties and preparation are far from complete research and there are wide ranges of applications in stock.

This article is designed to assess the impact of DLC coating on basic tribological properties of sintered steels based on powder type Astaloy CrL and Astaloy CrM. It was shown that the use of hard coatings on the dense material at a contact load brings positive results. Therefore, scientists are looking for the possibility of using this technology for sintered materials, increasingly applied in the automotive industry, particularly in production of the specific parts. Effect of different types of coating and different types of surface treatments on sintered steel material contact fatigue resistance and friction wear are still in the focus of many researchers [6-17].

2 Experimental materials and methods

The samples were prepared from prealloyed Astaloy CrL (Fe-1.5% Cr-0.2% Mo) and Astaloy CrM (Fe-3% Cr-0.5% Mo) powder (by Höganäs). Two sets of samples were prepared by adding a graphite powder in amount of 0.3 %. After the addition of 0.6% HWC type lubricant, the samples were compacted at 600 MPa in the form of cylindrical specimens, dimension ϕ 30 x 5 mm. Then the samples were sintered in a controlled atmosphere (90% N_2 +10% H_2) at 1120°C for 60 min. The sintering atmosphere has a dew point of -57 °C. After sintering, the samples were cooled outside the furnace in an inert atmosphere. Subsequently, they were machined to the outer diameter of ϕ 28 mm with an internal hole of ϕ 10 mm and finally ground to achieve flatness in both circular areas. Density of samples was approx.7 $g.cm^{-3}$. Grinding also removes the surface layer, partially decarburized during sintering. Subsequently, all the samples were on the surface deep rolled, in order to achieve such a surface roughness as estimated by applying a DLC coating. In the case of deep rolling we used an equipment for testing the contact fatigue, AXMAT type - **Fig. 1a,b** where balls were exchanged for rolls (3 pieces - about 5 mm in diameter and 5 mm in length).

Deep rolling was performed with a constant force of 1100 N (time varies) by 230 rpm to achieve a mean value of surface roughness Ra (around 1µm). The effect of deep rolling on the properties of the materials will be referred to in the Results chapter. Thus, the samples were prepared in the

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company LISS AG (CZ-75661 Rožnov pod Radhoštěm) with use of the PVD technology of depositing DLC coating. The company had reported us only deposition temperature of 250 0C and coating thickness of 1μm.

The 2 sets of samples having undergone the preparation as described above, were then subjected to contact fatigue tests (using the pin on disc system) using the AXMAT type device – **Fig. 1a**, operating at 500 rpm. The sample replaces the upper part of the axial bearing, with 18 balls of ϕ 3.969 mm size, made of bearing steel, rolling on its surface. The balls are located in the lower part of the axial bearing and are lubricated with MOGUL SAE 80 transmission oil, circulated and constantly filtered during testing.

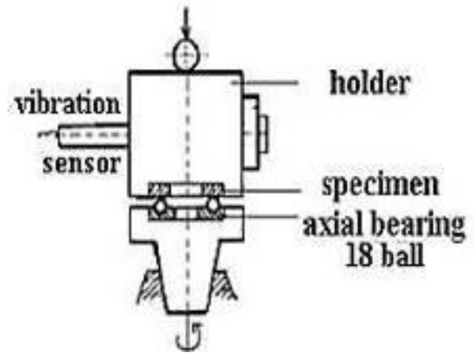

Fig. 1 a The principle of Axmat Device

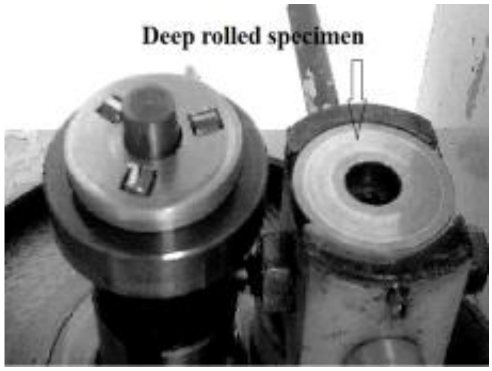

Fig. 1 b Specimen with deep rolled track and tool for deep rolling

Tribological tests were performed on a CSM Tribometer under the following conditions: tribological partner - pin - Al_2O_3 ball of ø 6 mm, sliding speed 25 cm /s, 6 N load. The length of track was 1000 m, track radius - 8 mm, temperature 20 °C in dry condition as well as in a lubricant (Gear oil SAE 80 Mogul).

To determine the basic characteristics of the test samples, classic methods were used. The results thus obtained are presented in the following chapter with indication of the test method. The samples after the tests were subjected to metallographic-microscopic analysis, hardness and micro-hardness measurement. Hardness values were obtained by conventional tests such as the Vickers or micro Vickers hardness measurements.

3 Results

As it has been found out, the analysis of the DLC layer pointed out some further facts ultimately having a major impact on the overall results. Its appearance is seen in the **Fig. 2a**. It shows that the thickness of the layer precisely reproduces all surface imperfections (sanding, pores ...). This fact is also evident from **Fig.2b**, which is taken in a cross-section and reveals a pore covered with a DLC layer. Observations and measurements at many surface places have proven that its thickness varies. In view of the stated thickness of 1 μm, it may in some cases show a difference up to 10 percent. Most essential is the fact that the thin layer is unable to override the inequalities deeper than the thickness of the coating.

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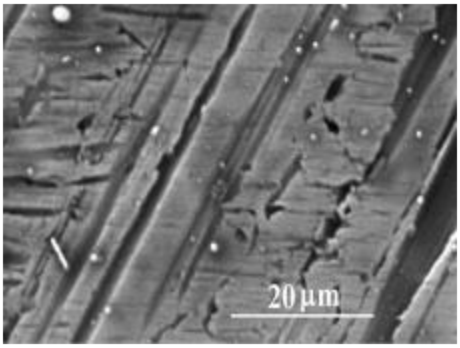

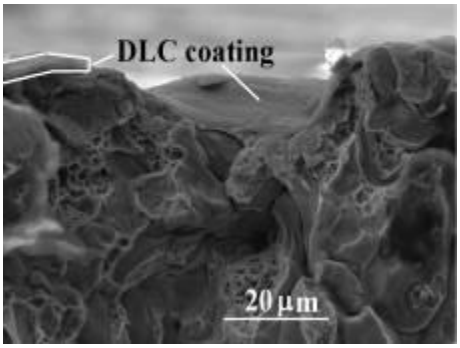

Fig. 2 a Appearance of the DLC coating **Fig.2 b** Cross section of the pitting site

Coating properties and their correlation with the base material as well as the influence of the surface finish by way of deep rolling can be assessed from **Table 1**.

Table 1 Properties of materials and coating

	CrL+0.3C		CrM+0.3C	
	HV 10	R_a (µ)	HV 10	R_a (µ)
Sintered	109	1.57	200	1.82
Deep rolling	156	0.06	218	0.12
With DLC	154	0.11	215	0.31
Without DLC	135	-	191	-

As it is apparent from **Table 1**, the deep rolling on soft Astaloy CrL material has a significant impact on its hardness. An adequate strain hardening occurred. In the case of Astaloy CrM the impact is substantially smaller, which is logical when we consider its nearly double initial hardness compared to Astaloy CrL. Effect of coating has proven itself in the measurement of hardness as counterproductive. The hardness of the coated base material decreased from 156 to 135, which is a result of tempering at the temperature of 250 °C, in which the coating takes place. Hardness after-coating including DLC films showed the value of 154 units, which is the summary hardness value of the DLC coating and the tempered material. Because indenter, in the measurement, pierced this layer and penetrates deep into the base material.
Friction measuring test are shown on **Fig. 3a,b**. As the results show, the coefficient of friction in dry condition is at the end lower for the coating that is deposited on the harder substrate. The same applies to the test in the lubricant. The limit values are 0.165 or 0.115 respectively. The essential difference is, however, in the kinetics of the process. In the case of test in dry condition, the friction coefficient increases for material Astaloy CrL + 0.3C over the entire period of time of the contact of friction materials. The coefficient of friction by the second material Astaloy CrM + 0.3C after the short term of increase, gradually declined to a constant value. In the case of testing with the lubricant, this aspect is not visible at all and the coefficient of friction is constant for the two materials all the time - the final value is 0.095 or 0.085 respectively.

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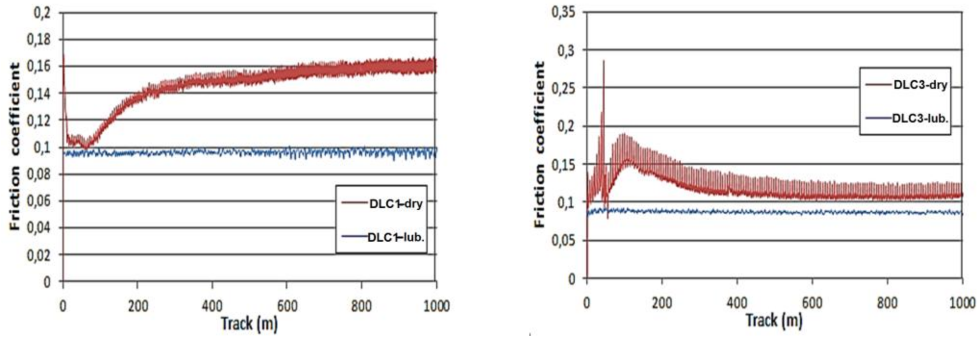

Fig. 3 a, **b** Development of coefficient of friction depending on the past track

Furthermore we monitored the wear that would be linked to the values of friction. A number of parameters have been studied as listed in **Table 2**.

Table 2 Measured parameters of samples

Astaloys	Friction coeficient	Width of track (μm)	Depth of track (μm)
CrL+0.3C + DLC - dry	0.165	~ 130 - 150	~ 0.4
CrL+0.3C + DLC - lub.	0.095	~ 130	~ 0.425
CrM+0.3C + DLC - dry	0.115	~ 190	~ 0.765
CrM+0.3C + DLC - lub.	0.085	~ 180	~ 0.7

The size of the wear can be inferred from the values of width and depth of the track after the test. The width of the track after passing 1000 m after examination in dry condition by the Astaloy CrL + 0.3C material is about 130-150 μm. By the Astaloy CrM + 0.3C, it is 190 μm. These facts were proved by profilographs going through the track - **Fig. 4**.

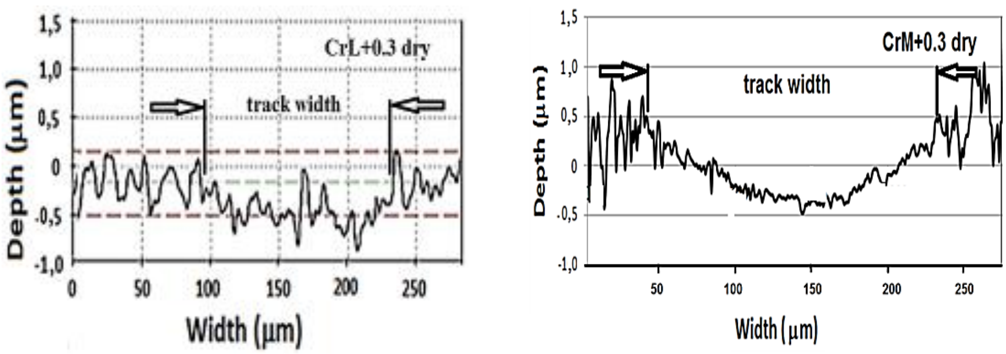

Fig. 4 Profilographs for materials after dry condition testing

The findings do not correspond to the values of the coefficient of friction. The width of the track is greater in material with a lower coefficient of friction. In case of lubricant testing the results are similar to **Fig. 5 a, b**. In both cases, the widths are slightly lower than in dry condition tests.

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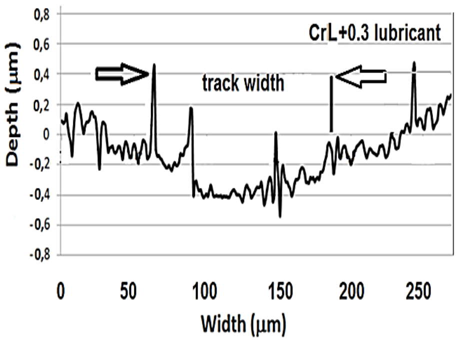
As for the depth of the track, there are the same results again. The track depth of the material Astaloy CrM is higher than that of the material Astaloy CrL. The effect of the lubricant is to this value is almost the same respectively the difference is so small that it is impossible to provide a relevant conclusion. Anyway, we can state that the wear of the Astaloy CrL + 0.3C material under the specified conditions was lower than that of the other examined material. This applies both to the dry condition and lubricant testings.



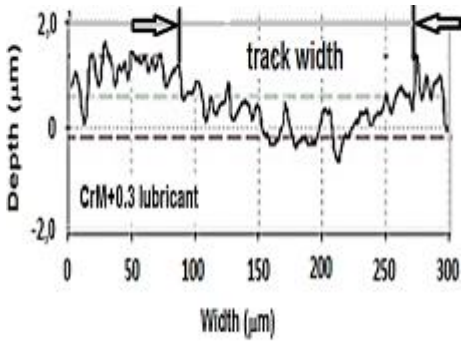
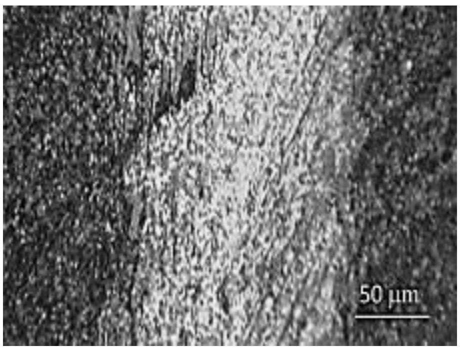


Fig. 5 a, b Profilographs for materials after the lubricant testing

By depth of tracks it can be stated that the DLC coating was not corrupt by abrasion or was not removed. By a simple calculation it is possible to determine that the volume of DLC film with a thickness of 1 μm and a width of 130 μm is 0.00435 mm^3, by the track width of 190 μm it is 0.0063 mm^3. All depths of traces are sufficiently less than the thickness of the DLC layer. Again, by the calculation it can be determined that in both extreme cases i.e. in the case of track width and depth of 130 μm and depth of 0.4 μm, the volume of worn material is 0.0017mm^3 and by the combination of 190 x 0.765 μm it is 0.0048 mm^3.

Both values are considerably smaller than the volume of DLC layer itself, under the same conditions of comparison. This means that the actual wear was concerned only with the DLC layer. However, there were a few cases where a small area of coating was damaged. These two extremes are evident in Fig. 6a, b. In the first case, we can see the track where the layer was not corrupt to the base material and on the next figure the small areas where the removal of the DLC layer occurred.



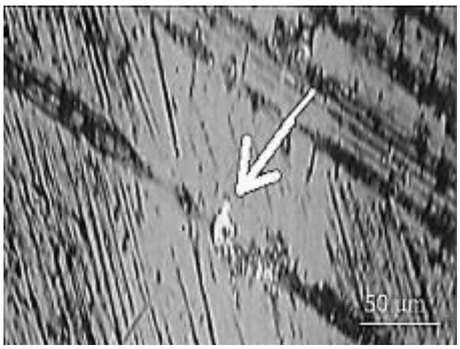


Fig. 6 a, **b** Appearance of track without breaking and with rupture of the DLC layer

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Contact fatigue test showed the results on **Fig. 7**. The agreed contact fatigue limit σ_C to the value of 50.10^6 cycles is logically higher for material with higher hardness. As shown in **Table 3**, this increase in fatigue strength for the material Astaloy CrL+0.3C is 28% and for the Astaloy CrM+ 0.3C material it is 21%.

Table 3 Rolling contact fatigue results

	Sintered		With DLC coating	
	CrL+0.3C	CrM+0.3C	CrL+0.3C	CrM+0.3C
σ_C (MPa)	660	850	820	1000
%	28		21	

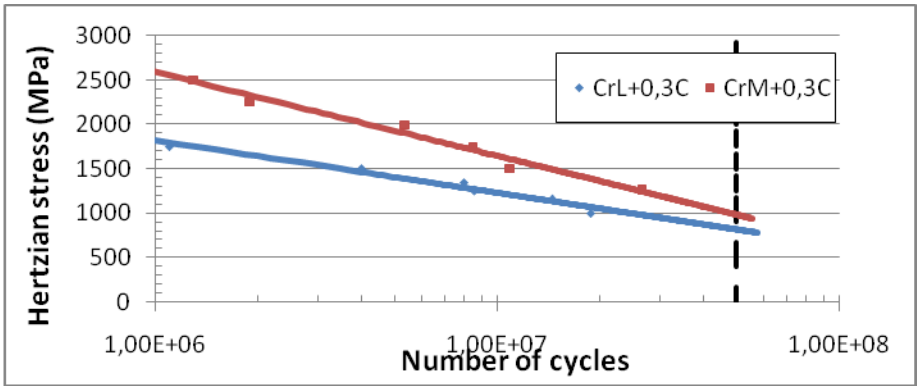

Fig. 7 S-N curves of investigated materials coated with DLC

Microscopic observation of the traces showed that during the contact stress at certain locations to cracking of DLC coating occurs - in a way as it is clear from **Fig. 8**. The primary damage leads to the formation of the pitting as a result of the rolling contact stress - **Fig. 9**. Here one can see the character as well as the extent of the removal of the layer in the track area. The fact that layer was removed from the areas where pitting was not created may be indicative of a small adhesion of the coating to the base material.

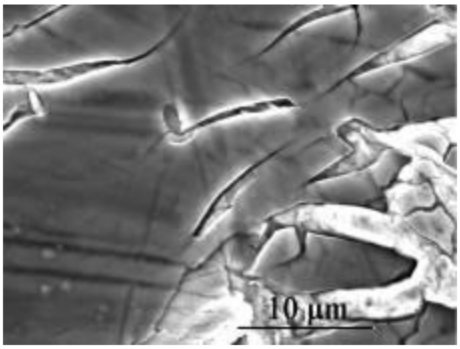

Fig. 8 DLC coating cracking during CF testing

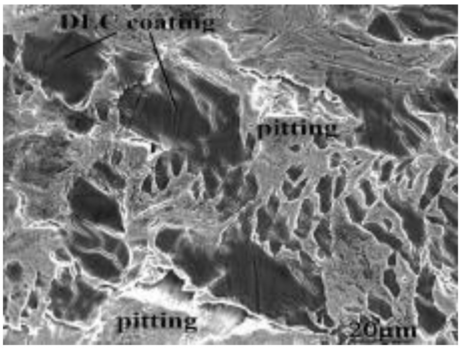

Fig. 9 Emergence of pitting in the track

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4 Discussion

Achievements evoke discussion mainly as to the discrepancy between the friction and wear and also regarding small increase in fatigue strength by contact fatigue tests. In our opinion, the problem of distortions in the first case is given by the fact related to the surface properties of materials. As it is apparent from Table 2, roughness Ra of the Astaloy CrM material is 0.31 μm compared to the roughness of the Astaloy CrL, which is only 0.11 μm. During the test of the wear by Astaloy CrL material, the indenter surface is sliding along the surface, the contact area is greater than that of the second material of the Astaloy CrM. Sliding in this case takes place mostly on the tops of the peaks which means smaller contact area. To confirm this effect, testing of roughness was evaluated, i.e. the value of Rp (which is the average height of the peaks on measured length). For the Astaloy CrM material, this value was 5.6 μm, while by the Astaloy CrL material it was only 3.34 μm. Moreover, this fact also supports the progress of the coefficient of friction during the test. For the Astaloy CrL material, as indicated above, and as presented in Fig. 3a, the coefficient of friction increases practically from the beginning to the end of the total track of 1000m. In the next case, for the Astaloy CrM material, the situation is opposite - Fig. 3b. At the start the coefficient of friction is high due to the high pressure per unit area (the area is small because the pressure is only transmitted to the tops of the projections). Here occurs their "strong" wear and increase of the contact surface at the same time. This also results in the fact that relates to the overall wear. Therefore, the wear of Astaloy CrM material is higher than that of the Astaloy CrL. It is assumed that over a longer time of testing this fact of different initial roughness could be eliminated.

As for the impact of the DLC coating on the value of fatigue limit stress of the contact in thickness 1 μm appears to be insufficient, even though the value of fatigue strength was increased. To this conclusion we have arrived at our experiences with other thicker coatings on the same materials – e.g. [18]. Furthermore, it was shown that the adherence of the coating in this case has not reached the required value. It may be necessary to find the optimal ratio between roughness, porosity and thickness of the coating. Many authors point to the fact that by polished bulk materials the ideal coating thickness is at values less than 1 μm [19].

5 Conclusions

The realized tests have shown that the DLC coating applied to sintered steels from tribological qualitative terms can be assessed as positive.

1. The coefficient of friction is lower in a material with higher hardness (the CrM material) and this is also true when tested in lubricant.
2. Wear does not respect this fact. During a relatively short period of testing there are other factors such as surface roughness come into play.
3. The contact fatigue resistance is also higher in material with higher hardness.
4. The relatively low increase in resistance to contact fatigue is primarily due to the small thickness of the coating, which is not able to cover the surface roughness and porosity of the sintered material. This and possibly other factors (e.g. surface roughness) can be related as causes of poor adhesion of the layer.

References

[1] J. Takadoum, H. H. Benani, M. Allouard: Surface and Coatings Technology, Vol. 8, 1997, No. 1-3, p. 232-238, DOI:10.1016/S0257-8972(96)02905-2

DOI 10.12776/ams.v21i1.467 p-ISSN 1335-1532
e-ISSN 1338-1156

[2] D. Jakubéczyová, P. Hvizdoš, M. Selecká: Applied Surface Science, Vol. 258, 2012, No. 12, p. 5105-5110, DOI:10.1016/j.apsusc.2012.01.138
[3] A. Erdemir, C. Donnet: Journal of Physics D: Applied Physics, Vol 39, 2006, No 18, p. 311-327, DOI:10.1088/0022-3727/39/18/R01
[4] I. A. Polonsky et. al.: Wear, Vol. 215, 1998, No. 1-2, p.191-204, DOI:10.1016/S0043-1648(97)00256-1
[5] P. Schlund, et al.: International Journal of Refractory Metals and Hard Materials, Vol. 17, 1999, No. 1, p. 179-185, DOI:10.1016/S0263-4368(99)00010-4
[6] R. Wei, P.J. Wilbur: Diamond and Related Material, 1993, No. 2, p. 898-903, DOI: 10.1016/0925-9635(93)90247-Y
[7] D. Rodziňák, P. Hvizdoš, J. Čerňan, K. Semrád: Kovove Materialy - Metalic materials, Vol. 50, 2012, No. 5, p. 365-371, DOI: 10.4149/km_2012_5_365
[8] D. Rodziňák, J. Čerňan, R. Zahradníček: Acta Metallurgica Slovaca, Vol. 19, 2013, No. 4, p. 282-291, DOI: 10.12776/ams.v19i4.142
[9] P. A. Deamley, A. M. Elwafi, R.J. Chittenden, D. C. Barton: Journal of Tribology, Vol. 136, 2014, No.2, p. 11, DOI: 10.1115/1.4026078
[10] R. Wei, P. J. Wilbur, M. Liston, G. Lux: International Journal of Fatigue, Vol. 162-164, 1993, p. 558–568, DOI: 10.1016/0043-1648(93)90541-S
[11] L. Rosado, V.K. Jain, H.K. Trivedi: Wear 1997, Vol. 212, No 1, p. 1-6, DOI:10.1016/S0043-1648(97)00147-6
[12] R. Bidulský, M. Actis Grande: Key Engineering Material, Vol. 409, 2009, p. 390-393, DOI: 10.4028/www.scientific.net /KEM.409.390
[13] M. Kumagai, S. Takagi, F. Yakabe, Y. Jinbo, Y. Hasagewa: Journal of The Surface Finishing Society of Japan 01/2006; 57(6):428-433. DOI: 10.4139/sfj.57.428
[14] R. Michalczewski, W. Piekoszevski, M. Szcerek et. all.: Key Engineering Materials, Vol. 527, 2013, p. 77-82, DOI: 10.4028/www.scientific.net/KEM.527.77
[15] C. Brecker, G. Gerlack: Friction, Wear and Wear Protection, Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany 2009, p.560-568, DOI: 10.1002/9783527628513.ch72
[16] J. Bidulska, R. Bidulsky, M. Actis Grande: Acta Metallurgica Slovaca, Vol. 16, 2010, No. 3, p. 146-150, ISSN 1335-1532
[17] R. Bidulsky, M. Actis Grande, J. Bidulska, M. Vlado, T. Kvackaj: High Temperature Materials and Processes, Vol. 28, 2009, No. 3, p. 175-180
[18] J. Čerňan, D. Rodziňák, J. Briančin: Powder Metallurgy Vol. 57, 2014, No. 4, p. 258-264, DOI:10.1179/1743290114Y.0000000094
[19] T. S. Stewart., R. Ahmed: Wear, Vol. 253, 2002, No. 11, p. 1132-1144, DOI: 10.1016/S0043-1648(02)00234-X

DOI 10.12776/ams.v21i1.467 p-ISSN 1335-1532
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