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Tribological behaviour of W–Ti–N coatings in semi-industrial strip-drawing tests

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ABSTRACT

W–Ti–N sputtered coatings were tested in semi-industrial conditions by strip drawing to access their ability for sheet metal forming. Both laboratory and industrially developed coatings were tested. In a first part of the study the upscale of the deposition of the W–Ti–N coatings was achieved. The main difference between the two types of coatings was the evolution of the chemical composition with the N content. However, similar trends in the hardness and scratch-test results were encountered being the best compromise of these properties reached for ~40 at.% N in the industrially deposited films against the 35 at.% N for laboratory deposited ones.

Excellent results were found in the strip-drawing tests for coated samples in comparison with uncoated tools. In any of the conditions tested no adhesion between the tool and the sheet material was found when 1 g m⁻² of lubricant was used. Similar results were found between laboratory and industrially deposited coatings.

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1. Introduction

The use of liquid lubricants in the sheet metal forming industry has brought an increasing number of problems due to the strict restrictions which are being imposed by EU in relation to the environment protection. However, there are many processes, as for example in deep drawing of ductile materials, where the absence of lubricants leads invariably to adhesion problems and premature wear of the moulding tools. One of the possible solutions for these problems is the surface modification of the tools by applying hard coatings which can, simultaneously, decrease the friction coefficient in the contact. For example, Le and Sutcliffe (2002) showed that these coatings deposited by PVD techniques can improve the wear resistance and contribute to the reduction or even elimination of the use of liquid lubricants.

In previous research works, Cavaleiro et al. (2003) performed a complete study of the W–Ti–N system in order to optimize the deposition by sputtering of thin coatings of this system. The chemical, physical and mechanical properties of the coatings were evaluated which made possible to select the Ti content for getting the best results: 20 wt.% Ti. By using the W + 20 wt.% Ti target they carried out a further detailed study involving the tribological behaviour of the coatings in dry and lubricated conditions (Silva et al., 2008). This study was carried out in the aim of the European

project ECOSTAMP (2001). Good indications could be drawn about the N content for achieving the best compromise between a very high wear resistance and a reduced friction coefficient.

The aim of this work was to study the potentiality of implementing coatings of the W–Ti–N system in industrial applications. For that, the tribological behaviour of the coatings was studied by a semi-industrial test: strip-drawing test. Furthermore, the scale up of the process was studied by using a sputtering apparatus with industrial dimensions allowing comparing the coatings performance in relation to the laboratory deposited ones.

2. Experimental details

The coatings were deposited by d.c. reactive magnetron sputtering in two different apparatus: (1) a one cathode laboratory scale HARTEC equipment working in balanced mode using a sintered W + 20 wt.% Ti target and (2) a four cathodes semi-industrial TEER equipment working in unbalanced mode using Ti targets embedded with round W pellets (see Fig. 1). From now on, the first coatings will be denominated as LabDep, whereas the industrially deposited ones as IndDep.

In the IndDep procedure, the ratio between the areas of Ti and W in relation to the preferential eroded zone of the target was approximately 20%:80%. Before deposition, the substrates were ion cleaned by establishing a discharge close to the substrate holder. The cleaning and deposition conditions are shown in Table 1. No intentional substrate heating was used during the deposition being the substrate temperature determined by both the plasma cleaning and

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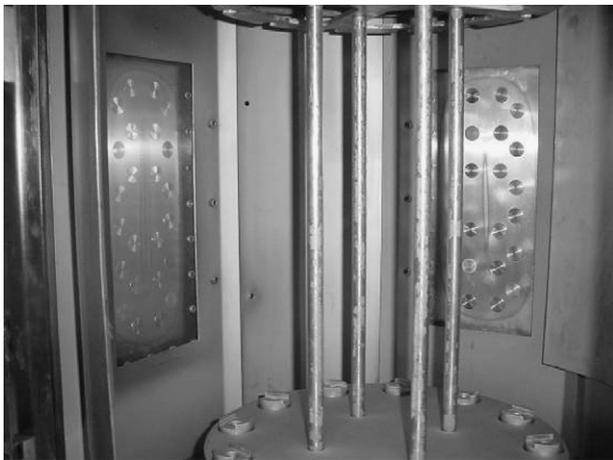


Fig. 1. Inside of the deposition chamber of the semi-industrial PVD equipment showing the puzzled target with round pieces of W (25 mm diameter) embedded in the Ti target (325 mm × 750 mm).

deposition processes. It is expected that the temperature does not overcome 180 °C. In fact, coating in same conditions heat treated carbon steel (tempered at 180 °C) did not lead to its softening after deposition.

In the case of the laboratory equipment, Silva et al. (2005) deposited the thin films of W–Ti–(N) by sputtering a W+20 wt.% Ti target in a N₂/Ar atmosphere varying the partial pressure ratio, p_{N_2}/p_{Ar} in the range from 0 to 1/2. A 0.5 μm thick W–Ti interlayer was deposited between the film and the substrate to improve the adhesion. The specific target power density was 11 W cm⁻² (P_{dep}) and a negative substrate bias (V_s) of 70 V was applied in all depositions. The total deposition pressure (p_{dep}) was 0.3 Pa and the deposition time was 35 min (5 min for the W–Ti interlayer and 30 min for the W–Ti–N layer, which allowed reaching a final thickness in the range from 2.5 to 4.5 μm). Before deposition, the substrate surfaces were ion cleaned with an ion gun. The cleaning procedure included a first electron heating up to temperatures close to 450 °C and, afterwards, Ar⁺ bombardment for 8 min (ion gun settings at 20 A, 40 V, substrates at –100 V).

The substrates in M2 (AISI) steel, used for the laboratory characterization of the coatings, were heat treated in order to achieve a final hardness of 9 GPa. Before being placed in the deposition chamber they were polished down with SiC paper of decreasing grain size (500, 1000 and 4000 GRIT) followed by 3 μm diamond paste and cleaned in ultrasonic baths of acetone and alcohol (final roughness, $R_a = 0.03 \pm 0.002$).

The chemical composition was evaluated by electron probe microanalysis apparatus (EPMA, Cameca SX-50). The structural analysis was performed by X-ray diffraction (XRD) using a Philips X'Pert diffractometer with Co K α radiation. The cross-section morphology of thin films was examined in a scanning electron microscope (SEM), JEOL JSM-5310. The adhesion/cohesion critical load of thin films was evaluated by commercial scratch-testing equipment (CSEM Revestest). The hardness and elasticity modu-

Table 1
 Deposition procedure and processing parameters of the W–Ti–N coatings.

Parameters	Substrate cleaning	Ti interlayer	W–Ti–N deposition
Discharge pressure	0.3 Pa	0.3 Pa	0.3 Pa
p_{N_2}/p_{Ar}	0	0	[1/20–1/2]
Discharge power (P_{dep})	100 W/target	2000 W/target	2000 W/target
Target current (I_A)	[0.3–0.5 A]	[6–6.5 A]	[6–6.5 A]
Target potential (V_A)	[200–250 V]	[300–350 V]	[300–350 V]
Substrate bias (V_s); pulsed current	500 V; 250 kHz	60 V; 250 kHz	60 V; 250 kHz
Deposition time (t_{dep})	15 min	5 min	60 min

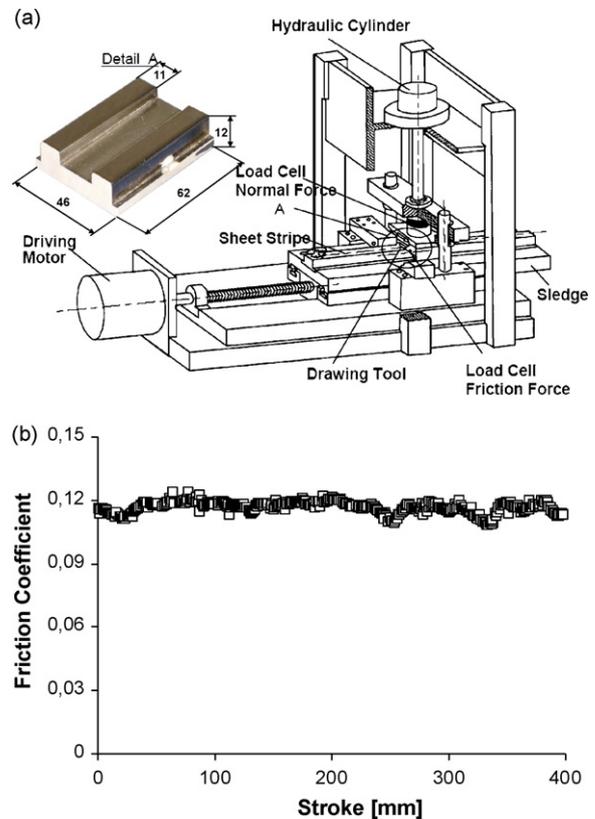


Fig. 2. (a) Schematic view of the strip-drawing test equipment. (b) Typical curve of the evolution of the friction coefficient with the stroke during a strip-drawing test – DDS-40G steel sliding against an uncoated D2 steel tool with 2 g m⁻² of lubricant A under 5 MPa normal pressure.

lus measurements were performed in a depth sensing indentation equipment (Fischer Instruments – Fischerscope H100). For surface roughness, a commercial profilometer was used (Mahr perthometer S4P with Focodyn Pherther laser head).

The evaluation of the forming behaviour of W–Ti–N coated parts was accessed by strip-drawing test (Fig. 2) following the procedure developed by Liewald et al. (2005, 2006). The tool (see inset in Fig. 2) was in D2 (AISI) steel and it was surface prepared with the same procedure as the laboratory samples (final roughness, $R_a = 0.04 \pm 0.004$ μm). The contact area of the tool during the test was close to 1200 mm². The forming behaviour was evaluated as a function of the lubricant content and the normal load applied between the tool and the sheet material.

The real friction coefficient (μ) was calculated during the sliding test by two load cells installed in the equipment allowing measuring the tangential and normal loads. The drawing speed was 100 mm s⁻¹ and the stroke way, 440 mm. A typical friction curve is shown in Fig. 2(b). The μ -average value for all tests was calculated between 168 and 368 mm stroke way. The tribological results are average values of five tests. The tests

Table 2
 Properties of selected stamping oils.

Designation	Density at 15 °C (g/ml)	Viscosity ^a at 40 °C (mm ² /s)	Flash point (°C)
A	1	1.1	100 ^b
B	0.89	20	181
C	0.88	27	154
D	0.89	26	180

^a ASTM D-445.
^b Ebullition point.

were performed in laboratory ambient conditions (temperature close to 24 ± 2 °C and 40 ± 5% of humidity) without (dry) or with lubrication. Four types of lubricants currently used in stamping shops of automotive factories were selected for the tests, three are used as process lubricants in deep drawing processes – A (emulsion), B, C – and one used for corrosion protection of the metal sheets—D (see Table 2). The tests were carried out against different types of materials: (1) high strength low-alloy steel (HSLAS-F – HV_{0.05} = 2.0 GPa, Ra = 1.11 ± 0.17 μm), uncoated cold rolled steel (DDS – HV_{0.05} = 1.2 GPa, Ra = 1.23 ± 0.10 μm), or coated with an electrogalvanised zinc layer (DDS-40G – HV_{0.05} = 0.6 GPa, Ra = 1.21 ± 0.09 μm) and Al/Mg/Si alloy (AA6016-T4 – HV_{0.05} = 0.7 GPa, Ra = 0.94 ± 0.03 μm). The test pieces were 800 mm in length, by 50 mm in width and 2 mm thick. The first and the last 200 mm were ignored, only 400 mm being considered for the results.

3. Results and discussion

3.1. Chemical composition and structure

The chemical composition of the coatings produced in the laboratory equipment was extensively reported previously by Cavaleiro et al. (2003) and Silva et al. (2005, 2008). Films with N content from 0 up to 55 at.% were deposited. Fig. 3 shows that the films deposited in the semi-industrial equipment can also be deposited with N contents of this order. However, there is a significant difference in the trends observed between both equipments in relation to the evolution of W/Ti ratio with increasing N contents. In fact, as shown in this figure, the W/Ti increases with the N content whereas an inverse trend was found in previous works. There, the decrease in the W/Ti ratio with increasing N content was attributed to the stronger bonding established between Ti and N avoiding the preferential re-sputtering of Ti in relation to W. This re-sputtering, induced by the lower atomic mass of Ti in relation to

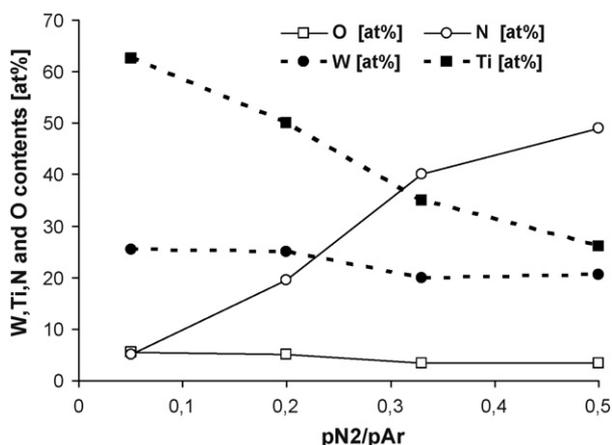


Fig. 3. Evolution of the chemical composition of reactively deposited W–Ti–N coatings as a function of the partial pressure ratio of the reactive gas (p_{N_2}/p_{Ar}).

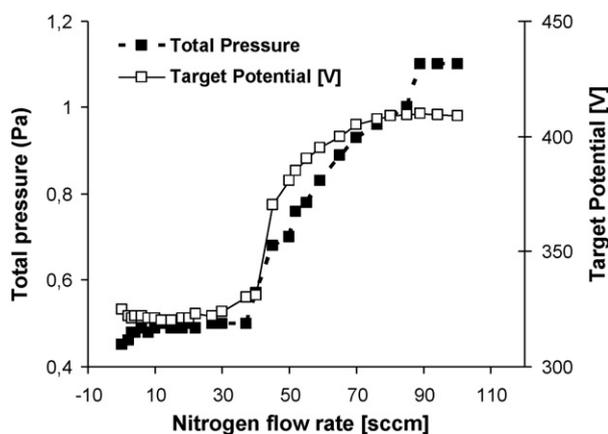


Fig. 4. Evolution of the target potential (V_t) and discharge pressure (p_{dep}) with increasing nitrogen flow rate in an experiment carried out in a reactive discharge with the puzzle W + Ti target. The sudden increases of V_t and p_{dep} are typical of target poisoning during Ti deposition.

W is a similar phenomenon as the one demonstrated by Winters (1982) who showed that during sputtering of a compound the lighter element was preferentially sputtered away independently of the sputtering rates of the individual elements of the compound. Hence, a very low Ti/W ratio in the 0 at.% N film is achieved comparing with the original Ti/W ratio in the target (0.3 against 1.0, respectively).

For IndDep films, the situation is quite different: the target is not an alloy of W and Ti (i.e. W and Ti are mixed in the target structure), but a puzzle of parts of W inserted in a big part of Ti (i.e. W and Ti can be considered as individual targets). By this reason, the target poisoning process is quite different: it is uniform in the alloyed target and heterogeneous in the puzzle target, occurring much faster in the Ti parts than in the W parts due to the much higher affinity for N of Ti than W. In fact, Goldschmidt (1967) presented values for the heat of formation of W–N compounds lower than those of Ti–N compounds. Fig. 4 shows the evolution of the target potential and discharge pressure as a function of the flow rate of the reactive gas where a sudden variation is observed for N₂ flow ~40 sccm. Mientus and Ellmer (1999) showed that this abrupt change in the plasma parameters is typical of the target poisoning when TiN deposition is concerned, whereas Hones et al. (2003) found that this phenomenon was much harder to occur when working with a W target. Thus, for low partial pressures of reactive gas, the sputtering rate of Ti is much higher than the one of W due to the larger area of exposed Ti. However, with increasing partial pressure of N₂, the Ti parts start to be poisoned and the sputtering rate drastically decreases. As the W parts are not still poisoned the consequence will be a higher W/Ti atoms ratio arriving to the substrate and an overall decrease of the deposition rate (see Fig. 5).

Similar evolution of the structure as a function of the N content was found in industrially deposited films in relation to those deposited by Cavaleiro et al. (2003) and Silva et al. (2005) in laboratory equipment. As in these works, Fig. 6 shows that low N content films display the b.c.c. α-W(βTi) (ICDD numbers 04-0806 and 44-1288) phase which evolves to the f.c.c. NaCl type TiN/W₂N (ICDD numbers 87-0631 and 25-1257) phase with increasing N content.

3.2. Mechanical properties

Fig. 7 compares the results obtained previously for the hardness and scratch-test critical loads of W–Ti–N films deposited with increasing N contents with those of IndDep films. There are several points to be remarked, as follows:

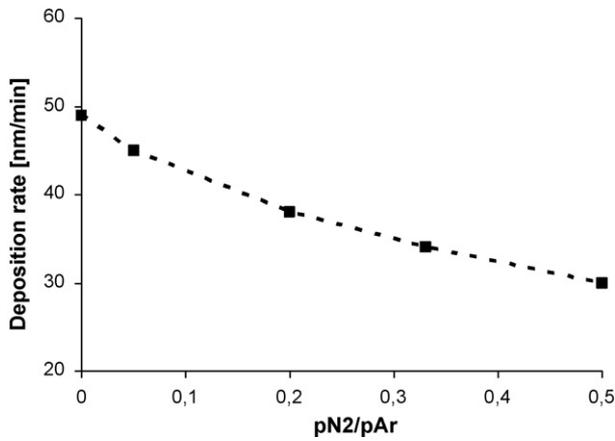


Fig. 5. Evolution of the deposition rate as a function of the partial pressure of the reactive gas for IndDep W-Ti-N coatings.

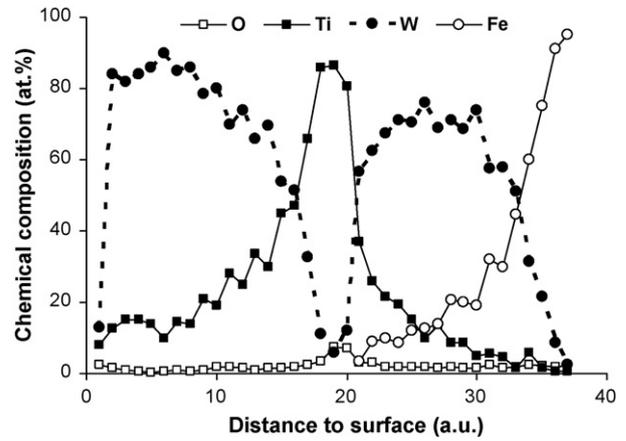


Fig. 8. Depth profiling chemical composition analysis of a W-Ti very thin film evaluated by Auger spectroscopy.

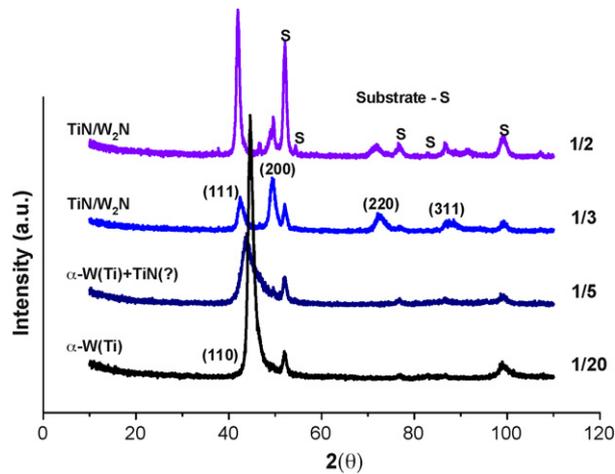


Fig. 6. XRD patterns of IndDep W-Ti-N coatings deposited with increasing partial pressure of the reactive gas ($p_{N_2}/p_{Ar} = 1/20, 1/5, 1/3, 1/2$).

• Similar trends were obtained for the hardness evolution in both series; however, globally the values are somewhat lower in industrially deposited films. As explained by Braga et al. (2006) in a previous paper, the difference can be attributed to the lower stress state level induced by a lower substrate temperature. In fact, in

industrially deposited coatings, as the substrate is not intentionally heated, the temperature is low, as referred to above less than 180 °C. Contrarily, in the laboratory deposited coatings the deposition temperature is close to 450 °C. The stresses are originated during the cooling down to room temperature due to the difference in the thermal expansion coefficients between the films and the substrates. The maximum hardness value (>35 GPa) is achieved in IndDep films for a N content a little higher than for LabDep films (40 against 35 at.% N achieved for p_{N_2}/p_{Ar} of 0.2 and 0.33, respectively).

• The critical loads are much lower in IndDep films. Recently, a detailed analysis of the interface was carried out using Auger spectroscopy analysis after consecutive surface erosion by ion bombardment. For that, a very thin film was prepared, the deposition procedure consisting in: (1) a normal ion cleaning before deposition followed by (2) a 5 s Ti interlayer and (3) a 15 s W-Ti film. Fig. 8 shows the in-depth chemical composition results. As can be concluded by the presence of W between the Ti interlayer and the substrate, it seems that during the pre-cleaning of the substrates by ion bombardment there are some atoms coming from the target able to reach the substrate. This is caused by the fact that for sustaining the discharge close to the substrate the plasma should also be ignited close to the targets (see Table 1). As a consequence, the atoms ejected in this stage are being deposited on the substrates impeding their efficient surface cleaning and contributing for a bad adhesion. A recent experiment using shutters in front of the targets showed that the adhesion problems could be solved (see isolated point in Fig. 7) by avoiding the deposition of this contamination layer. In this case, applied loads up to 70 N did not promote any chipping or failure in the coatings as confirmed in the inset micrographs.

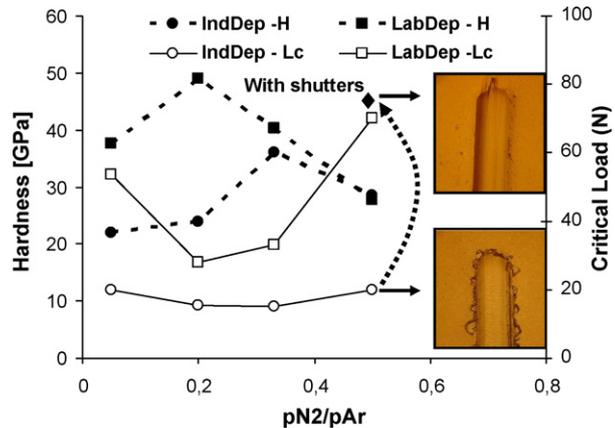


Fig. 7. Hardness (H) and scratch-test critical loads (Lc) of W-Ti-N coatings as a function of the N content. The experimental point marked with the ◆ symbol is related to a coating deposited with shutters in front of the targets. The micrographs correspond to the end of the scratch tracks.

Recently, Silva et al. (2008) tested the tribological behaviour of these coatings by pin-on-disk in dry and lubricated conditions. In both cases, the best wear and friction results were obtained for the coatings with the highest hardness, reason why they were selected for semi-industrial in-service tests, in spite of their lower scratch-test critical loads. Thus for LabDep coatings the one-fifth partial pressure ratio was selected for the deposition, whereas one-third was the value for the IndDep coatings.

3.3. Strip-draw test

3.3.1. LabDep coatings

The first set of strip-drawing tests was performed with LabDep coatings. The test was firstly applied to uncoated material in order

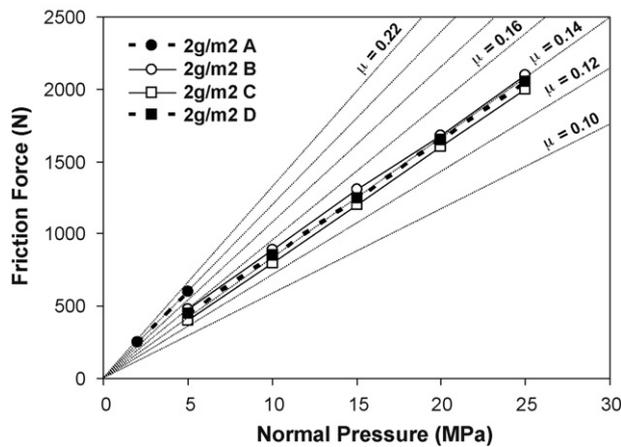


Fig. 9. Evolution of the friction force as a function of the normal applied pressure for a DDS steel sliding against an uncoated D2 steel tool.

to study the importance of the type of lubricant in the test results. The cold rolled DDS steel was used for these tests. Fig. 9 shows that even with 2 g m^{-2} of lubricant, one of the lubricant brands studied (emulsion one – A) did not permit finishing the test without adherent material is detected between the sheet and the tool. The lubricant film between the sheet and the tool breaks down and sheet particles are sticking to the tool giving rise to “adhesion”. For the same tool but now coated with the W–Ti–N ($\sim 35\text{ at.}\% \text{ N}$) film a significant improvement in the resistance to adhesion could be achieved. In fact, without lubricant or using as lubricant the A fluid, with content as low as 0.5 g m^{-2} , no adherent material was found on the coated tool, situation which is only observed with the uncoated tool when 3 g m^{-2} was used.

The influence of the lubricant type can also give rise to different performance in coated samples as it is shown in Fig. 10. It is clear from this figure either the importance of the lubrication amount in the decrease of the friction coefficient (whatever the lubricant type is), particularly for low applied pressures, or the inverse trend shown by the two tested lubricants with increasing normal applied pressure. For both tested lubricants, globally can be stated that there is a decrease of the friction coefficient with increasing amounts of applied lubricant. However, this trend is attenuated with the increase of the pressure as demonstrated for lubricant A. For high pressures, lower amounts of lubricant permit getting lower friction coefficient. It should be remarked that the coated sample behaves better (no adhesion was detected even with the highest applied load) when tested without lubrication than the bare D2 steel with 2 g m^{-2} of A lubricant.

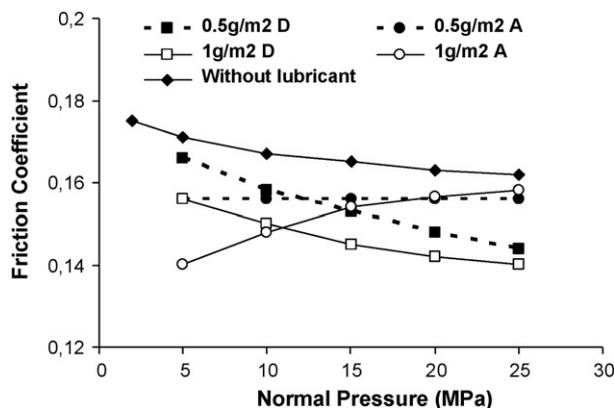


Fig. 10. Evolution of the friction coefficient as a function of the normal applied pressure for the DDS steel sliding against a W–Ti–N coated D2 steel tool.

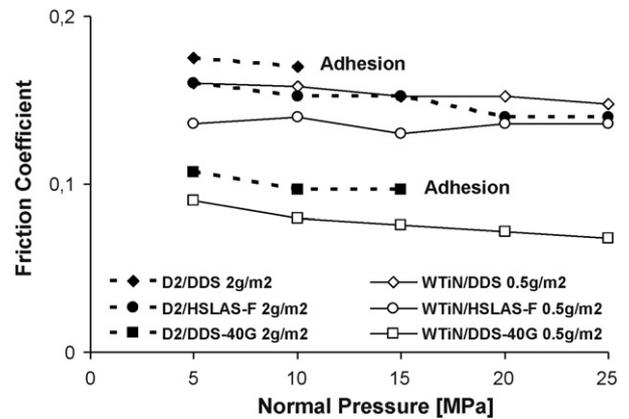


Fig. 11. Friction coefficient in lubricated contact (lubricant D) of uncoated and W–Ti–N coated D2 steel sliding against different sheet materials.

The advantage of W–Ti–N coated compared to uncoated tools can be confirmed when different materials are used as testing sheets as it is shown in Fig. 11. Even using low lubricant contents much better tribological behaviour is found in coated tools concerning the improvement of either the resistance to adherent counterbody material (“adhesion”) or the friction coefficient values. For uncoated tools, only in one case the test could be performed up to the maximum tested normal pressure without adhesion (sliding against DDS sheet). The friction values are almost similar between coated and uncoated tool but a much lower lubricant content was used in the first case (0.5 against 2 g m^{-2} , respectively).

Although not so remarkable, the behaviour of the coated tools in dry conditions is quite good. Fig. 12 summarizes the results of the friction coefficient at increasing applied loads when W–Ti–N coated D2 steel is rubbed against different materials sheets with (1 g m^{-2} lubricant D) and without lubrication. In unlubricated conditions for DDS steel, with and without surface treatment, in any loading conditions adhesion was never observed, whereas problems started to occur at 15 MPa for the HSLAS-F sheet. For the Al alloy the results were not so good since for 5 MPa , adhesion was detected in one of the performed tests. It should be remarked that in none of the case it was possible to perform the test without lubrication for the uncoated tools. In all experiments, immediately after the initial contact, there is a sudden increase of the friction coefficient with the corresponding detection of extensive adhered material.

The coated tool revealed a great efficiency in lubricated tests. With only 1 g m^{-2} of lubricant D, it was possible to perform the test up to the maximum testing loads without the detection of any signs of adhesion. Even for ductile materials, such as the Al alloy,

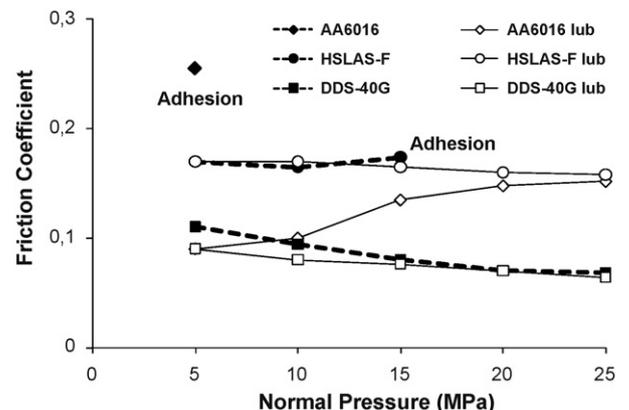


Fig. 12. Friction coefficient of different sheet materials sliding against D2 steel coated with a W–Ti–N film in dry and lubricated (lubricant D) conditions.

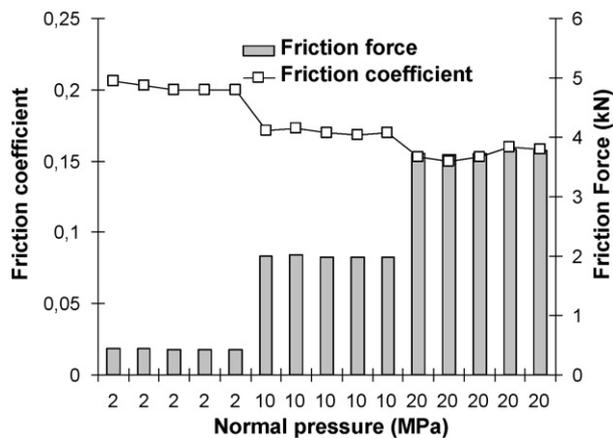


Fig. 13. Friction forces and coefficients obtained for industrially deposited W-Ti-N coated samples in strip-drawing tests performed in lubricated conditions (lubricant D) with DDS steel sheets.

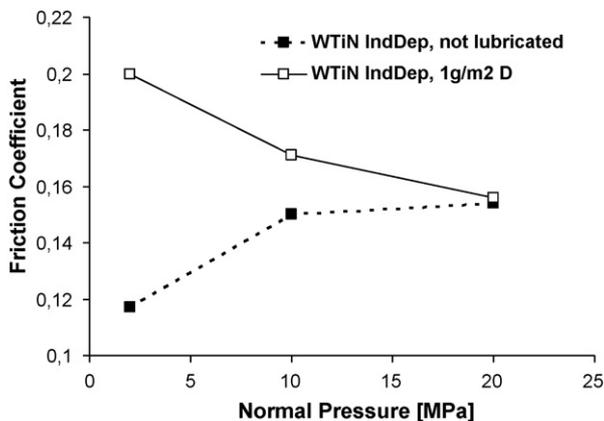


Fig. 14. Evolution of the friction coefficient as a function of the applied normal pressure for industrially deposited W-Ti-N coated samples in strip-drawing tests with DDS steel sheets.

no adhesion problems were detected for coated tools in lubricated conditions.

3.3.2. IndDep coatings

The strip-draw test was also performed in the industrially deposited W-Ti-N coatings against DDS steel sheets. Two series of tests were performed, one without and the other with 1 g m^{-2} lubricant D. Three normal pressures were applied, 2, 10 and 20 MPa. As before, five tests were performed for each loading condition. As can be observed in Fig. 13 very low dispersion was obtained in the results showing the good homogeneity of the coatings.

A quite unexpected result is concerned with the comparison between lubricated and unlubricated contact. In fact, when the global tests results are analysed (see Fig. 14) it can be concluded that, for low normal applied pressures, the friction coefficient for unlubricated tests is lower than for lubricated ones. This result is not in accordance with those achieved for laboratory deposited coatings for which an inverse trend was observed.

Excepting this antagonic behaviour, the global results are very similar between IndDep and LabDep samples. For the loads of 10 and 20 N, the friction coefficient varies in the range from 0.14 to

0.17 which is closely the same range obtained for LabDep coatings sliding against the DDS steel (see Fig. 10). These results allow concluding that the solution adopted for the industrial deposition of the coatings lead to materials which have approximately the same behaviour in semi-industrial strip-drawing tests.

4. Conclusions

W-Ti-N films were deposited in both laboratory and industrial equipments. The evolution of the chemical composition of the coatings as a function of the N content followed inverse trends which were explained by the different type of used targets in both processes. The hardness was lower for industrially deposited films which was attributed to a lower residual stress value induced by a lower deposition temperature. However, in selected conditions 35 GPa could be achieved. The scratch-test critical load was very low due to a contamination layer deposited during the substrate plasma cleaning. By using shutters this problem could be solved.

Both industrially and laboratory deposited coatings showed an excellent behaviour in strip-drawing tests. Tests without lubrication could be run without adhesion for all the steel sheet materials. Only when an Al alloy was used adhesion could be observed. With uncoated tools adhesion always occurred independently of the testing loads and antagonist sheet material. In lubricated conditions, the friction coefficients were always lower with coated tools even if the lubricant amount was reduced (0.5 against 2 g m^{-2} for the uncoated sample). The performance between industrially and laboratory deposited samples is similar in lubricated contacts. However, for industrially deposited coatings a lower friction coefficient was measured in unlubricated tests for low loads, trend inverse to the observed for laboratory deposited coatings.

Acknowledgements

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