MICRO-SCALE ABRASIVE WEAR TESTING
OF Cr-Nx COATINGS

S. LAKEL [1], K. ALMI [1],Y. BERRICHE [2]

[1] Laboratory of Fundamentals Sciences - University of LAGHOUAT – BP 37G, Laghouat, Algeria
Tel: 062 92 25 54 – Fax: 062 92 25 54, e-mail: s.lakel@yahoo.fr
[2] Laboratory of advanced materials – University of ANNABA – BP 12 ANNABA

Abstract. Information of the wear resistance proprieties of coatings is urgently required by suppliers and users to provide confidence in the performance of engineering coatings. The balcrattring test has been used to investigated the abrasive wear resistance of a series of thin (3.5-5.8 µm) PVD coatings of CrNx ($0.18 \leq x \leq 1$) on identical a DIN 1.4301 steel substrates. Two different abrasives, SiC and diamond were used. The hardness of the films was found to be in the range of 8-40 GPa. The aim of this paper is to evaluate the abrasion resistance of hard coatings, and low abrasive wear resistance of these coatings are compared. The results obtained show an excellent correlation between coatings hardness and abrasive wear resistance. The results were compared to those of a chromium nitride reference coating. The results obtained show an excellent correlation between coating hardness and abrasion resistance.

Key words: abrasion wear resistance, ballcratering, CrNx, tribology.

1. INTRODUCTION

The introduction of PVD coatings industry is one of the main success stories in the industrial application of modern coating technology over the last 30 years. The first PVD coating material to have a commercial application on cutting tools was TiN in the early 1980s and since the 1990s most cutting tools are PVD coated particularly in applications where sharp edges are required, *e.g.* threading, grooving, end-milling, etc. and in cutting applications that have a high demand for a tough cutting edge, *e.g.* drilling [1]. In solid carbide cutting tools (end-mills and drills) PVD is the standard coating technology [2]. The TiAlN and TiCN PVD coatings is currently the most widely deposited PVD coatings for cutting tools but other coatings such as CrN and Ti-B-C offer better solutions in certain applications [1, 3, 4].

Numerous applications where surface engineering has been applied successfully like cutting or forming processes require enhanced wear resistance [5]. Out of the main wear mechanisms identified to determine lifetime of tools, a

considerable amount of abrasive wear takes place when particles, which are harder than the tool material, are involved. These particles can typically be carbides or oxides (e.g. when cutting steel) or highly strain-hardened fragments (e.g. wear debris generated during deep-drawing). Typically, abrasion resistance is evaluated using wear tests where the bulk mass loss or dimensional changes are monitored [6]. However, for the characterization of the abrasion resistance of surface engineered tool materials on a laboratory scale, a test method where very small samples can be used is highly attractive [7, 8]. The best known tribological application of a ballcratering method is a micro-scale abrasion test [8–9] and commercial tribometers based on this method are available (e.g. CSEM Calowear and Plint TE-66 micro-scale abrasion tester). Initially, the ball cratering method has been developed for measurements of coating thickness and in this capacity it is still used as a standard test in many industrial laboratories [8,10,12]. Subsequently its use has been extended to measurements of abrasive wear resistance of thin coatings or surface layers and the technique developed is known as a micro-scale abrasion test [8–9]. The name micro-abrasion originates from the very small wear depths and volumes generated in the wear sample by the rotating ball covered with fine abrasive particles. This allows for conducting wear measurements on surface films of several microns in thickness [8–11]. Micro-scale abrasion tests use abrasive slurries or pastes containing fine abrasive particles of micron, or even sub-micron, size [8, 10, 13]. This small size of abrasive grits is deliberately chosen to obtain good quality optical images of wear craters for accurate determination of a coating thickness or wear volume [13, 14]. Wear volume is calculated by measuring the diameter or depth of the wear crater and applying simple geometrical equations [8–10, 15]. For coated surfaces the wear coefficients of both the coating and substrate can be determined from a single test [8–10]. Micro-scale abrasion test has also been increasingly used for testing the abrasive wear resistance of bulk materials such as non-ferrous metals and their alloys [8, 16, 15–18], tool steels [8, 17, 19], plain carbon steel [10, 15].

2. EXPERIMENTAL DETAILS

2.1 COATING DEPOSITION

All coatings investigated have been deposited using an unbalanced magnetron sputtering plant. Cr-N coatings were grown by reactive sputtering in Ar/N_2 atmospheres using a Cr target. Different N/Cr atomic ratios as characterized by wavelength-dispersive electron probe microanalysis have been adjusted by the N_2 flow rate resulting in the phase compositions (characterized by X-ray diffraction) given in table 1. As substrates, ground and polished austenitic stainless steel (DIN 1.4301, hardness 200 HV) samples with dimensions Ø 25 - 5 mm and 20 mm- 20 mm-5 mm were used.

Coating	Chemical composition	Phase composition	Hardness (GPa)	Coating Thickness (μm)
Cr-N 1	Cr	Cr	9.2	5.6
Cr-N 2	$CrN_{0.19}$	$Cr(N)+Cr_2N$	29.2	5.1
Cr-N 3	$CrN_{0.48}$	Cr_2N	32.7	4.6
Cr-N 4	$CrN_{0.97}$	CrN	29.1	4.2
Cr-N 5	$CrN_{0.99}$	CrN	34.3	3.9
Cr-N 6	$CrN_{1.00}$	CrN	38.6	3.8

Coating hardness was determined from the load vs. displacement data measured using a computer controlled micro hardness tester (Fischerscope H100).

2.2 ABRASIVE WEAR CHARACTERISATIONS

The abrasion resistance was evaluated using a small-scale abrasive wear test (CSEM CaloWear) Fig.1. There, a steel sphere (DIN 1.2067, diameter, 25.4 mm) is rotating against the coated sample in the presence of an aqueous suspension of abrasive particles. In this work, aqueous suspensions of SiC (average grain size, 3.5 μm) and diamond (average grain size, 1 μm) have been used. The sphere was rotated against the sample with a velocity of 0.2 m/s.
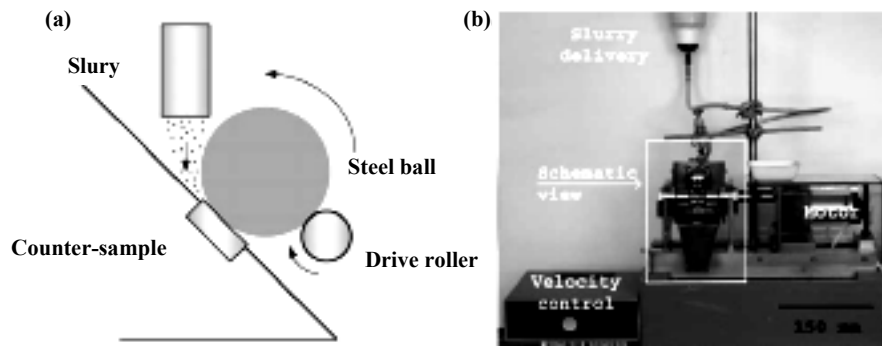

Fig. 1 – The principle of the CSM Calowear Tester: a) schematic diagram; b) experimental set-up.

Optical micrographs of typical wear craters in coated samples are shown in Fig. 2. The diameter of the resulting wear crater was determined as a function of

the sliding distance by means of a calibrated optical microscope, SEM and Talysurf profilometry to determine the dominating wear mechanisms and the extent of wear damage.The wear crater was measured at intervals corresponding to increments in the sliding distance of about 16m after removing the steel sphere. After each measurement, the sphere was relocated to the identical position and the typical total sliding distance was about 80m. The normal force L on the specimen is determined by the weight of the sphere and the lateral position of the drive sharft [8]. To optimize test conditions for this work, L was varied between 0.18 and 0.45 N, measured by a sensitive load cell.

As can be seen from the total sliding distance of 80m and the increments of 16m that five individual data points for all plots of crater volume versus sliding distance have been obtained. The deviation of these five values was typically below ± 8 %.
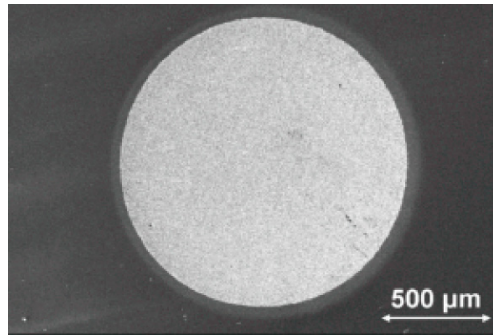

Fig. 2 – Optical micrographs of the wear crater in a $CrN_{1.0}$ coating (SiC slurry).

3. RESULTS AND DISCUSSION

The aim of a first test series was to optimize test conditions for the abrasive wear test. The abrasive wear rates of both coating and substrate material is derived from the increasing wear volume depending on sliding distance and normal force during the testing process using the equation described in [20]:

$$SL = \frac{V_c}{\kappa_c} + \frac{V_s}{\kappa_s} = \Omega_c V_c + \Omega_s V_s.$$
(1)

S is the sliding distance and L is the applied load. V_c and κ_c are the wear volume and load specific wear rate of the coating. V_s and κ_s are the corresponding parameters for the substrate. Ω_c and Ω_s are the wear resistance of coating and substrate (usually the wear resistance, which is the inverse of the wear rate). Knowing the substrate wear resistance and the coating thickness, the coating wear rate could easily be calculated from the measured wear volume.

Thus the abrasive wear rate of the coating and substrate are calculated by a modification of the equation (1):

$$\frac{SL}{d_a^4} = \left(\frac{\kappa_s - \kappa_c}{\kappa_s \kappa_c}\right) \cdot \left(\frac{\pi t}{4d_a^2} - \frac{\pi R t^2}{d_a^4}\right) + \left(\frac{1}{\kappa_s}\right) \cdot \left(\frac{\pi}{64R}\right). \tag{2}$$

There, S is the distance slid by the sphere, d_a is the external diameter of the wear crater, t is the coating thickness and R is the radius of the sphere. A plot of $\left(SF_N/d_a^4\right)$ against $\left(\left(\pi t/4d_a^2\right) - \left(\pi r t^2/d_a^4\right)\right)$ is thus linear with the intercept and gradient determined by the values of κ_s and κ_c (Fig. 3). κ_s can be determined directly from the intercept, and this value is then used together with the gradient to provide κ_c.
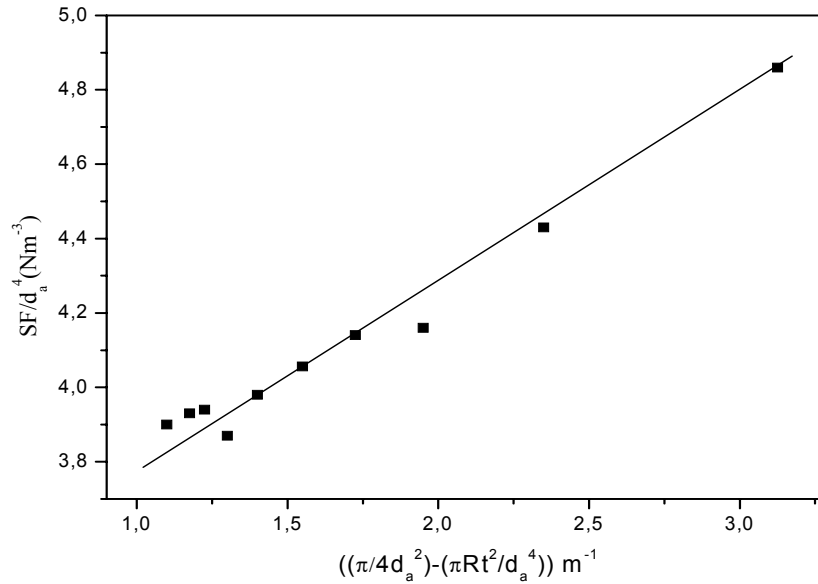

Fig. 3 – Plot of abrasion wear data obtained for a $CrN_{1.0}$ coating according to equation (2) using a SiC slurry.

Fig. 4 shows the abrasive wear rate of coating and substrate determined for a coating of chemical composition $CrN_{1.0}$ deposited onto an austenitic stainless steel substrate as a function of the normal force applied on the sample. The standard deviation of κ_c for the loads investigated is well below 4 %. However, to minimize the error in determining κ_s, the normal load for all further investigations was adjusted in the range between 0.3 and 0.4 N.
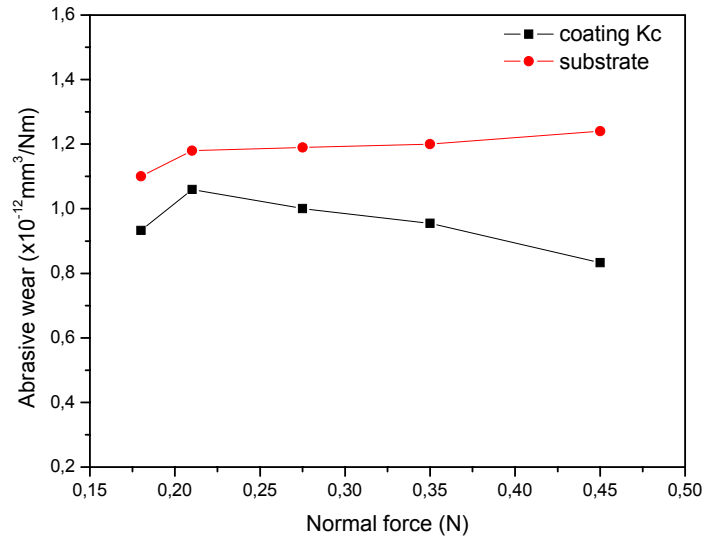
Fig. 4 – Dependence of the abrasive wear rate of coating and substrate on the normal force applied for a $CrN_{1.0}$ coating on austenitic stainless steel (SiC slurry).
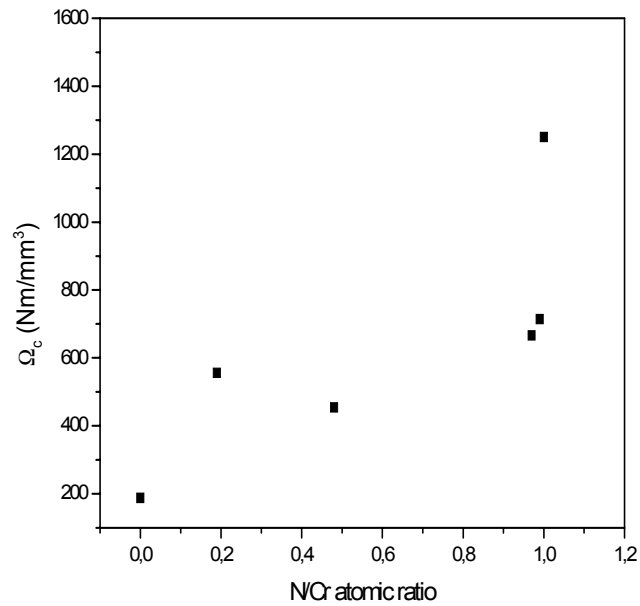

Fig. 5 – Dependence of the wear resistance of Cr-N coatings on their chemical and phase compositions (see Table 1, SiC slurry).

The abrasion coefficients of Cr-N coatings determined using SiC abrasive slurry and optimized test parameters is illustrated in Fig. 5 as a function of their chemical composition. There, the phase composition as characterized by X-ray diffraction. The abrasion resistance for the Cr coating is about 188.5 Nm/mm^3. For the dual-phase coating consisting of a Cr (N) solid solution and Cr_2N and for the Cr_2N coating, Ω_c increase to about 500 Nm/mm^3. Coating close to the composition of stoichiometric CrN yield an abrasion resistance of about 1 200 Nm/mm^3 which is in good agreement to literature values [8, 21].
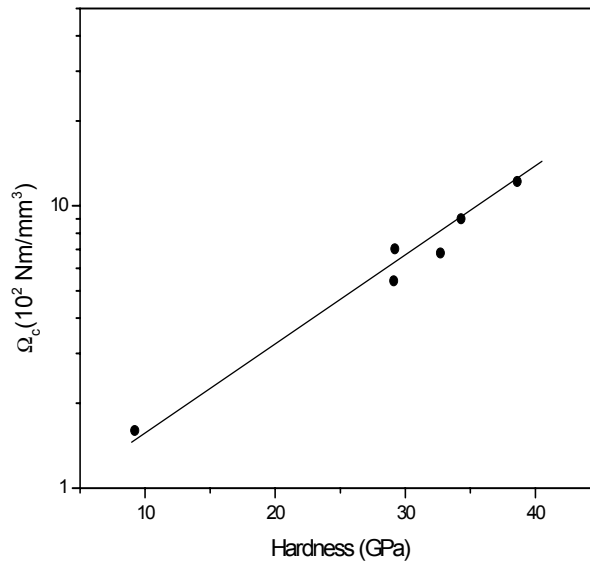

Fig. 6 – Abrasive wear resistance as a function of hardness of Cr-N coatings.

The abrasive wear behavior of the coatings is shown in Fig. 6, which plots the abrasive wear resistance of Cr-N coatings as a function of their hardness. There is evidently a clear correlation between hardness and abrasive wear, which agrees with the results found in literature [8, 21, 22].

The data point given in Fig.6 has been obtained for diamond slurry, where the standard deviation yields a value of about 4 %. Although the coating investigated show a wide range of hardness values, Fig. 6 shows an excellent correlation between the coating abrasive wear rate and the hardness.

CONCLUSIONS

Within this work, several sputtered coating from Cr through Cr_2N and dual-phase coatings consisting of the solid solution Cr (N) and Cr_2N to CrN single phase

coatings have been investigated with respect to their abrasion resistance and hardness. The results obtained show an excellent correlation between coating hardness and abrasion resistance. Moreover, the micro-abrasion testing employed offers a simple and inexpensive method for pre-selecting and ranking of coating materials for abrasive wear situations.

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