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MECHANICAL PROPERTIES OF CrN/Cr COATINGS WITH DIFFERENT THICKNESSES

MEHANSKE LASTNOSTI PREVLEK CrN/Cr RAZLIČNIH DEBELIN

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Prior to the deposition of CrN coatings, a thin chromium layer is usually deposited to enhance adhesion. In this study, several CrN coatings of different thicknesses (0.8–4.8 µm) were deposited by reactive sputtering in a Balzers Sputron deposition system at a substrate temperature of about 150 °C. Polished tool steel disks were used as substrates. Up to three interlayers of chromium were included in the coatings, while the deposition parameters were kept constant for all the coatings. After each deposition run (Cr+CrN), the mechanical properties were measured.

The influence of coating thickness on mechanical properties was evaluated. Vickers microhardness was measured by a static indentation technique as well as by dynamic, stepwise loading. The adhesion to the substrate was measured by scratch test. There was no influence of thickness on the adhesion – the coatings adhered well to the substrate except for the thinnest one (0.8 µm). By measuring the curvature of the coated samples we were able to evaluate the internal stress, too. A decrease of internal stress for consequently deposited thicker coatings was observed.

Keywords: reactive sputtering, PVD coatings, CrN, microhardness, internal stress

Pred nanosom CrN-prevleke se po navadi nanese tanko plast kroma za poboljšanje adhezije. V tem prispevku poročamo o CrN-prevlekah različnih debelin (0,8–4,8 µm), ki smo jih nanesli z reaktivnim naprševanjem v napravi Balzers Sputron pri temperaturi podlag okoli 150 °C. Za podlage smo uporabili polirane jeklene ploščice. Do tri vmesne plasti smo vključili v prevleke, medtem ko so bili pogoji nanosa enaki za vse prevleke. Po vsaki šarži (Cr+CrN) smo izmerili mehanske lastnosti vzorcev.

Ovrednotili smo vpliv debeline prevleke na mehanske lastnosti. Mikrotrdoto po Vickersu smo merili s statičnim obremenjevanjem kakor tudi z dinamičnim obremenjevanjem po korakih. Adhezijo prevlek smo merili z metodo razenja. Debelina prevleke ni imela nobenega vpliva na adhezijo – vse preveleke so se dobro oprijemale podlage z izjemo najtanjše (0,8 µm). Z merjenjem ukrivljenosti prekritih vzorcev smo izračunali notranje napetosti v prevleki. Opazili smo zmanjšanje notranjih napetosti v debelejših prevlekah.

Ključne besede: reaktivno naprševanje, PVD-prevleke, CrN, mikrotrdota, notranje napetosti

1 INTRODUCTION

Among many ways to enhance the mechanical properties of hard coatings, one is to deposit multilayer structures. A simple way is to produce metal/metal nitride multilayers. Unlike other multilayer systems (e.g. TiN/NbN) just one target is needed and the film sequence is dictated only by switching the reactive gas inlet. The first step in this regard is an interlayer of metal between the steel substrate and the hard coating, which is widely used to enhance adhesion [1]. One of the first metal nitride/metal multilayers studied is the TiN/Ti system [2]. There are also reports on the CrN/Cr system [3,4].

The main advantage of such a sequence is an increase of fracture toughness while retaining the hardness. The soft (metallic) layers act as barriers for crack propagation[2]. Another advantage is the relief of internal stress, which enhances adhesion. For multilayers with individual layer thickness in the range of 10 nm, an increase of microhardness has been reported [3]. In this paper, several CrN coatings were deposited with various numbers of Cr-interlayers. Three basic mechanical properties were measured (internal stress, adhesion and microhardness) and a comparison was done between single and multilayer coatings.

2 EXPERIMENTAL

All coatings were deposited by reactive sputtering (PVD) in a plasma beam sputtering apparatus (Sputron, Balzers) at a deposition temperature of approximately 150 °C. Details of the deposition procedure are described elsewhere [5]. AISI D3 tool steels were used as substrates which were polished to R_a = 0.01 µm prior to deposition, while for internal stress measurements Si (111) wafers were used as substrates. The depositions lasted for 1, 2 and 3 hours, respectively. For the first 10 minutes, as a standard procedure, pure chromium was deposited in order to enhance adhesion. After deposition the mechanical properties of the samples were measured and afterwards some of the samples were returned to the deposition chamber for another run (**Figure 1**). Altogether, six different coatings were prepared (**Table 1**). The approximate deposition rate was 0.8 µm/h. As the chromium interlayers were very thin, their thickness

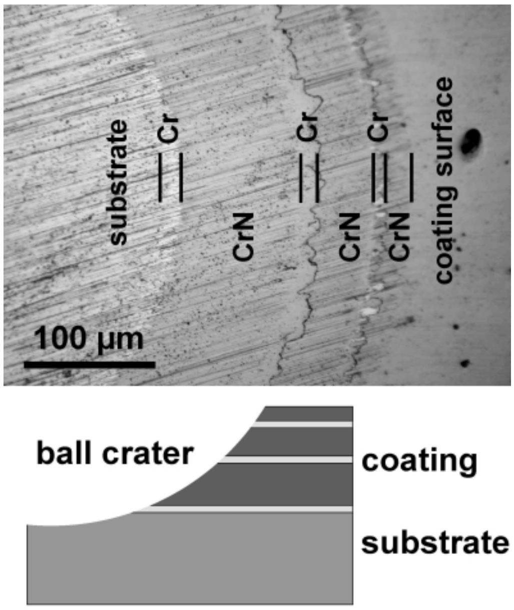

Figure 1: Ball crater of the 6-layer CrN/Cr coating
Slika 1: Krogelni obrus 6-plastne prevleke CrN/Cr

Table 1: Deposition times and thickness of the coatings analysed
Tabela 1: Časi nanosa in debelina analiziranih prevlek

total deposition time	coating thickness	deposition sequence
1 h	0.8 µm	Cr (10') / CrN (50')
2 h	1.6 µm	Cr (10') / CrN (110')
3 h	2.4 µm	Cr (10') / CrN (170')
4 h	3.2 µm	Cr (10') / CrN (170') / Cr (10') / CrN (50')
5 h	4.0 µm	Cr (10') / CrN (170') / Cr (10') / CrN (110')
6 h	4.8 µm	Cr (10') / CrN (170') / Cr (10') / CrN (110') / Cr (10') / CrN (50')

was hard to evaluate in the ball craters, so only a rough estimate can be given, i.e. around 0.2 µm (**Figure 1**).

Microhardness was measured with two different testers. A Mitutoyo MVK-H2 apparatus was used for conventional measurements, using static loading and visual evaluation (optical microscope, magnification 400×). Seven different loads were applied: (0.25, 0.5, 1, 2, 5, 10 and 20) N. Using the Fischerscope H100 apparatus, the load is stepwise increased and conversely decreased [6]. Maximum loads applied were (30, 100, 250, 500 and 1000) mN. For both testers, a Vickers diamond indenter was used.

The adhesion was measured by a Revetest (CSEM) scratch tester (loading rate 100 N/min, scratching rate 10 mm/min, loading range 0-100 N). The internal stress was evaluated by measuring the curvature of the sample with a Taylor-Hobson Talysurf 2 profilometer. The thickness of the coatings was measured by the ball crater technique.

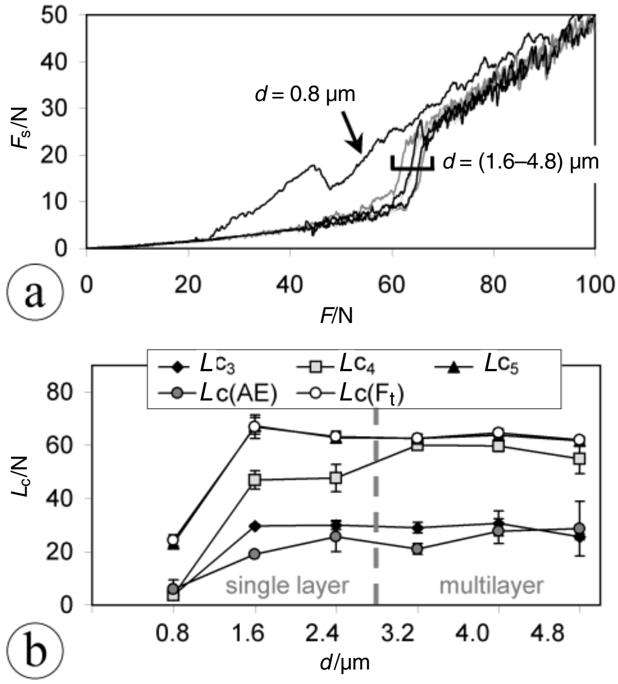

Figure 2: Adhesion of the coatings: a) scratching force F_s as a function of increasing load, b) critical loads *Lc* for various coatings
Slika 2: Adhezija prevlek: a) sila razenja F_s v odvisnosti od naraščajoče obremenitve, b) kritične sile *Lc* za različne prevleke

3 RESULTS AND DISCUSSION

3.1 Adhesion

Figure 2a shows the scratching force as a function of load during scratch testing. The curves for various samples (coating thickness 1.6 µm up to 4.8 µm) are very similar – the scratching force jump appears within a range of (60–65) N for all the coatings. The only curve substantially different from the rest is the one from the thinnest sample (coating thickness 0.8 µm).

Three scratches were done on each sample to get basic statistics. Besides the above mentioned critical load for the scratching force jump – $Lc(F_t)$, four other critical loads were evaluated: flaking on the scratch edge (Lc_3), partial delamination of the coating (Lc_4), total delamination of the coating (Lc_5), as well as the onset of acoustic emission – *Lc*(AE). The latter was detected by an ultrasound sensor while the remaining critical loads were evaluated by optical examination of the scratch track. The critical loads for the scratching force jump ($Lc(F_t)$) and the total delamination of the coating (Lc_5) are essentially identical.

The critical loads are presented in **Figure 2b**. The inferior adhesion of the 0.8 µm thick coating is obvious from all five parameters. The differences among the other coatings are within the standard error. The only recognizable difference is a lower Lc_4 value for single layer coatings as compared to the multilayer ones.

3.2 Internal stress

The internal stress (σ) was measured from the curvature of the samples. If the coating thickness is sufficiently lower than the thickness of the substrate, Stoney's formula can be applied [7]:

$$\sigma = \frac{1}{6R} \frac{d_s^2}{d_c} \frac{E_s}{1-\nu_s} \quad (1)$$

R is the sample's radius of curvature, d_c and d_s are the coating and substrate thicknesses, respectively, E_s is the substrate Young's modulus and ν_s the substrate Poisson's number. Note that no coating elastic constants are needed.

Figure 3a presents the radius of curvature for samples of different thicknesses (for convenience, the inverse is plotted). The curvature of plain substrates is negligible (R > 15 m). The inverse of the radius of curvature linearly increases with the coating thickness, however, there is a smaller slope for the multilayer coatings. This behaviour is more visible in **Figure 3b**, where internal stress for all coatings is plotted. Although the internal stress does not differ for more than ±15%, a negative trend for multilayer coatings is apparent compared to the single layer coatings. This can be attributed to partial relaxation of the stress by intermediate chromium layers.

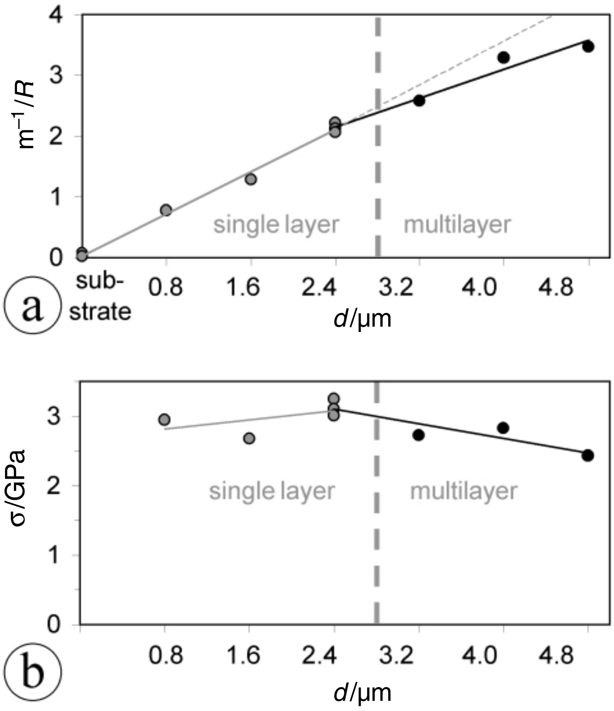

Figure 3: a) Radius of curvature and b) internal stress for various coatings

Slika 3: a) Krivinski radij in b) notranje napetosti različnih prevlek

3.3 Microhardness

The main problem for microhardness measurement of thin coatings is the substrate influence. For static measurements the load must be high enough to produce an indentation easy to measure using an optical microscope. Usually this requirement extends the plastic deformation zone into the substrate, thus the result is a combination of coating and substrate microhardness. Using dynamic loading by measuring the stress-strain curve, the load can be sufficiently lowered to keep the plastic deformation zone within the coating. However, the error of the measurement greatly increases by

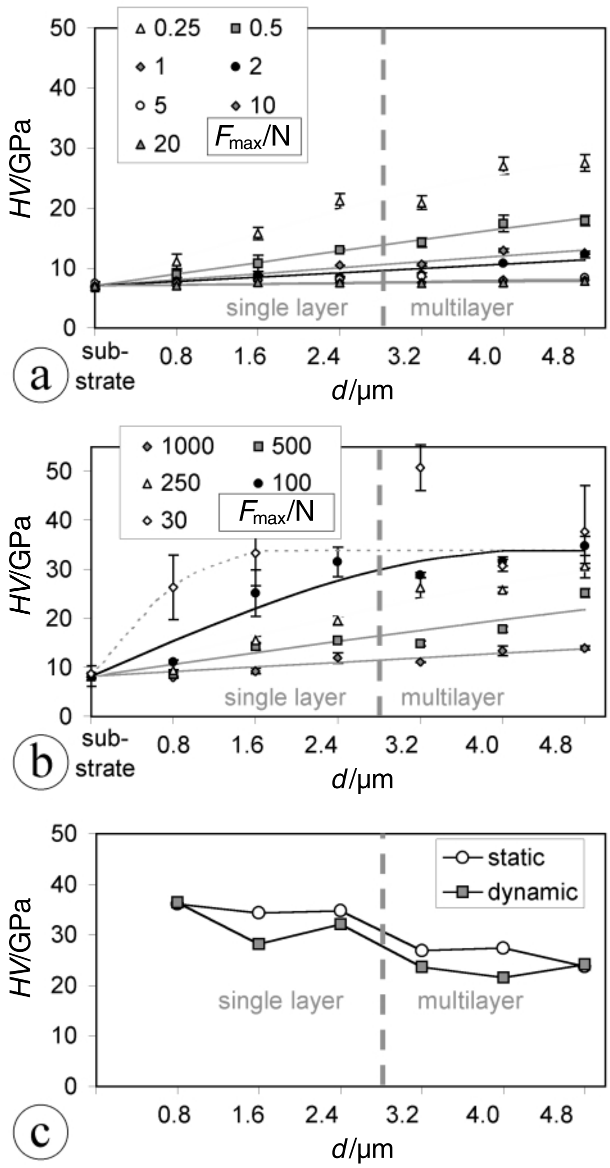

Figure 4: Microhardness, obtained by applying different loads: a) static measurement, b) dynamic measurement, c) corrected microhardness for both modes

Slika 4: Mikrotrdota, izmerjena pri različnih obtežitvah: a) statična meritev, b) dinamična meritev, c) korigirana mikrotrdota za oba načina decreasing the load. There are three ways to analyse the microhardness of the coating-substrate system: load-variation method (applying different loads), constant-load method (probing differently thick coatings with the same load) and cross-section method [8]. In this work, the load-variation and the constant-load methods were used.

In **Figure 4a** the microhardness of various coatings is plotted, obtained at different loads by static measurement. Applying the highest loads (5 – 20 N), the plastic deformation zone is so large that there is virtually no influence of the coating. In fact, the bulk microhardness of the substrate is obtained and coating hardness is constant for the thickness range investigated. Using lower loads, the microhardness increases due to higher percentage of the coating in the plastic deformation zone. Nevertheless, no plateau is observed, therefore even at the thickest coating there remains some substrate influence. Using lower loads, applying dynamic indentation, this plateau is reached (**Figure 4b**). The best fit at 100 mN load yields a value of 33.9 GPa. The measurements at the lowest load (30 mN) are at the edge of reproducibility so they cannot be reliably applied.

The substrate influence can be bypassed if a suitable model is applied. In our work, the model proposed by Jönsson and Hogmark [9] was used. In **Figure 4c** the corrected microhardness is presented for coatings deposited at various deposition times. Data from both measurement techniques were used, however, the results obtained by dynamic measurement tend to be about 10% smaller than the ones obtained statically. This difference is due to different loading characteristics although the maximum load is the same. Nevertheless, the dependence on the coating thickness is similar. The multilayer coatings show about 25 % lower corrected microhardness values than the single layer ones, but within each group no preferential difference can be found. The reason for the lower microhardness of the multilayer coatings can be attributed to the relatively soft chromium layer between the individual hard CrN layers.

4 CONCLUSIONS

A series of CrN/Cr coatings have been deposited by reactive sputtering with a thickness between 0.8 (2 layers) and 4.8 µm (6 layers). The emphasis was given on the analysis of mechanical properties and their dependence on the film thickness and the number of layers. We found the following:

1. The adhesion is equal for all coatings (total delamination critical load at 65 N), the only exception is the thinnest coating (0.8 µm) whose adhesion is much worse.
2. The internal stress of the coatings is around 3.0 GPa, while for the multilayer coatings it tends to decrease to 2.5 GPa.
3. The microhardness is strongly load-dependent as the substrate influence is also measured. Its influence vanishes if sufficiently small loads are applied (100 mN). Applying the model, proposed by Jönsson and Hogmark, to correct the substrate influence, the multilayer coatings appear to have 25 % lower microhardness than the single layer ones.

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