

The influence of metallic interlayers on the adhesion of PVD TiN coatings on high-speed steel

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Abstract

In nearly all applications the adhesion of the coating to the substrate is crucial for the components performance and length of life. To enhance the adhesion it is common to use a metallic interlayer, most often titanium. In this study seven different metallic interlayers, namely W, Mo, Nb, Cr, Ti, Ag and Al, have been evaluated with respect to their influence on the adhesion of PVD TiN coatings to polished high-speed steel, ASP 2060. The purpose of this work is to investigate how some physical properties of a metal affect its capability to function as an adhesion interlayer. Samples were prepared using dc magnetron sputtering for the interlayer and reactive dc magnetron sputtering for the TiN coating. The deposition process included both pre-treatments and in situ treatments of the substrate surface in order to eliminate possible contaminations. The adhesion of the coatings was investigated with two different methods: scratch testing and Rockwell adhesion testing. The results indicate that differences in hardness between the metallic interlayers influence the practical adhesion more than differences in *E*-modulus. Furthermore, in order to optimize adhesion, the hardness of the interlayer should be close to the hardness of the substrate. It was also suggested that stresses, both in the TiN coating and in the metallic interlayer, affect the adhesion properties negatively. In addition, the necessity of interlayer in TiN on HSS can be questioned as the reference samples, without interlayer, showed adhesion properties comparable to the highest ranked interlayer containing samples in our assessment.

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1. Introduction

Due to its extreme hardness, high thermal and chemical stability and low electrical resistivity TiN is frequently used as coating material in the industry since a few decades. The field of application is wide and varies from hard protecting coatings on mechanical tools to diffusion barriers in the micro-electronics industry. Even though other coatings are present on the market today, TiN is still used in certain areas and we consider it a suitable modeling material for this study. In the majority of the applications, a crucial factor for the length of life and the performance of the coated component is the coatings adhesion to the substrate. In areas where the coated surface is exposed to mechanical wear, flaking of the coating can lead to an aggravated wear situation. The resulting effect is not only that the

underlying substrate surface becomes exposed; the flakes can also work as abrasive particles on the remaining surfaces. The desired course of event is instead a gradual wear of the coating during the tools total lifetime. A prerequisite for this is a good adhesion to the underlying material.

Mittal distinguish between three different forms of adhesions [1] namely; (i) fundamental adhesion, (ii) thermodynamic adhesion and (iii) practical adhesion. Fundamental adhesion is defined as the sum of all molecular and atomic interactions across the interface between the coating material and the substrate material. Thermodynamic adhesion signifies the change in free energy when an interface is formed (or separated). Practical adhesion is described as the force required to remove the coating from the underlying substrate irrespective to the locus of failure. In the present work the practical adhesion is the most interesting one because that is the adhesion measured experimentally.

Practical adhesion can be referred to as a function of fundamental adhesion and ‘other factors’. There are numerous such ‘other factors’, e.g. stresses in the coating, thickness and

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mechanical properties of the coating, mechanical properties of the substrate, deformation work consumed by plastic deformation and viscous dissipation, mode of failure and the mode and rate of applying the force or the energy to detach the coating, i.e. the technique used for adhesion measurement. In other words, it is not only physical, mechanical and chemical features of the coating-substrate system that contribute to the result of an adhesion measurement but also the measurement technique itself. This is important to keep in mind when discussing measured adhesion properties. Accordingly, it is always best to use the adhesion measurement technique whose mode and rate of applied force best corresponds to the situation in the intended application.

In order to improve adhesion there are mainly three things to aim for:

- Low energy in the interface; which is related to the microstructural match, rather than miss-match, between substrate and coating.
- The creation of strong and stable chemical bonds between substrate and coating.
- A low stress gradient in the interface; which improves the ability to sustain externally imposed shear stresses in the interface. Detrimental gradients can, for example, arise from differences in thermal expansion of substrate and coating.

Several techniques are used for improving adhesion. These can be divided into three different categories: (i) pre-treatment—cleaning of the substrate prior to insertion in the deposition chamber, (ii) in situ treatment—pre-heating and sputter cleaning of the substrate, and (iii) interlayer deposition—a thin layer of a material, usually a metal, deposited between the substrate and the coating. The idea of pre-treatment and in situ treatment is to remove contaminations, such as grease, oxides, etc., from the substrate surface to increase surface reactivity that facilitates the formation of strong chemical bonds. The purposes of applying an interlayer are to minimise stresses in the interface and/or to dissolve contaminations.

To improve the adhesion of TiN and similar coatings it is common to use a thin layer of titanium or in some cases chromium as an interlayer. The reason for using these metals is to a large degree process related in that it is convenient to build a nitride and a metallic interlayer using the same metal. But to be fair, titanium does have good properties in terms of governing adhesion which has been explained by two factors [2]: (i) titanium,

which is a reactive metal, dissolves surface contaminants such as oxides, (ii) an interlayer of titanium can lead to an improved match between the mechanical properties of the substrate and coating, which minimises the stress gradient in the interface.

This paper presents a study of seven different metallic interlayers and aims to investigate their abilities to work as adhesion enhancing interlayers. Modern PVD processes most often involve thorough cleaning which makes the requirement of contaminant solubility questionable. The work was performed with the intentions to facilitate the development and successful deposition of new coating materials, which may be prone to adhesive failure, and to look at the possibility to further improve the adhesion of TiN. The metals investigated were W, Mo, Nb, Cr, Ti, Ag and Al and they were chosen to cover a large variation in properties such as hardness, heat expansion, *E*-modulus and crystal structure.

2. Experimental

2.1. Materials

The substrates used were high-polished plates (20 mm × 20 mm × 3 mm) of ASP2060, a high-speed-steel (HSS) produced by ERASTEEL Kloster AB. HSS was chosen as substrate since it is a common material to be coated with PVD coatings in industrial applications. The interlayer metals chosen originate from the groups 4, 5, 6, 11 and 13 in the periodic table. All but one (Al) belongs to the transition metals of the d-block. Three of these: Cr, Mo and W, are all in group 6, i.e. they have the same number of electrons in their outer shell. Although their crystal structure and thermal expansion are roughly the same their mechanical properties vary considerably. Ti, Nb, Cr, Mo and W are all relatively hard metals, and in contrast to these also two soft metals, Ag and Al, were included. In Table 1 some properties of the metals are summarised.

Each sample consisted of a HSS substrate on which a 100 nm thick metallic interlayer was deposited by dc magnetron sputtering, whereupon a 3 μm thick TiN coating was deposited by reactive dc magnetron sputtering.

2.2. Coating deposition

The coatings were deposited in a Balzers BAI640R system, equipped with two planar magnetrons, one rectangular and one circular. Furthermore, the system is equipped with an arc posi-

Table 1
Compilation of the metals used as interlayers and some of their properties [3,4]

Material	Hardness (GPa)	<i>E</i> -modulus (GPa)	Crystal structure	Heat expansion coefficient (K ⁻¹)
W	33.6	411	bcc <i>a</i> ≈ 31.6 Å	4.5 × 10 ⁻⁶
Mo	15	329	bcc <i>a</i> ≈ 31.5 Å	5.0 × 10 ⁻⁶
Nb	13	105	bcc <i>a</i> ≈ 30.3 Å	7.1 × 10 ⁻⁶
Cr	10.4	279	bcc <i>a</i> ≈ 29.1 Å	8.5 × 10 ⁻⁶
Ti	9.5	116	hcp <i>a</i> ≈ 29.5 Å, <i>b</i> ≈ 46.8 Å	8.5 × 10 ⁻⁶
Ag	2.5	83	fcc <i>a</i> ≈ 40.8 Å	19.2 × 10 ⁻⁶
Al	1.6	70	fcc <i>a</i> ≈ 40.5 Å	23.3 × 10 ⁻⁶

Table 2
The nine samples with interlayer and top-coating

	Sample								
	TiN ^R	TiN ^C	W	Mo	Nb	Cr	Ti	Ag	Al
Interlayer	–	–	W	Mo	Nb	Cr	Ti	Ag	Al
TiN coating	TiN ^R	TiN ^C	TiN ^R	TiN ^C	TiN ^C	TiN ^R	TiN ^C	TiN ^C	TiN ^C

tioned in one of the chamber walls, which was used to increase the degree of ionization in the plasma. Prior to coating deposition, the substrate was washed with an alkaline detergent and ethanol in ultrasonic baths. The substrates were mounted on a rotating substrate holder placed above the evaporation sources. The chamber was evacuated and the substrates were heated to 300–400 °C and sputter etched in an argon-plasma, all in order to remove residual surface contaminations. A 100 nm thick interlayer was deposited by sputtering a metal target mounted on one of the magnetrons. A substrate bias of –110 V, an arc current of 80 A and an argon pressure of 2.5×10^{-3} mbar were used during this deposition. A 3 μm thick TiN coating was then deposited, by sputtering a Ti target mounted on the other magnetron while a nitrogen flow of 50 sccm was introduced in the chamber. During this, the substrate bias was set to –50 V, the arc current to 80 A and the total pressure to 4×10^{-3} mbar. Since not all interlayers were deposited using the same magnetron, simply dictated by the availability of different targets, neither were all TiN coatings. Given that the magnetrons have different geometries and characteristics, different sputter parameters had to be used depending on which magnetron was operated during the TiN deposition in order to receive as identical TiN coatings as possible. Having said that, the two magnetrons result in TiN coatings with almost identical Ti–N ratios, 1.016 for the rectangular and 1.018 for the circular. The hardness of the produced TiN coating is 26 ± 4 GPa when the rectangular magnetron is used and 32 ± 4 GPa when the circular is used. The *E*-moduli of the TiN coating is 390 ± 50 and 470 ± 50 GPa, respectively. The resulting deposition rate was also higher (roughly 65%) when the rectangular target was used. All metals used as interlayers were sputtered using a power of 3 kW, except Ag where the equipment limited the maximum power to 2.25 kW. Nine samples were made, see Table 2, of which two were reference samples without interlayers. TiN^R is produced using the rectangular magnetron and TiN^C is pro-

duced using the circular magnetron. The other seven samples are labelled after the metal used as interlayer.

2.3. Adhesion testing

The coatings adhesion to the HSS substrates was evaluated with two different kinds of adhesion measurement techniques: scratch test and Rockwell adhesion test. Scratch test is a generally accepted way to assess adhesion of hard coatings in both industry and research. The Rockwell adhesion test [5] is a method developed by the Union of German Engineers (Verein Deutscher Ingenieure, VDI) for evaluation of thin coatings.

The instrument used for the scratch test was a *CSEM Revetest*. The coated surface is scratched with a conical Rockwell C tip with a tip radius of 200 μm . The transversal load is continuously increased with 10 N/mm, from 0 to 100 N, which results in a 10 mm long scratch. On each sample two scratches were made and studied with a FEG-SEM LEO 1550 in order to find critical loads for adhesion failure. It turned out that it was necessary to define two different critical loads in order to give an accurate description of the adhesion failures. The first critical load, L_1 , describes at which load the first sporadic isolated adhesion failure took place, i.e. an adhesion failure that is discrete, see Fig. 1a. The second critical load, L_2 , describes at which load a more continuously spread adhesion failure begins, i.e. the onset of an adhesion failure that propagates as the load increases, see Fig. 1b.

The Rockwell adhesion test was performed with a conventional hardness tester (VPM 308) equipped with a Rockwell C tip, with tip radius 200 μm , which was pressed through the coating using a high load. Two indents at two different loads, 120 and 187.5 kg, were made and the indents were studied with SEM and classified into one of six classes, shown in Fig. 2. Class HF 1 is characterised by a small amount of

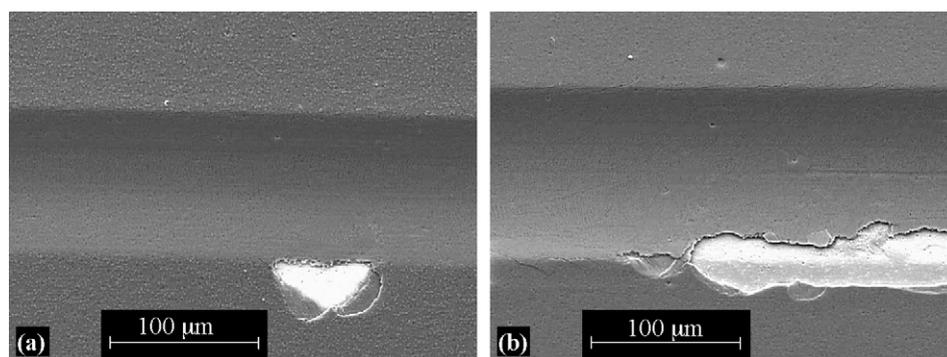


Fig. 1. Typical appearances of adhesion failures defined by critical load L_1 (a) and L_2 (b), respectively.

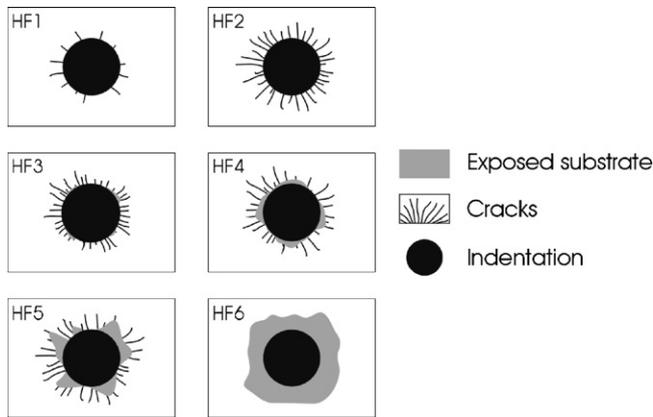


Fig. 2. Sketches of the six classes used for classification of indents in the Rockwell adhesion test, after [5].

cracks in the coating around the indentation while class HF 2 shows a higher density of cracks. In class HF 3 small parts of the coating have been detached. The extent of detached coating material increases through classes HF 4 to HF 5, while all coating material surrounding the indentation is gone in class HF 6.

3. Results

3.1. Coating morphology

All deposited TiN coatings have a columnar structure similar to the one shown in Fig. 3. The thicknesses of the TiN coatings were $3.1 \pm 0.5 \mu\text{m}$ and the thicknesses of the metallic interlayers were all within the range 100–150 nm.

3.2. Scratch testing

Two identical scratches were made in each sample. The critical loads for adhesion failures caused by scratching are shown in Table 3. These loads represent adhesion related failures. Minute cracks were occasionally seen at lower loads but as they are related to the cohesive strength of the TiN coatings they are not of interest in this work.

Mo and Nb showed only sporadic adhesion failures, whereas Cr, Ti, Ag and Al all showed continuously spread adhesion failures, see Fig. 4. Mo and Nb are the samples that generally showed the best adhesion. The failures of these latter samples are more

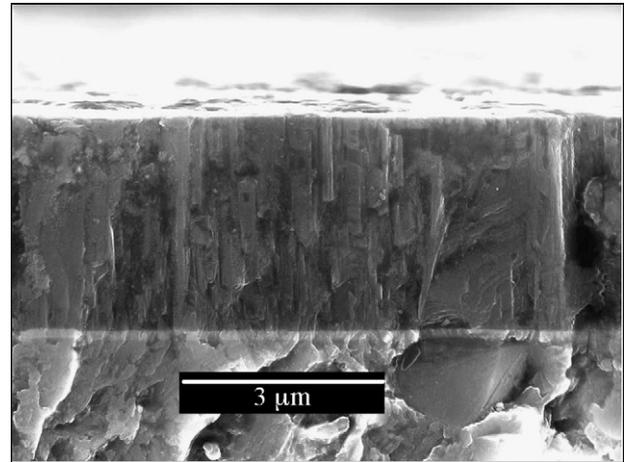


Fig. 3. A cross-section of the sample Mo showing a representative columnar structure.

of a sporadic nature and occur at relatively high critical loads. The critical load for Ti was only somewhat lower however the adhesion failure was more continuously spread. A similar result was found for Cr. Ag and Al both showed extended adhesion failures beginning at low critical load. On sample Ag the coating was detached in the form of long flakes travelling far away from the scratch during scratching. For this reason only one scratch could be made in the Ag sample. This failure mode was not present in the case of Al. W is the only sample for which both critical loads could be defined. First, sporadic adhesion failures took place at moderately low loads, which although were much higher than the critical loads for Ag and Al, then changing to more continuously spread failures at somewhat higher loads.

The critical loads for the samples without interlayers, TiN^R and TiN^C, are not identical. For TiN^R the critical load is continuously spread and of the same size (and type) as the critical loads for Ti and Cr. On TiN^C only sporadic adhesion failures are seen, the type and average value of the critical load is comparable to the critical loads for Mo and Nb.

3.3. Rockwell adhesion test

The indents in the samples without interlayer as well as the indentations in Mo, Nb, Cr and Ti, all caused a certain amount of cracks but no spallation of the coating; see Table 4 and Fig. 5. Nb showed a somewhat smaller amount of cracks around the

Table 3
Critical loads from scratch testing for each sample

	Sample								
	TiN ^R	TiN ^C	W	Mo	Nb	Cr	Ti	Ag	Al
Scratch 1									
L ₁ (N)	–	92	52	86	87	–	–	–	–
L ₂ (N)	83	–	56	–	–	84	83	20	24
Scratch 2									
L ₁ (N)	–	80	60	87	85	–	–	–	–
L ₂ (N)	80	–	70	–	–	77	80	–	18

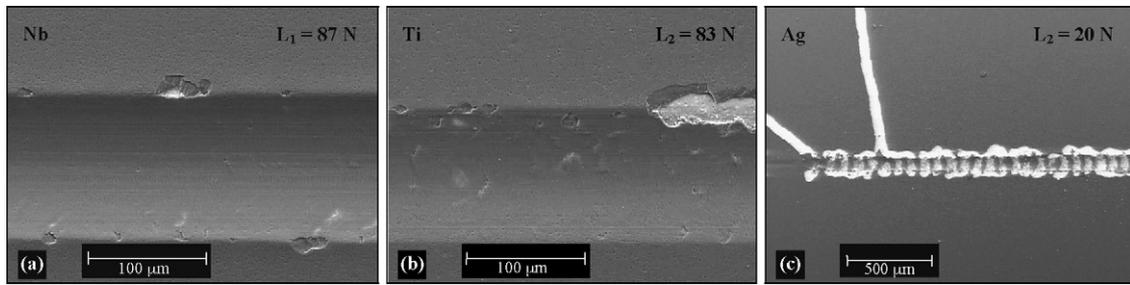


Fig. 4. Representative images showing different kinds of adhesive failures. Local failure corresponding to L_1 is shown for Nb (a) and the onset of continuously spread adhesion failures corresponding to L_2 are shown for Ti (b) and Ag with associated long range spallation (c).

Table 4
Results from the Rockwell adhesion test

	TiN ^R	TiN ^C	W	Mo	Nb	Cr	Ti	Ag	Al
120 kg	HF 2	HF 2	HF 1	HF 2	HF 1	HF 2	HF 2	HF 6	HF 4
187.5 kg	HF 2	HF 2	HF 6	HF 2	HF 2	HF 2	HF 2	HF 6	HF 5

indent made with the lower load than around the indent done with the higher. No such difference could be seen between the indentations in the samples without interlayer, Mo, Cr and Ti. On Ag, extended spallation of coating could be seen around both indents, see Table 4 and Fig. 5. Delamination occurred also around the indents made on Al but to a more limited extent.

Here, the higher load caused more delamination than the lower. The biggest difference between the two loads is seen on W. The lower load caused no adhesion failures what so ever, in fact hardly any cracks were induced, whereas a large area of the coating was detached around the indentation made with the higher load.

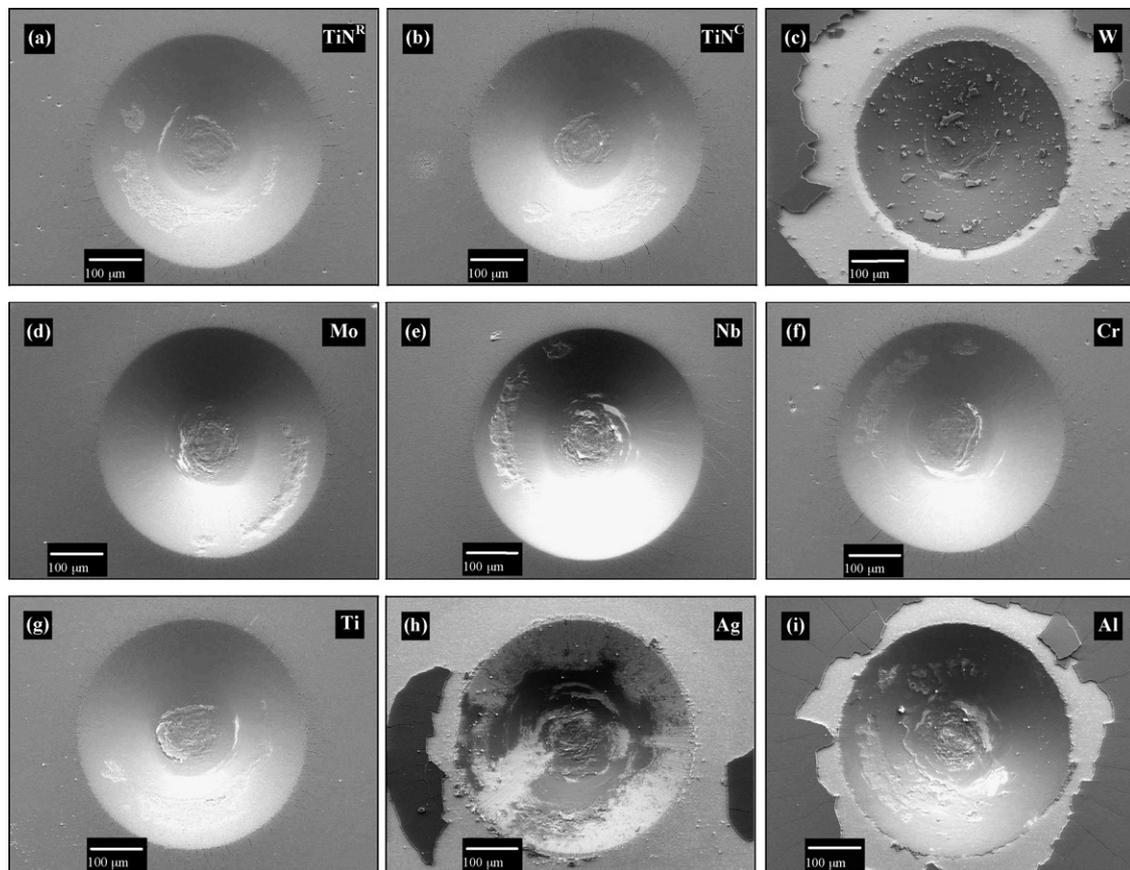


Fig. 5. Indentations made with 187.5 kg load in TiN^R (a), TiN^C (b), W (c), Mo (d), Nb (e), Cr (f), Ti (g), Ag (h) and Al (i). A certain amount of cracks can be seen on the edges of the indents in TiN^R, TiN^C, Mo, Nb, Cr and Ti but no spallation of the coatings. On the samples W, Ag and Al can different degrees of coating spallation be seen suggesting poor adhesion.

4. Discussion

The hardness of each produced TiN coating was measured with nanoindentation. The hardness was roughly the same, varying within the margin of errors of the TiN coatings deposited without interlayers, and without any trends considering presence and type of interlayer.

Concerning the results from the scratch test it is notable that TiN^R and TiN^C, without interlayers, show different behaviours which results in different types of critical loads. This indicates that the TiN produced with the two different magnetrons are not completely identical and therefore it is not possible to strictly compare the samples with each other. However, as the behaviours are apparently not directly inherited by the samples produced with interlayers, the comparison is done with this as a conceivable source of error.

The thicknesses of the interlayers are all within the range of 100–150 nm. No attempt was made to optimize these thicknesses, in order to maximise the adhesion of the different coating systems.

The samples containing interlayers can be divided into four different groups where the samples placed in group 1 show the best adhesion results in the adhesion tests while the samples placed in group 4 show the worst. This is illustrated in Fig. 6a, showing the result from the high load Rockwell adhesion versus the mean value of the critical loads in the scratch testing. The adhesion is thus better the closer you get to the upper right corner in the figure. In group 1 the samples Mo and Nb are placed, showing good adhesion in both scratch test and Rockwell adhesion test. Ti and Cr show equally good Rockwell adhesion but in the scratch test their critical loads are somewhat lower and they are thus placed in group 2. In group 3 the sample W is placed showing a poor adhesion in the scratch test and an exceptionally poor adhesion in the Rockwell test at the higher load. Its very good performance at low load is not shown in the figure. In group 4 Ag and Al are placed which show extremely poor adhesion in both the scratch test and the Rockwell test.

It becomes clear that the samples placed in groups 1 and 2 have superior adhesion properties than the rest. The credibility of the results is reinforced by the fact that the conventionally used interlayers Ti and Cr are found among these. The samples without interlayer are not shown in the figure but they would be placed in the same area as groups 1 and 2. Since the samples without interlayer show comparable adhesion to the highest ranked samples in this comparison the necessity of interlayer deposition can be questioned. As mentioned earlier in the paper, the purposes of applying an interlayer are to minimise stresses in the interface and/or to dissolve contaminations. The fact that efficient pre-treatments and in situ treatments of the substrate surface have been used in this work, which is also the case in PVD processes of today, contaminants are to a large extent eliminated prior to coating deposition. In deposition processes with inadequate pre- and in situ treatments the ability of an interlayer material to dissolve possible surface contaminants can still be crucial for the adhesion.

In a “clean” process, however, the function of an interlayer is reduced to minimise stresses in the interface. Such stresses can be growth or temperature related but can also arise due to differences in mechanical properties between substrate and coating when the system is exposed to external strain.

Fig. 6b shows the metals *E*-modulus versus hardness. The values are obtained from literature as these are impossible to measure directly on the interlayers. The properties of the TiN, measured with nanoindentation, and the ASP 2060 are also shown in the figure. Mo and Nb, which are the interlayers that gave the best adhesion, have hardness values that are quite similar but their *E*-moduli lie far apart. An analogous relationship is seen for the metals placed in group 2, i.e. Ti and Cr. W has considerably higher values of both hardness and *E*-modulus which probably is the reason why it ends up in a group of its own. Tungsten's high hardness and brittleness make the metal sensitive for overload as fracture occurs shortly after its yield strength is exceeded. This explains why the W sample shows such a remarkable difference in adhesion in the Rockwell test at the two loads.

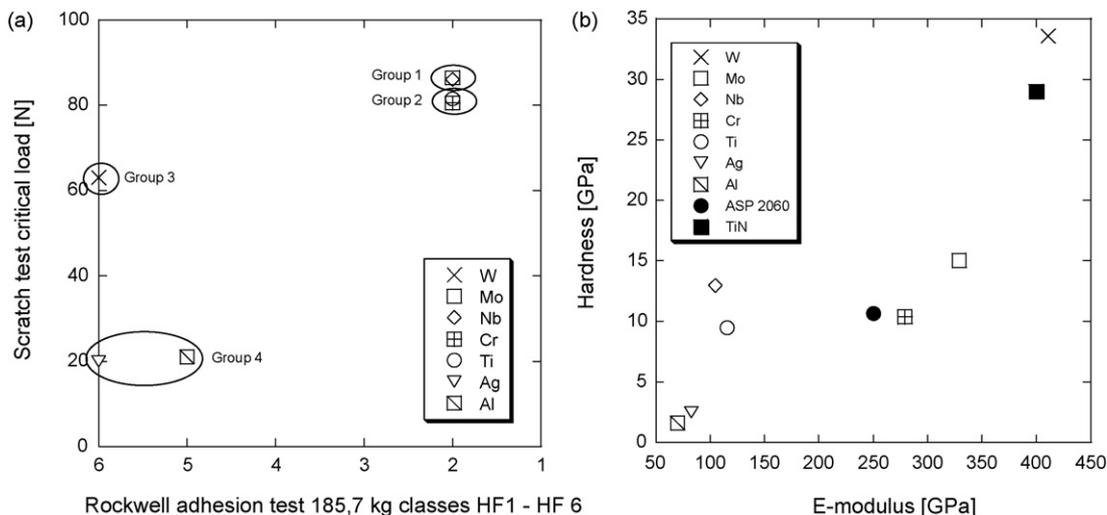


Fig. 6. (a) Compilation of the results from the adhesion test for each sample. The x-axis represents the result from the high load Rockwell test and the y-axis the mean value of the critical loads in scratch testing. (b) *E*-modulus vs. hardness shown for each sample plus the ASP 2060 substrate and TiN coating.

Table 5
Properties [4] and stresses for the different interlayers

Interlayer	E (GPa)	$\Delta\alpha$ ($\times 10^{-6} \text{ K}^{-1}$)	ν	σ (GPa)	σ_y (GPa)	σ/σ_y (%)
W	411	−5.48	0.28	−0.86	11.21	−8
Mo	329	−4.98	0.31	−0.65	5.00	−13
Nb	105	−2.88	0.40	−0.14	4.32	−3
Cr	279	−1.48	0.21	−0.14	3.47	−4
Ti	116	−1.48	0.32	−0.07	3.17	−2
Ag	83	9.23	0.37	0.33	0.82	41
Al	70	13.33	0.35	0.39	0.55	72

The two metals Ag and Al, that indisputably cause the worst adhesion, are both much softer than the rest. All this suggests that it is the hardness that is the limiting factor in this comparison. It also emerges from the figures that the metals placed in group 1 in Fig. 6a have hardness values that lie between those for the substrate and the TiN coating.

The residual stresses in some selected TiN coatings were calculated with the $\sin^2 \psi$ -method from XRD measurements [6]. The stresses obtained were $\sigma_{\text{Nb}} = -3.1 \pm 0.1$ GPa, $\sigma_{\text{Ti}} = -6.3 \pm 0.2$ GPa and $\sigma_{\text{Ag}} = -4.3 \pm 0.1$ GPa. To some extent the amount of stresses correlate with the results from the adhesion testing assuming a higher residual stress affects the adhesion properties negatively. The sample Nb, which shows the best adhesion of the three, does indeed contain the lowest amount of stresses. However, the residual stress in the sample Ti is significantly higher than the stress measured in the sample Ag even though Ag shows a considerably worse adhesion than Ti. Our interpretation of this is that the amount of residual stress in the TiN coating becomes decisive once the mechanical properties of the interlayer are sufficiently good. However the effects of residual stresses in the TiN can easily be smaller than the effects of inadequate mechanical properties of the interlayer.

Another factor that can influence the adhesion is stresses arising in the interlayer. Such stresses are either growth related or temperature related. Growth related stresses are impossible to assess for these thin interlayers but will likely be similar to those of the top coating. The temperature related stresses are governed by differences in heat expansion coefficients presented in Table 1 for the different metals. These should be compared to the heat expansion of the substrate, $10.6 \times 10^{-6} \text{ K}^{-1}$, and the one for TiN, $9.35 \times 10^{-6} \text{ K}^{-1}$. Comparing these, the heat expansion of the substrate and the TiN are quite similar and none of the metallic interlayers would improve the situation in this respect. Since the TiN coating and the substrate have roughly the same thermal expansion, the tension introduced in the interlayer by differences in thermal expansion coefficients within the system can be approximated by Eq. (1):

$$\sigma = \frac{E\Delta\alpha\Delta T}{1 - \nu} \quad (1)$$

E is the E -modulus and ν is the Poisson's ratio of the interlayer, see Table 5. $\Delta\alpha$ is the difference between the thermal expansion coefficient of the interlayer and that of the rest of the system approximated by the mean value of the coefficients for the substrate and the TiN coating. ΔT is the difference between deposition temperature and room temperature, 275 K. All data

for the metals are obtained from literature [4]. In order to estimate how large effect the introduced thermal stresses have on each interlayer they are compared to the yield strengths of the metals which are approximated to one third of the hardness. According to the calculations small compression stresses are introduced in the majority of the interlayers. The highest compressive stress is introduced in W but with respect to tungsten's high yield strength the stress level is relatively low, only 8% of the yield strength. In Mo, the introduced thermal stress comes to 13% of the yield strength but even this is very low. In Ag and in particular in Al the situation is worse; in both interlayers tensile stresses are induced instead of compression stress, amounting to 41% of the yield strength for Ag and 72% of the yield strength for Al. No clear relation between thermal stress and adhesion can be seen comparing W, Mo, Nb, Cr and Ti. This is probably because the introduced compression stresses are relatively low. The high tensile stresses introduced in Ag and Al can, on the other hand, be one of the reasons for the poor practical adhesion that these two metals lead to.

As long as we strictly consider physical properties of the interlayer versus the substrate and the coating we consider the line of argument and subsequent general conclusions to be valid for all Ti-based coatings as well as for coatings of other types. It should although be remembered that the interlayer metals have different abilities to form strong chemical bonds to both the substrate and the coating elements. This is certainly important for both the fundamental and the practical adhesion but these effects have not been investigated in this work.

5. Conclusions

The following conclusions are made regarding how the metallic interlayers W, Mo, Nb, Cr, Ti, Ag and Al affects the practical adhesion of TiN to the high-speed-steel ASP 2060, when evaluated with scratch testing and Rockwell adhesion testing:

- Mo and Nb both give the best adhesion closely followed by the more conventionally used Ti and Cr.
- The elastic modulus of the metallic interlayer does not affect the adhesion as much as its hardness.
- In order to get good adhesion the thermal expansion coefficient of the interlayer should not deviate too much from the thermal expansion coefficient for the rest of the system.
- The amount of residual stress in the TiN coating becomes decisive once the mechanical properties of the interlayer are sufficiently good. In our case the effects of residual stresses in

the TiN were smaller than the effects of mechanical properties of the interlayer.

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