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Feasibility of TiBN PVD Coating for Mill Rolls – Laboratory Testing of Anti-adhesive and Fatigue Resistance Properties

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Abstract

The ceramic coatings deposited by Physical Vapour Deposition (PVD) are known for their high hardness. They offer a wide variety of friction coefficients. They have been used for a number of years for the coating of cutting tools; they have shown high efficiency in this field. Considering this, some of these coatings might prove efficient in the field of metal rolling. Yet to the best of the authors' knowledge, they have never been used industrially for rolling mill rolls. TiBN is one of the PVD coatings with the highest hardness. It is also known for its anti-adhesive property. In this study, the interest of TiBN in cold rolling is analyzed. In this application, the potential productivity is determined by the maximum pair (reduction, speed) above which seizure and transfer degrade tribological properties. The fatigue resistance is also studied and therefore, the potential influence of the coating on the roll life under conditions similar to those of rolling. Two well adapted tribological tests have been used for these two purposes: a plate-on-ring test, and a twin-disk Amsler test. The results of this study show a high potential of TiBN as coating for rolling mill rolls: good anti-adhesive property, very high resistance to wear and fatigue. It shows better performance than hard chromium, which is now widely used in this application.

Keywords: Strip rolling, Tool life, PVD coating, TiBN, TiN, Rolling mill rolls, Chromium plating, Tribotests.

Introduction

Two major parameters affecting the cost of reduction cold rolling are the productivity determined by the pair "reduction / speed", and the lifetime of mill rolls before the degradation of their surface state which necessitates their regrinding. These two parameters (productivity and lifetime) are related to the anti-adhesive properties and to wear resistance of the rolls. Indeed, under certain contact rolling conditions (load, speed, lubrication, temperature), metal particles worn from the strips tend to stick to the roll surface, changing its roughness, and therefore the roughness of the rolled strips. This phenomenon of adhesive transfer also generates a complete change in the effectiveness of lubrication, due to the change of chemical composition of the surface layer of the rolls; it usually results in a sudden increase in the coefficient of friction [Montmitonnet 2000]. The conditions for the occurrence of transfer are strongly linked to the nature of the rolls coating [Ould 2011]. On the other hand, the wear resistance of the coating is essential to maintain as long as possible the roughness of the rolled strips at the level required by customers, thereby reducing the frequency of rolls change. A coating intended for mill rolls must show good performance in terms both of wear resistance and anti-adhesive property. It is also necessary for the proposed deposits to have good adhesion to roll steel, and to have good fatigue resistance because of the cyclic nature of stress during rolling.

Apart from uncoated steel rolls, only chromium-electroplated rolls are used today in industry of reduction of steel strips. Hard Cr coatings have shown very good performance in this application, thanks to their hardness much higher than steels, and their good anti-adhesive properties. Yet an environmental threat seems to hang over hexavalent chromium, related to its strong toxicity (Picas,

2006; Navinšek, 1999). To substitute hard chromium in this application, coatings must be designed with performance as good, or even better.

For many years, ceramic coatings deposited by PVD and PECVD have been used very successfully to improve wear resistance of cutting tools, decrease friction to increase tools lifetimes [Prenzel 2001] [Zhang 2008]. These coatings are known for their hardness, much higher than hard chromium; they offer a wide variety of friction coefficients [Montes de Oca Valero 2002] [Mandibide 2003]. To the authors' knowledge, these coatings have not been used industrially for mill rolls. Azushima and Morita (1992) and Jimbo and Azushima (2001) studied TiN as a roll coating; they concluded that higher reduction could be obtained compared with uncoated rolls. TiN performed as well as hard Cr. [Ould 2011] however noted that with TiN, adhesive transfer appears at loads of the same order as with uncoated rolls, but lower than with Cr-plated rolls. The difference between the two studies could be partly attributed to different TiN coating roughness. Yet, it is improbable that TiN would allow safe rolling with as large a reduction as hard chromium. Anyway, TiN has not been introduced in the rolling industry since the publication of [Azushima 1992], almost 20 years ago.

In the present study, the interest of TiBN as a coating for mill rolls is explored. TiBN is one of the PVD coatings with the highest hardness, and it is known for its excellent anti-adhesive properties. Its coefficient of friction does not seem either to be incompatible with this application. The anti-adhesive properties of TiBN and its fatigue resistance are studied hereafter and compared with hard chromium and uncoated steel, thanks to two well adapted tests:

- The first one is a plate-on-ring test: rings are made of roll steel, and plates are cut from strips already cold-rolled or intended for rolling. The rings may be PVD-coated. Conditions relevant for strip rolling are met in this test:

temperature, pressure, plastic deformation, lubrication in the mixed / boundary regime. This test has proved efficient to evaluate the anti-adhesive property of coatings.

- The twin-disk Amsler test evaluates the fatigue resistance of the coatings, reproducing rather well the cyclic stress met by the roll surface in strip rolling, with a significant amount of sliding and under very high pressure.

TiBN and TiN PVD coatings have been carried out at a temperature below 180°C to avoid tempering effects, and their mechanical properties have been tested and given in **Table 1**.

Plate-on-ring test

The plate-on-ring test measures the evolution of the coefficient of friction between an axis-symmetrical ring and a plate (**Fig. 1**), at varying load for a fixed rotational speed. The ring and the plate represent respectively the roll and the cold rolled steel strip. Plates with various thickness can be used, and rings with an active zone of variable width. In the present case, the plates are either hot rolled strips (to study a first cold rolling pass), or already cold rolled strips (to study further cold rolling). The plates are 1 mm in thickness, the width of the active zone of the ring is 8 mm.

The speed is first ramped linearly ; the slope and duration are chosen by the operator. For a given rotational speed, the load may be changed stepwise during the test (**Fig. 2**); the duration and the load increment are also selected by the operator and are kept constant all along the test.

The test can be performed dry or with lubrication. Here, the ring-plate device is immersed in a two-litre tank filled with a steel rolling oil (**Fig. 1**). The temperature can be measured using a thermocouple introduced into the oil tank through openings in the lid, at a distance from the contact. It can also be measured on the back of the plate through a hole in the support. Two transducers (accuracy $\pm 0.1\%$) record the normal force and torque, from which Coulomb's friction coefficient is derived. To avoid the concentration of wear on a very small zone, the plate is given a small cyclic movement, the speed of which is negligible compared to the sliding velocity generated by the rotation of the ring; here, the amplitude is 10mm.

TiBN is PVD-deposited on rings made out of an HSS steel (1,30%C, 6%W, 5%Mo, 4%Cr, 3%V). The plates are cut from rolled strips (high carbon steel C100S). The main characteristics of the materials are given in **Table 1**. Several tests have been carried out to evaluate the coefficient of friction of the TiBN coating, and compare it with uncoated rings and chromium-plated rings under the same test conditions. This test should highlight phenomena often observed during the rolling process, such as seizure and adhesive transfer (severe wear); it should make it possible to identify the conditions of their occurrence. We could thus evaluate the influence of TiBN coating on these conditions, and therefore the potential interest of using it as a coating for mill rolls.

Results are provided hereafter for a rotational speed of 600 rotations per minute (rpm); the load steps are 100 N every 3 minutes, from an initial 200 N. **Fig. 2** gives the

correspondence between average and maximum pressures and the load applied, for the uncoated HSS rings / high carbon steel plate. These values are for a Hertzian contact; note however that plate wear tends to give a conformal contact, so that the pressure may decrease significantly.

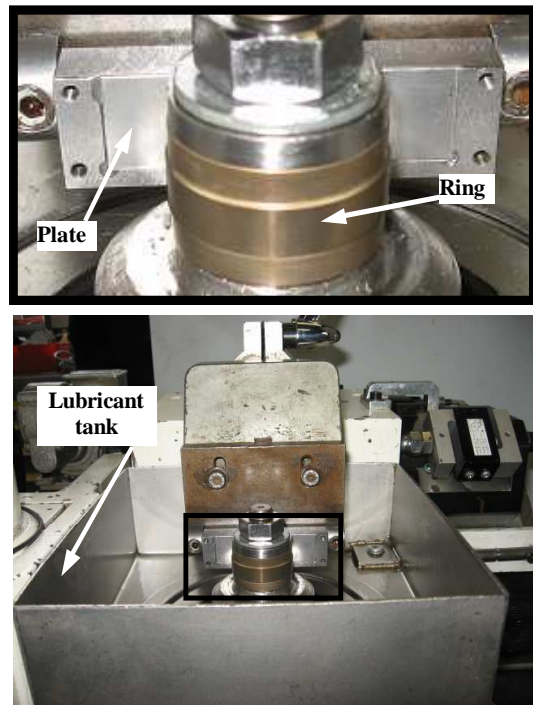


Figure 1. The plate-on-ring test device.

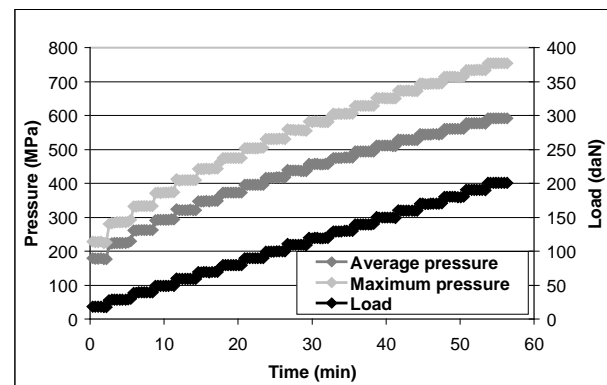


Figure 2. Pressure - load correspondence; uncoated HSS ring against a high carbon steel plate (Hertzian contact).

Table 1. Characteristics of the materials of this study.

Material	Roughness Ra (μm)	Scratch test critical load	Hardness Hv	Coating Thickness
TiBN-coated ring	0,12 μm	22,5 N	3600 Hv	2,1 μm
TiN-coated ring	0,104 μm	65 N	2946 Hv	2,7 μm
Cr-coated ring	0,26 μm	-	1080 Hv	9,4 μm
HSS ring	0,054 μm	-	840 Hv	-
Z100CDV5 roller	0,041 μm	-	740 Hv	-
High-C steel plate	0,16 μm	-	300 Hv	-

An experiment with TiBN has been repeated twice under the same conditions, showing good reproducibility (Fig. 3). It also shows no sudden rise of the coefficient of friction until the end of test at 2000N, which would have betrayed surface damage.

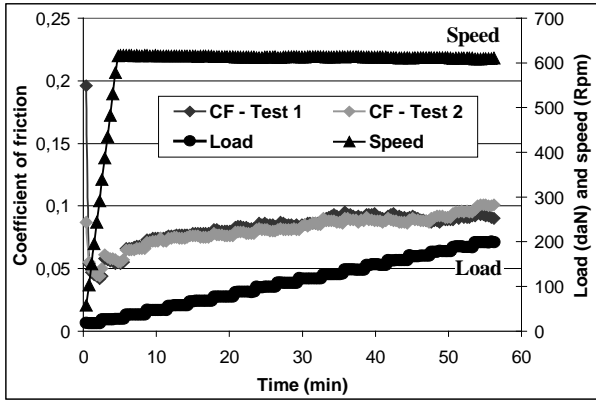


Figure 3. Reproducibility of the plate-on-ring test. HighC steel plates / TiBN-coated HSS rings / neat rolling oil.

For comparison, for the uncoated HSS ring against high carbon steel plate at a speed of 600 Rpm (Fig. 4, curve CF - HSS), three distinct stages had been observed (Ould et al., 2011):

- first, during the speed ramp, the coefficient of friction μ decreases (stage I); this is attributed to hydrodynamic effects;
- then, up to 1 kN, it is relatively stable (stage II).
- finally, μ becomes unstable and increases (stage III); observing the ring and plate, strong abrasive wear and adhesive transfer occur (see Fig. 6 below).

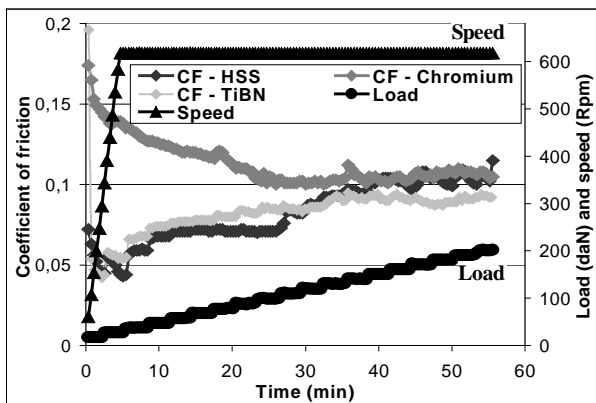


Figure 4. Friction coefficient evolution with load. Comparison of uncoated, TiBN-coated and Cr-coated rings.

Stage III has been also observed with TiN-coated rings at a disappointingly small load of 900N (Fig. 5). With TiN, the stage III friction growth is more sudden than with uncoated rings. The brutality of this transition after a very stable stage II makes the phenomenon easy to observe and interpret: adhesive transfer has occurred (Ould et al. 2011).

The effects of adhesive transfer are very negative, both on friction (which increases) and on the surface state of the plates (which becomes very heterogeneous). The common practice in strip rolling is to avoid “roll coating” or “roll pick-up”, i.e. the formation of these thick transfer layers, e.g. by hard chromium coatings.

Comparing the curves for TiBN, TiN, uncoated HSS and the reference Cr-plated rings (Fig. 4 5):

- In stage II, friction is sometimes slowly decreasing, maybe due to continuing roughness decrease by wear, as measured for Cr and TiN; sometimes increasing slowly (TiBN, HSS), maybe owing to thermal effects and growing load reducing hydrodynamic oil film thickness (and inducing metal transfer for HSS, not with TiBN).
- Stage III (severe wear stage) is absent for both TiBN-coated and Cr-plated rings, contrary to uncoated rings or TiN. This first result suggests that TiBN coating allows working under large loads, i.e. roll with large reduction.
- $\mu_{TiBN} < \mu_{Cr}$. Roughness is certainly a major factor: the initial roughness of the TiBN coating is substantially lower than that of the Cr-plated rings ($Ra_{TiBN} = 0,12 \mu m$, whereas $Ra_{Cr} = 0,26 \mu m$)
- Comparing evolutions of μ with load for TiN-coated and TiBN-coated rings (Fig. 5), TiBN allows applying larger loads without adhesive transfer (above 2000 N with TiBN, versus 900 N with TiN): these two coatings have a quite different potential for mill rolls application.

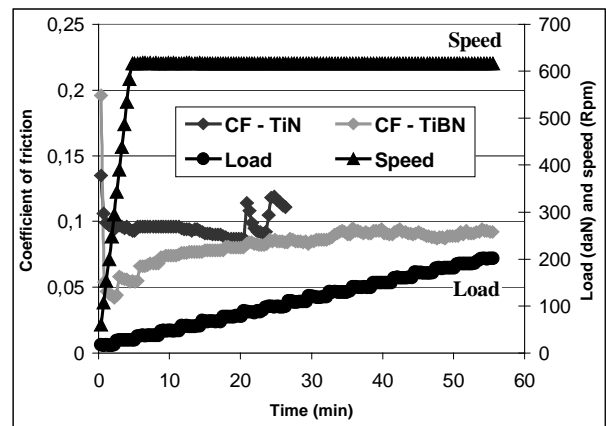


Figure 5. Friction coefficient evolution with load. Comparison of TiBN-coated and TiN-coated rings.

The study of the surfaces of rings and plates before and after the onset of adhesive transfer and severe wear shows the following evolutions of the surface states of the uncoated HSS ring and the corresponding plates (Fig. 6):

- before adhesive transfer, the plate surface roughness goes down to $Ra \approx 0,02-0,03 \mu m$; meanwhile, the roughness of the uncoated rings seems to increase slightly;
- Adhesive transfer marks the plates with wave- or scale-shaped features, with a fast, major increase of roughness from 0,16 to 0,4 μm ;
- Simultaneously, the abrasive wear rate of plates booms.

A test with a TiN-coated ring has also been stopped at the onset of adhesive transfer (Fig. 7a) for better observa-

tion of its influence on the surface states of ring and plate.

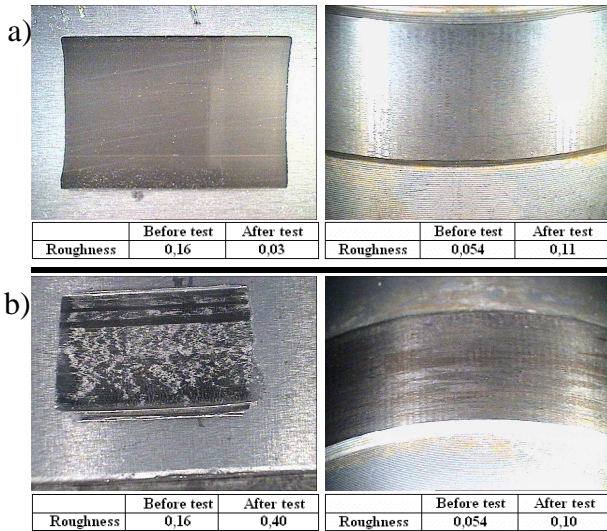


Figure 6. Influence of adhesive transfer on the surface state of plate (left) and uncoated HSS ring (right) : before transfer (a), after transfer (b). Roughness is quoted as Ra (μm). Roughness measurement conditions: sampling length $L = 4 \text{ mm}$, cut-off $\lambda_c = 0,8 \text{ mm}$.

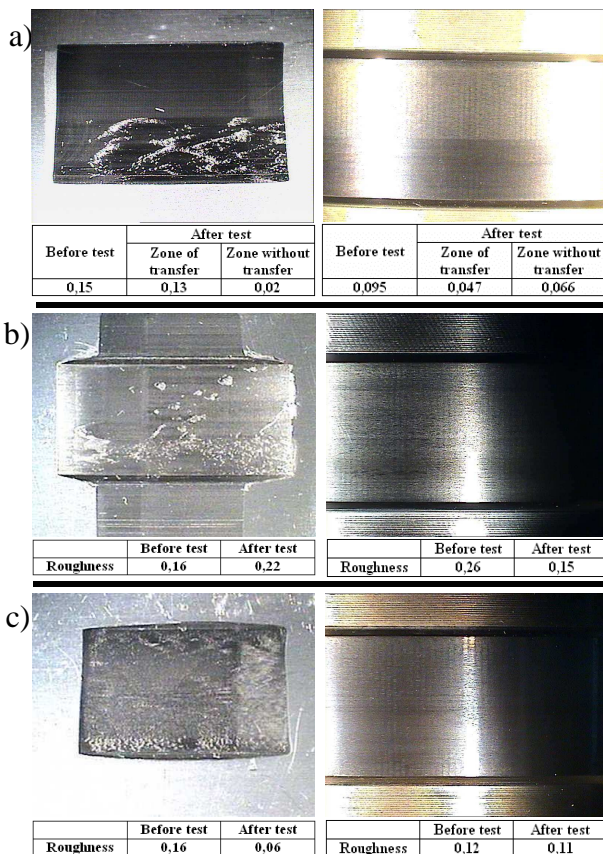


Figure 7. Surfaces state of plates (left) and rings (right) after tests: TiN-coated with a partial transfer layer (a), Cr-plated ring (b) and TiBN-coated ring (c). Roughness is quoted as Ra (μm). Roughness measurements conditions: sampling length $L = 1,25 \text{ mm}$, cut-off $\lambda_c = 0,25 \text{ mm}$ for (a), $L = 4 \text{ mm}$, $\lambda_c = 0,8 \text{ mm}$ for (b) and (c).

A conspicuous difference in the roughness of the plate ($0,02 \mu\text{m}$ versus $0,13 \mu\text{m}$) has been observed between the zone where transfer has started (lower part in **Fig. 7a**) and the rest of the contact zone. A major difference of the aspect of the plate surface is also observed, together with a darkening of the ring.

The same analysis has been done at the end of tests with Cr-plated and TiBN-coated rings (**Fig. 7b,c**). It shows that:

- the plate roughness increases with Cr-plated rings, from $Ra = 0,16 \mu\text{m}$ to $0,22 \mu\text{m}$. On the contrary, it decreases with TiBN, from $0,16 \mu\text{m}$ to $0,06 \mu\text{m}$. Correspondingly, the wear of the plates during the tests with Cr-plated rings is much faster; but it is necessary to recall that the initial roughness of the Cr-plated rings is higher ($Ra = 0,26 \mu\text{m}$) than that of TiBN rings ($Ra = 0,12 \mu\text{m}$).
- The roughness of the chromium plated rings decreases substantially, from $0,26 \mu\text{m}$ to $0,15 \mu\text{m}$; peaks are erased whereas valleys are preserved, showing that it is wear and not filling of roughness by wear debris. By contrast, the roughness of the TiBN coating is practically unchanged (initial $Ra = 0,12 \mu\text{m}$, final $Ra = 0,11 \mu\text{m}$).

The results of the plate-on-ring tests show that TiBN coating avoids adhesive transfer, which makes it superior to the uncoated HSS ring and the disappointing TiN.

Compared with the popular anti-adhesive, anti-wear hard chromium coating, TiBN gives lower friction thanks to its anti-adhesive property and low roughness. Moreover, TiBN-coated rings seem to generate less wear of the plates, and it is worn itself undeniably more slowly than the Cr coating, because of its exceptionally high hardness.

Amsler Test

The twin-disk Amsler test evaluates the fatigue resistance of a coating under cyclic contact loading. The frequency is determined by the rotational speeds imposed on two identical rollers, 50 mm in diameter and with rounded contact surface (**Fig. 8**) so as to obtain a point contact. The base material is here a cold work tool steel Z100CDV5 (1% C, 5% Cr, 1% Mo, 0,25% V), either uncoated, or TiBN or Cr-coated as previously (**Table 1**). The two rotational velocities of rollers are different, to work under rolling / sliding conditions. Here, based on slide / roll ratio occurring in strip rolling, roller speeds of 380 and 350 rpm have been selected, giving a sliding speed of $0,08 \text{ m/s}$ ($SRR = 8\%$). The frequency of local load is therefore about 6 Hz, and the load on a material element of the roller lasts about 0,98 ms at each rotation. Two transducers (accuracy $\pm 0,1\%$) record the normal force and torque, from which Coulomb's friction coefficient is derived. Two types of tests have been carried out on this machine:

- a friction test during which the imposed load increases stepwise from 237,5 N to 1663 N. The selected increment is 237,5 N, the load step length is 30 minutes (**Fig. 9**). Apart from the evolution of the coefficient of friction with load, the goal is to determine the failure load of the coating, manifested by a friction peak.

- an endurance test for a fairly long period, about fifty hours. The load selected here is the last one before coating failure in the friction test; it is applied progressively in the first hour (load increment is 237,5 N and load step length is 10 minutes), then kept constant (Fig. 10). All tests are lubricated with neat oil.

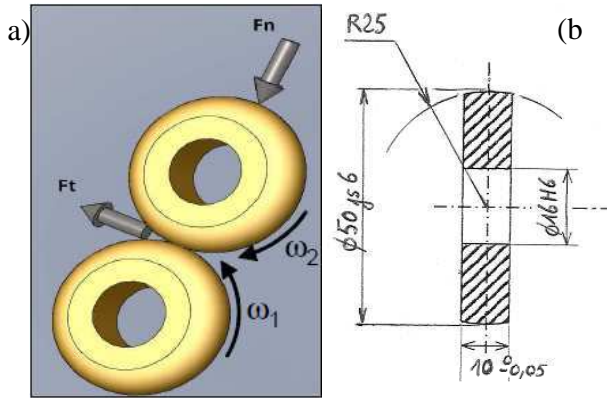


Figure 8. A general sketch of the Amsler test (a) and geometry of the rollers (b).

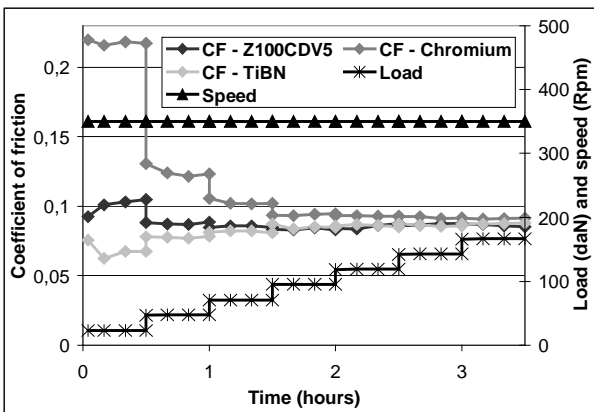


Figure 9. Amsler friction test (load increment: 237,5 N and load step length : 30 min, neat oil lubrication): evolution of the coefficient of friction with increasing load.

In the friction tests carried out on uncoated, Cr-plated and TiBN-coated rollers, the evolution of μ with load is rather similar to what has been observed in the ring-on-plate tests (Fig. 9). At the beginning of the test, μ_{Cr} is substantially higher than that μ_{HSS} , itself higher than μ_{TiBN} . The evolution with load is also different: for TiBN rings, μ increases with the load before stabilizing. On the contrary, for both Cr-plated and uncoated rings, the coefficient of friction decreases and stabilizes. Final, stabilized coefficients are almost identical ($\mu \approx 0,09$). Both coatings resist all applied loads, no chipping nor seizure is observed at the end of the tests.

Therefore, endurance tests have been carried out at the maximum load of 1663 N, giving a Hertz average pressure of about 2,2 GPa, for a total duration of fifty hours (1,08 10^6 cycles). Once the full load is reached, friction stabilizes again at $\mu = 0,09$ until the end (Fig. 10).

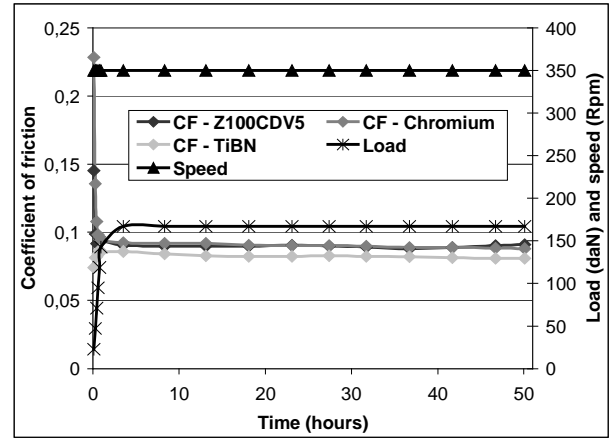


Figure 10. Amsler endurance tests (50 hours, pressure 2,2 GPa, neat oil lubrication).

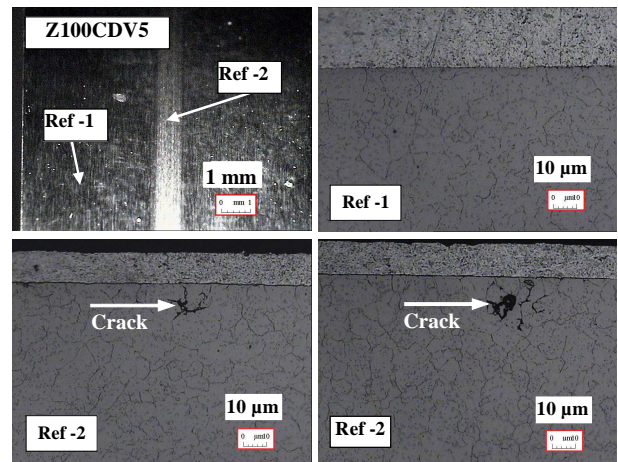


Figure 11. Cross sectional optical microscopy of uncoated Z100CDV5 rollers after endurance test. Top Right: outside contact track. Bottom: inside contact track.

The micrographic analyses of the uncoated rollers after the endurance tests show intergranular fatigue cracks under the contact zone (Fig. 11). No crack is detected outside the contact track. At a load of 2,2 GPa, the Hertz point is approximately 0,25 mm below the contact surface, much deeper than the cracks observed here (9 to 14 μ m). This suggests that these are fatigue cracks induced by the much more superficial shear stresses due to friction. It is also noted that the TiBN coating and Cr coating remain on the rollers until the end of the test (Fig. 12 13). However, the residual thickness of the coatings has been measured after fifty hours tests; it shows a reduction by $\approx 0,3 \mu$ m due to slow abrasive wear. No cracks have been observed on the coated rollers, either in the coating or beneath it. The large thickness of Cr may have maintained the high shear stress area inside this hard and wear-resistant coating. In the case of the thinner TiBN, the compressive residual stresses generated by the PVD process, with a maximum at the interface [Mandibide 2003], may be the reason behind the improved fatigue resistance. However, the mechanisms by which these coatings improve fatigue resistance deserve further study.

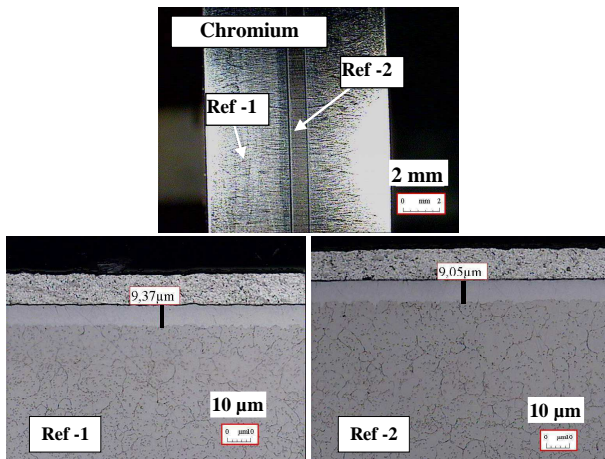


Figure 12. Optical microscopy of cross sections of the Cr-plated Z100CDV5 rollers after endurance test. Initial coating thickness: 9,4 µm. Left: outside contact track. Right: inside contact track.

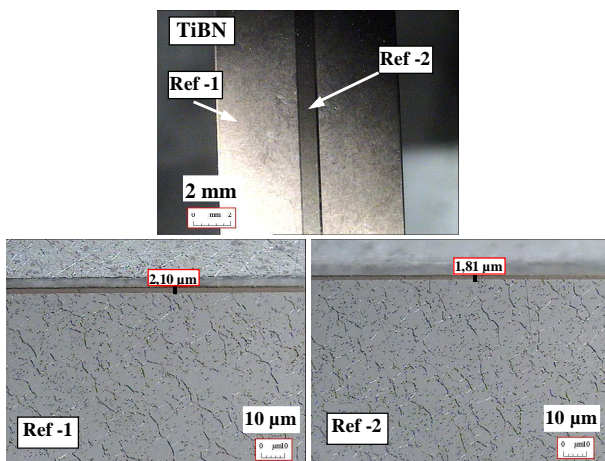


Figure 13. Optical microscopy of cross sections of TiBN-coated Z100CDV5 rollers after endurance test. Initial coating thickness: 2,1 µm. Left: outside contact track. Right: inside contact track.

Conclusion

In the plate-on-ring tests conditions, severe adhesive wear and roll pick-up appear at a load of ≈ 1000 N for uncoated rings and TiN-coated rings, degrading friction, whereas a TiBN coating, just as hard Cr, delays this phenomenon until beyond 2000 N. Considering these results, it is most probable that TiBN, if used as a coating of mill rolls, would allow substantially higher reduction than what is possible today with uncoated HSS steel rolls.

Wear and fatigue resistance are both essential to roll lifetime. The Amsler tests have shown a very significant improvement of fatigue life brought by both TiBN and Cr coatings, which sustain more than 1 million cycles under high stress and 8% sliding, maintaining protection over the underlying tool steel substrate.

The superiority of TiBN over Cr shows in the plate-on-ring test, through a slightly smaller friction, but still more in the much less intense wear of the plates. Moreover,

thanks to TiBN hardness, the coating roughness evolve very slowly.

Therefore, a TiBN coating may substantially increase the lifetime of the rolls (in so far as it is governed by evolving roughness), even compared to a chromium coating.

Acknowledgement

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References

- Azushima A., Morita Y., 1992. Lubrication characteristics of surface treated rolls. *ISIJ Int* 32, p.1232-1237.
- Jimbo Y., Azushima A., 2001. Lubrication characteristics of physical vapor deposition TiN-coated roll for high productivity in cold rolling of low-carbon steel sheet. *WEAR* 249, p.215-221.
- Mandibide C., 2003. Caractérisation des revêtements PVD à base de nitrures de métaux de transition pour application mécanique (Characterization of transition metals nitride – based coatings for mechanical applications), Ph.D. Dissertation, INSA Lyon (in French).
- Montes de Oca Valero J.A., 2002. Elaboration du carbure et du nitrure de titane par des procédés chimiques et physiques en phase vapeur : caractérisation de la microstructure (Elaboration of TC and TiN by CVD and PVD processes: microstructural characterization), Ph.D. Dissertation, University Bordeaux I (in French).
- Montmitonnet P., Delamare F. and Rizoulières B., 2000. Transfer layer and friction in cold metal strip rolling processes, *WEAR* 245, p.125-135.
- Navinšek B., Panjan P., Milošev I., 1999. PVD coatings as an environmentally clean alternative to electroplating and electroless processes. *Surf. Coatings Tech.* 116-119, p. 476-487.
- Ould C., Gachon Y., Montmitonnet P., Badiche X., 2011. Tribological Testing of Anti-Adhesive coatings for Cold Rolling Mill Rolls – Application to TiN-Coated Rolls. In: *Proc. ESAFORM Conf.* (April 27-29, 2011, Belfast, Northern Ireland).
- Picas J. A., Forn A., Mattäus G., 2006. HVOF coatings as an alternative to hard chrome for pistons and valves. *WEAR* 261 (5-6), p. 477-484.
- Prengel H.G, Jindal P.C., Wendt K.H, Santhanam A.T., Hedge P.L., Penich R.M., 2001. A new class of high performance PVD coatings on cemented carbide cutting tools, *Surf. Coat. Techn.* 139, p. 25–34.
- Zhang Z.G., 2008. Elaboration de dépôts nano-composés par pulvérisation cathodique magnétron pour la substitution du chrome électrolytique (Elaboration of nanostructured coatings by Magnetron Cathodic Sputtering as substitutes of electrolytic Cr), Ph.D. Dissertation, UTBM.