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**Abstract:** Engineered surfaces can provide superior resistance to abrasive and adhesive wear. There are many types of hard coatings readily available including Diamond-Like-Carbon (DLC) coatings, nitrides, carbides, oxides, etc., and due to the many processing alternatives including alloying element, thickness, deposition technique, etc. each of these types contains a virtually endless number of choices. Fundamental materials properties such as hardness and toughness are often not adequate to reflect how a particular coating will perform, and some of these properties, toughness for example, can be hard to measure and are usually not available. In contrast, bench-scale measurements of friction and wear can provide reliable and meaningful data. However, many of these techniques are expensive and time-consuming and determining the best coating to meet the requirements of a specific application amongst this endless variety of materials can be cost-prohibitive. In this case, ball-on-three-disk (BOTD) test methods are used to provide a rapid, cost-effective, and accurate measure of the wear and abrasion resistance of representative samples of many of the types of coatings being designed with modern techniques. This work provides a case study of steel coated with seven types of nitrides, novel Cr and Ni plated coatings, as well as baseline uncoated and manganese phosphate coated steel samples. The data illustrates the value of the BOTD test method as a bench-scale tribological test as well as significant insight to how subtle design features and changes in the testing conditions of coatings can lead to significant differences in performance.

## ***WEAR***

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# Evaluation of the Wear and Abrasion Resistance of Hard Coatings by BOTD Test Methods – A Case Study

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## Abstract

Engineered surfaces can provide superior resistance to abrasive and adhesive wear. There are many types of hard coatings readily available including Diamond-Like-Carbon (DLC) coatings, nitrides, carbides, oxides, etc., and due to the many processing alternatives including alloying element, thickness, deposition technique, etc. each of these types contains a virtually endless number of choices. Fundamental materials properties such as hardness and toughness are often not adequate to reflect how a particular coating will perform, and some of these properties, toughness for example, can be hard to measure and are usually not available. In contrast, bench-scale measurements of friction and wear can provide reliable and meaningful data. However, many of these techniques are expensive and time-consuming and determining the best coating to meet the requirements of a specific application amongst this endless variety of materials can be cost-prohibitive. In this case, ball-on-three-disk (BOTD) test methods are used to provide a rapid, cost-effective, and accurate measure of the wear and abrasion resistance of representative samples of many of the types of coatings being designed with modern techniques. This work provides a case study of steel coated with seven types of nitrides, novel Cr and Ni plated coatings, as well as baseline uncoated and manganese phosphate coated steel samples. The data illustrates the value of the BOTD test method as a bench-scale tribological test as well as significant insight to how subtle design features and changes in the testing conditions of coatings can lead to significant differences in performance.

## 1. Introduction

Coatings often provide a very cost-effective means to achieve properties that would be difficult or impossible to obtain with monolithic materials, but the utilization of coatings can be impeded by the lack of relevant performance data that would allow a designer to select a particular coating that would likely meet the requirements of a specific application. Generic materials property data such as hardness, service temperature, and coefficient of friction exist for many coatings, but experience has shown that the substrate and coating behave as a system and have to be evaluated together in addition to ensuring that the testing conditions are relevant to the application of interest. There is also a growing trend of available designer coatings deposited as micro- or nano-layered structures or micro- or nano-composites to improve selected properties. Although the advances in coatings have been significant, the vast number of different coatings and processing techniques for a given coating family (e.g. TiAlN or AlCrN), make it very difficult to characterize a particular family of coatings through examination of a single example. Additionally, there are often significant differences in what one would assume to be essentially the same coating between suppliers due to the use of proprietary or patented processing techniques. This lack of concrete materials property data may be one reason why highly engineered coatings such as the reactively sputtered nitride and diamond-like-carbon (DLC) coatings have achieved wide application in only relatively few applications such as cutting tools, and have achieved far less application in more diverse mechanical engineering applications [1]. The fact that many of the standardized tests have been developed with conditions geared toward cutting tools and other simple applications is another reason for their lack of widespread use.

An alternative to using physical properties of coatings for their selection is to use full scale testing to validate coating selection, but this approach has limitations such as it is often far too expensive and time consuming. One example where use of advanced coatings may provide significant improvement is in small arms components. However, the lack of credible and consistent data has limited the components to a select few long-standing approaches such as black oxide, manganese phosphate, and chromium electroplating for steel components, and anodizing for aluminum components. Small arms possess a multitude of actions requiring precise relative timing and containing parts with tight tolerances. Small arms are also exposed to erratic and diverse environments, severe thermal shock, and hot and corrosive gases. With this operating environment and stringent performance requirements, small arms require rigorous maintenance schedules in order to provide for the necessary high reliability and long life. Based on the complex environment and unique operating conditions, small arms provide a good example of one application where the use of higher performance engineered coatings has been impeded due to the lack of relevant and consistent data.

To demonstrate the impracticality of using full scale testing to validate a coating selection, consider selection of a hard coating to prevent abrasion and jamming of the weapon in the deserts of the mid-east. A firearm operates through coordination of various actions. One action of the weapon involves the firing pin striking the cartridge to fire the round that is held in the chamber by the bolt. In auto and semi-automatic weapons, as the gas expands, some of it is captured by a gas tube and directed to the back of the bolt where the increasing pressure drives another action that pulls the bolt backward during which a pin in a channel forces the bolt to rotate and unlock. As the bolt travel backward, the fired round is extracted from the chamber and moves backward until it reaches the ejection port where a spring ejects the round. During the backward motion of the bolt, an action spring is compressed to store some of the energy of the expanding gas. As the gas expansion dies out, the action spring drives the bolt carrier forward. A spring in the magazine is pushing the next round up toward the chamber and when the bolt rides over this area it extracts the round and pushes it forward into the chamber. The pin

and channel cause the bolt to rotate and lock at the front of its movement. All of these motions involve sliding and experience different loads, temperatures, sliding velocities, and differing exposure to sand/dust and abrasive particles from propellant by-products. With these varied tribological conditions, the various parts exhibit different extents of adhesive and abrasive wear. This makes it very difficult to determine the appropriate properties required to improve the overall performance of small arms systems.

The issues for using coatings in small arms are typical of many complex mechanical applications and raises important engineering questions relative to using a coating to improve immunity to abrasion and jamming. One important consideration is in determining if all sliding surfaces need to be coated. If this is the case, the cost of weapon system may increase prohibitively. Also, with the very diverse operating conditions of all the parts, one must determine if the same coatings be optimal for all of the surfaces. If this is not the case, the complexity and cost of the development effort would dramatically increase and validation of the performance of coating a particular part would become far more difficult. Determining this ahead of time would prove very difficult. An additional consideration is in full scale testing of the coated weapon, one must determine how the jamming of the weapon would be diagnosed relative to the performance of a specific action and how the coating that affects that action. Addressing this challenge is essential to validating the performance increase and justifying the cost of using a coating. Not only is there a difficulty in relating performance of the coating to the propensity for weapon jamming but another issue is the difficulty in replicating the blowing sand and environmental conditions of the field in a firing range. This issue is very complex because it not only involves the weapon-environment interaction, which is hard to duplicate, but also the man-machine interaction, which is very hard to quantify.

These issues are complex and even if answers can be found, it is obvious that full scale evaluation of coatings would be prohibitively expensive and will not answer what is the fundamental design question, which is - what is the minimal use of coatings that solves the problem with the minimal cost impact. Good ways to answer these types of questions relative to the use of coatings in many complex applications do not exist, and therefore impede the practical selection and use of coatings that have the potential to dramatically improve performance and solve critically pressing problems. Building a testing apparatus that simulates a particular action of the weapon is one way to improve this situation and has been used, but this approach is itself expensive since the development of the test rig is a task by itself, and this approach does not allow others to easily replicate the testing without also building a similar specialized testing apparatus [2]. Modification of industry standard bench scale testing appears to offer a practical and effective approach since the test equipment is designed once and offered for sale to the entire community of researchers, and the test methods are developed by consensus, are widely known, and are easily practiced [3]. Nonetheless, a wide variety of bench scale testers have emerged over the years, which has somewhat diluted the simplicity and value of this approach.

The performance of coatings can be captured generically by three factors:

- a) Abrasive wear resistance
- b) Adhesive wear resistance, and
- c) The coefficient of friction.

Although a particular type of wear is the primary concern in many applications, it is important to consider both types of wear when selecting materials and coatings because abrasive wear, when it is present, will usually dominate the overall wear, but adhesive wear dominates in the absence of

abrasion. The coefficient of friction is not only important because it indicates the ease of sliding, the energy that will be lost and not available for useful work, along with the temperature rise that may occur, but also because both adhesive and abrasive wear can be increased by higher coefficients of friction. Another primary consideration is to keep the test as simple, cost-effective, and reproducible as possible while still providing relevant contact geometry and tribological testing conditions to the application of interest.

We have recently adapted a ball on three disk bench scale test configuration for testing coated systems, and have shown this to be a superior and holistic way to evaluate coatings for particular applications that involve sliding [4]. The self-centering ball-on-three-disk configuration provides a very stable and reproducible contact geometry, and the inclined BOTD geometry allows the specimens to be totally immersed in dry abrasive, lubricants, and other fluids giving great versatility. Use of a rubber ball gives effective three body abrasion and provides cutting action that is closer to actual field conditions and results that are highly correlated with the commonly used ASTM G65 method. Use of a metal or ceramic ball allows adhesive wear and galling to be studied and the coefficient of friction to be measured. Use of a ceramic ball also allows the thickness of the coating to be determined. The BOTD has the additional advantage of providing three replicate measurements from a single trial. The work reported herein provides a case study of the use of the BOTD test methodology to comprehensively evaluate a series of coatings with tribological testing conditions relevant to low normal load sliding contacts such as those that are present in small arms.

## 2. Experimental

BOTD abrasion tests were conducted with a 0.5-inch diameter neoprene ball (70 Shore A) and AFS50/70 sand. Tests were conducted on fresh samples at ambient temperature for each of 60 RPM and 120 RPM for 180 minutes. The track of the ball in contact with the three pads has a diameter of 0.29 inches, so the sliding speeds were 0.023 m/sec and 0.046 m/sec, respectively. The sliding distance was 8337 meters for the 60 RPM tests and 16674 meters for the 120 RPM tests. BOTD measurement of the coefficients of friction and adhesive wear tests were conducted with a 0.5 inch diameter 52100 steel ball, which is a commonly used ball bearing steel with a hardness of 62 to 64  $R_c$ . Tests were conducted on fresh samples at each of 35°C and 220°C. The rotational speed was 60 RPM (0.023 m/sec) and the test duration was 30 minutes. The sliding distance was 1389.5 meters. The coefficient of friction is obtained from the first several minutes of the test after the coefficient of friction has stabilized, but before there is fluctuation in coefficient of friction attributable to the onset of significant adhesive wear.

The disks were 4140 steel (34  $R_c$ , 335 HV) in all cases and this served as the substrate for the coatings. The normal load of the ball on each of the three specimen disks was 340.2 grams (3.34 N). Three disks of the same specimen composition are used in each BOTD trial. Each BOTD trial results in a wear scar on each of the three disks, and the profiles of these scars were measured by a Zeiss Model 1400A profilometer. The scars appear to be spherical, so we obtained a single profile trace through the apex of each of the scars.

The coatings that were examined, as well as some of their published/measured properties, are shown in Table 1.

**Table 1: Specimens Examined by the BOTD Abrasion Test**

Specimen	Application Method	Process Temperature, C	Service Temperature, C	Hardness, HV	Coefficient of Friction	Thickness, um	Density, gms/cc
4140				335			7.8
AlCr Based	PVD	250	1100	3000	0.25	2.54	4
AlCrN	PVD	250	1100	3200	0.35	2.49	4
AlTiN	PVD	250	900	3300	0.4	2.6	4
Cr + Diamond	Electroplate	54	870	1960	0.15	1	
Cr + Diamond	Electroplate	54	870	1960	0.15	10	
Mn3(PO4)2						8.89	1.8
NiB	AutoCatalytic	175	1000	1400	0.44	5.5	7.8
TiAlN	PVD	250	900	3300	0.35	2.49	4
TiAlN	PVD	250	900	3400	0.35	5.07	4
TiAlN + a-C:H:W	PVD	250	800	3000	0.2	4.15	4
TiAlN + gas N2	PVD	480	700	3300		2.5	4

The coating test matrix is comprised of a number of electroplated, autocatalytic, and reactively sputtered coatings that provide a representative sample set, illustrating the modern trends in coating design that will be described below. Bare 4140 steel and manganese phosphate coated 4140 are one of the traditionally used materials for many small arms components and therefore constitute the baseline materials. Diamond Chrome is an innovative chrome coating provided by Superior Technology that contains a high loading of diamond particles to improve abrasion resistance. NiBoride is a particular version of nickel boron. The reactively sputtered coatings that were examined were obtained from Oerliken Balzers and represent some of the state of the art nitride and multilayer coatings currently available for tooling and other industries. TiAlN and AlCrN based coatings have been shown to provide superior performance to the earlier developed and more commonly used TiN coatings. These nitride based coatings provide for some of the hardest surfaces currently available.

### 3. Theory

Indices of the performance of a coating can be developed by modeling the coating using fundamental properties such as hardness, fracture toughness, and wear resistance. One of the many advantages of using models to predict the performance of coatings are the insights that such models provide for developing improved coatings. Measurement of material properties does not provide the same level of insight as a mechanistic model, but it can be much quicker and more practical. As such, direct measurement of properties is the most common method employed to compare performance differences between materials. Direct testing of friction and wear resistance with bench-scale testing is an example of a technique, which provides unambiguous data and direct insight into the relative performance of coatings and supplementing these bench-scale tests with surface analysis such as microscopic examination of the wear scars can provide additional insight into the mechanisms of wear and the factors controlling the performance of the coating.

As an example of a simple predictive model, Archard's equation is widely cited for prediction of both adhesive wear and abrasive wear:

$$V = \frac{C}{H} * N * S \quad 1$$

where  $V$  is the volume of material lost due to wear,  $N$  is the load,  $S$  is the sliding distance,  $H$  is the penetration hardness of the material being tested, and  $C$  is the probability of wear occurring during adhesive junction rupture, or in the case of abrasive wear  $C$  accounts for the fact that only a fraction of

the contacts of asperities during sliding cause plastic deformation, that the sharpness of the abrasive particle influences wear, and that the wear scar can be larger than the size of the conical tip of the abrasive particle [5].

A central feature of this model is that increasing the hardness of a material will reduce its propensity for both adhesive wear and abrasive wear, but this is too simplistic by itself. Crack propagation, for example, can be inhibited by some coating structures, but this is likely to have more of an impact on abrasive wear where the hard particle introduces a crack that then propagates than for adhesive wear where micro-welding between the coating and counter-surface constantly pulls out material. Another example is that in abrasive wear, the constant  $C$  itself is also a function of the hardness of the material in that hard materials are often more brittle and brittle materials exhibit larger wear scars than the size of the indenter, which is reflected in a larger value for  $C$ , whereas this is not the case for adhesive wear. Another limitation to the use of a simple equation such as equation 1 is that the elasticity of a material is also a factor in controlling wear, and this has led to the proposal that wear rates can be better predicted by the ratio of hardness,  $H$  to elastic modulus,  $E$ , as given in equation 2:

$$V = f\left(\frac{H}{E}\right) * N * S \quad 2$$

Hardness is a crude measure of strength, whereas the elastic modulus gives a measure of the material's tendency to elastically deform. The  $H/E$  ratio expresses a measure of the elastic limit of strain, which is an indicator of the amount of strain a coating can experience without permanent deformation occurring. The importance of this ratio has been long recognized in gear design where the ratio is used as part of what is called the plasticity index. The ratio of  $H^3/E^2$ , called the plastic resistance parameter, has also been used as an index of the resistance to plastic flow. Therefore, coatings with both higher  $H/E$  and  $H^3/E^2$  ratios should provide the best wear resistance.

Although adding consideration of elasticity to hardness more realistically models the performance of a coating, these two considerations are still far from a complete indicator of the actual performance. For example, it is well known that high friction can increase wear by both increasing tensile stresses that propagate cracks and inducing thermal stress. This is reflected by the fact that the wear rate of various steels is a linear function of the friction coefficient over a large range (0.45 to 0.85) of friction coefficients. Many hard coatings tend to have high friction. Friction results from mechanical interlocking of asperities, Van der Waals forces, and in some cases stronger chemical bonding. Breaking of these bonds requires deformation, but the shear strength is higher for harder materials and they are thus harder to deform, which results in higher friction. The relationship between higher shear strength and higher friction can also be inferred from the fact that hydrodynamic lubrication provides greatly reduced friction by providing a fluid film with very low shear strength.

A successful wear resistant coating must support high loads, provide low friction, and must not exhibit cohesive fracture or loss of adhesion to the substrate. One approach to achieve these qualities that has resulted from the perspectives above is to limit both dislocation motion and crack propagation because the motion of dislocations is central to the plastic deformation of materials and crack propagation is central to coating fracture. This approach has been implemented through development of multilayer and nanocomposite coatings. Both dislocation motion and crack propagation are significantly impeded at sharp interfaces between layers of materials and coatings formed from layers of materials with thicknesses in the 5  $\mu\text{m}$  to 10  $\mu\text{m}$  range and with sharp interfaces exhibit hardness that is substantially greater than the hardness of the individual layers. These types of coating structures are called superlattices. Limitation of dislocation motion and increased hardness are also achieved with micro-



composite coatings, in which 3 μm to 10 μm crystalline grains are embedded in an amorphous matrix with the grains separated by 1 μm to 3 μm.

The problem with an approach based on limiting dislocation motion is that dislocation motion contributes to stress relief and toughness, and limiting dislocation motion to increase hardness also reduces toughness and reduces the H/E ratio rather than maximizing it as is desirable. One way that has been used to achieve dislocation motion and increase toughness is to include ductile layers in a multilayer coating structure. The ductile layers provide a region for dislocation motion and stress relief and if integrated optimally can provide for both increases in hardness and toughness.

Modeling of the performance of coatings should also reflect that the coating is part of a system in which the substrate and the counter-surface will also make appreciable contributions. The simplest way that this, by using a reduced modulus of the contact materials in computing the H/E ratio, still does not address what can be appreciable contributions from delamination of the coating under the sliding load and roughness of the surfaces. These types of limitations with fundamental models and use of fundamental material properties to predict performance, place great value in using controlled and reproducible ways to directly measure the wear resistance of coatings under conditions that provide insight to their performance in representative testing conditions.

#### 4. Results

The wear scar dimensions and volumes are shown in Table 2. A positive value for the depth indicates that a scar formed in the specimen disk, whereas a negative value for the depth indicates a build-up of material due to transfer from the ball counterface onto the specimen disk. Each trial of the BOTD test is run for a fixed sliding distance, which causes breakthrough of some coatings, but not of others. When breakthrough occurs, the calculated wear rate is actually a composite of the coating wear rate and the substrate wear rate. This phenomenon makes the total scar volume an inaccurate representation of the coating performance. BOTD wear scars are spherical, but for abrasive wear testing with a neoprene ball, the radii of the scars are substantially larger than the radius of the ball and the radii increase in inverse proportion to the wear rate. The fixed duration of the BOTD test also makes the depth of the scars for each type of test (i.e. abrasive wear or adhesive wear) cover such a wide range that there is not a direct relationship between the volume of the scar and the scar depth.

Table 2 - Wear Scar Dimensions

Specimen	Abrasive Wear								Adhesive Wear							
	60 RPM				120 RPM				35 oC				220 oC			
	Width, mm	Depth, mm	Radius, mm	Volume, cubic mm	Width, mm	Depth, mm	Radius, mm	Volume, cubic mm	Width, mm	Depth, mm	Radius, mm	Volume, cubic mm	Width, mm	Depth, mm	Radius, mm	Volume, cubic mm
4140	4.087	0.101	20.689	0.666	4.240	0.151	14.965	1.074	0.95652	0.01751	6.54381	0.00631	0.40458	-0.00682	-3.73013	-0.00043
AlCr	2.885	0.001	1186.1	0.003	1.951	0.062	7.7	0.107	0.58945	0.00580	7.52677	0.00081	0.57873	-0.00439	-12.83599	-0.00053
AlCrN	0.630	0.032	1.6	0.006	2.526	0.085	9.4	0.215	0.52515	0.00737	4.85793	0.00088	0.51443	-0.00361	-9.47745	-0.00038
AlTiN	2.560	0.034	25.1	0.087	3.075	0.088	13.5	0.328	1.09316	0.02185	6.90171	0.01026	0.54658	-0.00753	-4.97127	-0.00089
DiaCr-100	3.373	0.003	492.2	0.013	3.901	0.007	283.2	0.041	0.27865	0.00094	11.17224	0.00003	0.41262	-0.00669	-3.22534	-0.00051
DiaCr-10	3.996	0.095	21.0	0.598	4.179	0.129	17.0	0.888	0.95920	0.01808	6.37354	0.00660	0.46620	-0.00653	-4.48718	-0.00056
MagPhos	4.078	0.097	21.6	0.633					1.01814	0.01881	6.89814	0.00767	0.60553	0.00716	6.40542	0.00104
NiB	3.712	0.088	19.7	0.478	3.746	0.128	13.8	0.704	0.60553	0.00564	8.25292	0.00088	0.88418	0.01427	7.56356	0.00515
TiAlN-51	2.635	0.003	345.8	0.007	3.116	0.007	180.3	0.026	0.43727	0.00544	4.43649	0.00042	0.63768	0.00679	7.73921	0.00110
TiAlN-LF	3.007	0.003	516.3	0.011	2.296	0.062	10.7	0.131	0.80380	0.01233	6.55422	0.00326	0.66983	0.00533	10.58247	0.00096
TiAlN-25	2.289	0.010	243.5	0.012	2.411	0.081	9.0	0.188	0.77165	0.01014	7.46117	0.00238	0.49300	-0.00460	6.91314	-0.00022
Duplex TiAlN	1.571	0.032	10.1	0.033	2.587	0.062	13.7	0.162	0.88954	0.01599	6.22080	0.00507	0.38582	-0.01035	-1.96767	-0.00059

The conventional way to handle breakthrough of some coatings but not of others in a wear test is to use a volumetric wear index  $k$  ( $\text{mm}^3/\text{N}\cdot\text{m}$ ) that is often reported in the literature, but the features of the BOTD scars described above violate the assumption that the volume of wear is linearly proportional to the sliding distance. We have shown that these considerations suggest that the wear rate index for the

BOTD tests reported herein should be based on scar depth rather than on scar volume using the following approach. The wear rate of a monolithic material, be it the uncoated substrate or a coating for which breakthrough does not occur during the test, is given by:

$$k_s = \frac{d_{obs}}{S * N} \quad 4$$

where  $d_{obs}$  is the scar depth, S is the sliding distance, and N is the load. This equation is also used when a build-up occurs on the specimen, but the sign of the wear rate is negative to signify a build-up, whereas the sign of the wear rate is positive when a scar forms. For a coated substrate where breakthrough occurs in the test, the wear rate of the coating,  $k_c$  is computed from the scar depth, the thickness of the coating ( $t$ ), and the substrate wear rate ( $k_s$ ) using equation 4 to give:

$$k_c = \frac{t}{S * N - \frac{d_{obs} - t}{k_s}} \quad 5$$

These computations for the BOTD results of the various coatings are shown in Table 3 for both abrasive wear and adhesive wear. Since the load was constant for all tests, the values of k could be multiplied by the load (3.34 N) to provide a prediction of the millimeters of wear per meter of sliding distance. The coefficient of friction for each coating is was also measured at the beginning of the BOTD adhesive wear test and the values are given in Table 3.

**Table 3: Results of the BOTD Abrasion and Adhesive Wear Test**

Family	Abrasive Wear		Adhesive Wear		Coef. Of Friction	
	60 RPM	120 RPM	35 oC	220 oC	35 oC	220 oC
	K, mm/(N m)	K, mm/(N m)	K, mm/(N m)	K, mm/(N m)		
4140	1.216E-04	9.077E-05	1.260E-04	-4.908E-08	0.22	0.21
AlCrN	4.221E-06	3.298E-06	2.484E-05	-2.595E-08	0.18	0.01
AlCr	1.190E-06	2.506E-06	2.246E-05	-3.159E-08	0.19	0.01
DiaCr-10	1.696E-05	3.938E-06		-4.701E-08	0.09	0.14
DiaCr-100	3.491E-06	4.046E-06	6.770E-06	-4.814E-08	0.17	0.02
MagPhos	7.870E-05			5.153E-08	0.07	0.03
NiB	3.531E-05	1.710E-05	2.193E-05	1.027E-07	0.29	0.05
TiAlN-25	3.240E-06	3.118E-06	3.182E-05	-3.310E-08	0.19	0.14
TiAlN-LF	3.213E-06	4.025E-06	5.608E-05	3.836E-08	0.21	0.18
TiAlN-51	3.015E-06	3.075E-06	3.726E-05	4.889E-08	0.35	0.14
TiAlN-Dup	4.214E-06	2.461E-06	7.844E-05	-7.447E-08	0.24	0.32
AlTiN	4.491E-06	3.576E-06		-5.416E-08	0.25	0.29

## 5. Discussion

The study reported herein was prompted by the desire to improve the immunity of small arms to abrasive wear that causes jams. A secondary objective was to understand what coatings could form the basis for operating small arms without liquid lubrication. This study therefore is typical of many applications wherein the tribological performance of a system needs to be improved in specific ways. As such, this work provides a good case study of the use of bench-scale testing for effective materials screening and qualification to solve specific problems.

Components in small arms are generally lightly loaded, and critical components in the bolt carrier assembly are often loaded only by their weight spread over a contact with substantial area, which produces contact stresses below 100 psi. Many of the components are fabricated from high strength low alloy steels and are coated with manganese phosphate, which is rapidly worn away under abrasive conditions. “CLP,” which stands for “clean, lubricate, and protect,” is used as a lubricant, and there is a wide ranging debate as to whether or not it increases abrasive wear, where an increase in abrasive would be attributed to a liquid lubricant trapping abrasive particles to form a slurry which leads to increased wear rates.

The coatings that were studied were selected to screen some of the state-of-the-art commercially available coatings that are being employed today to improve abrasion resistant coatings. Thus, the coatings that were examined contain examples of micro-composite coatings and nano-layered coatings (also called superlattice coatings). There are also examples of advances in conventional coatings, such as co-deposition of electroplated Cr with diamond particles, that achieve greater hardness than is typically found or monolithic Cr.

Although fundamental factors such as hardness and coefficient of friction contribute to a coating’s performance, the actual performance of coatings is indicated by the combination of the abrasive wear rate, the adhesive wear rate, and the coefficient of friction within the specific operating parameters of interest. To begin the comparison of the data in Table 3, the coatings can be grouped into a few families for which the measured properties of their members can be averaged. Although this does obscure some meaningful differences between members of a given family, which will be covered below, it does allow for a meaningful and easier comparison of the attributes of modern approaches to improving the performance of coatings. This comparison is shown Figures 1 by using the ratio of each property’s value for each coating relative to uncoated 4140 steel, which is considered as the baseline material for this study. The reference values for 4140 are also provided.

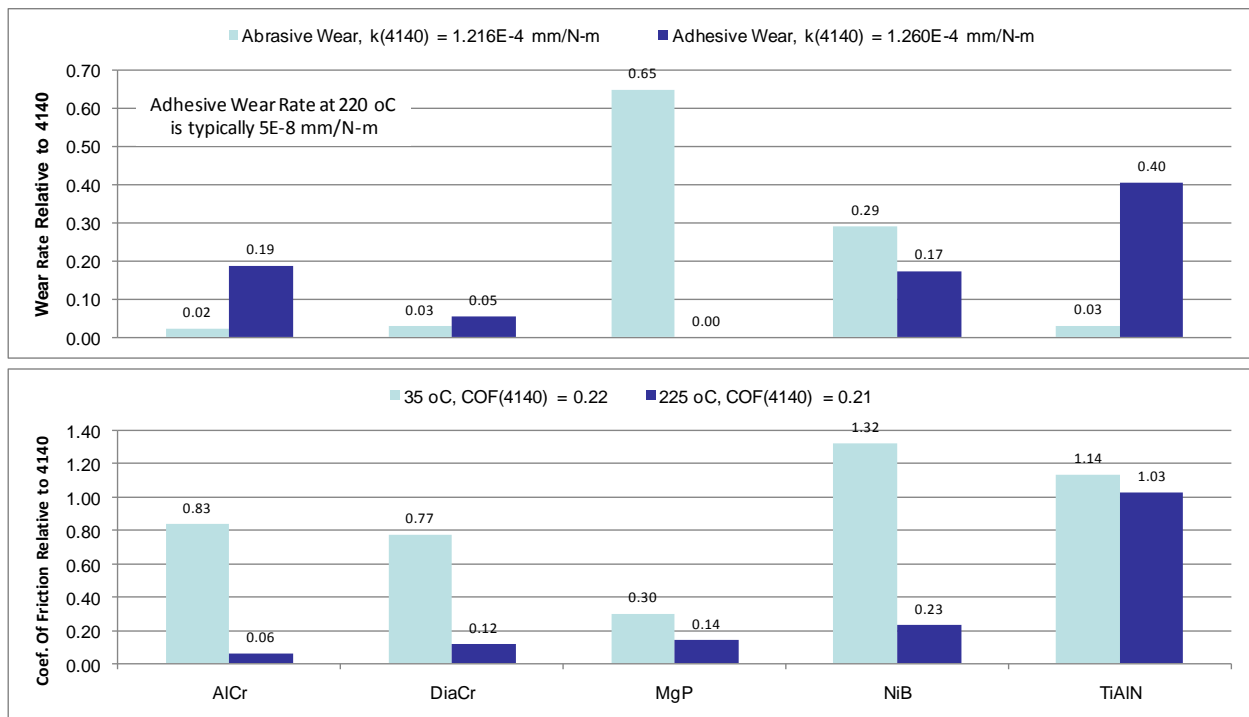


Figure 1 - Performance Comparison of Coatings for Use in Small Arms

Depending on what component of the small arm is coated and how the weapon is operated, the coatings could be exposed to ambient temperature, or to temperatures as high as about 220° C. This temperature range does not have meaningful impact on the relative abrasive wear rates between these types of materials and testing at the top of this temperature range would prohibit the use of a rubber surface, which is highly desirable in the abrasive wear test [6]. Therefore, abrasive wear was only measured at ambient temperature, and this is also expected to provide an accurate indication of the abrasive wear rate at 220° C. In contrast, the adhesive wear rates and the coefficients of friction change appreciably over the temperature range that was examined. The results in Table 1 show that the adhesive wear for all specimens at 35° C was a transfer of material from the test specimen to the 52100 ball (i.e. a scar formed in the test specimen), but at 220° C, some specimens showed transfer of material from the 52100 ball to the test specimen (i.e. a build-up) and some specimens showed a mix of transfer to and from the test specimen. Despite this complex behavior, the fact is that the adhesive wear rate at 220° C is very low in all cases, almost four orders of magnitude lower than at 35° C. The average adhesive wear rate at 220° C is about  $5 \cdot 10^{-8}$  mm/N-m regardless of whether material is transferred from the coated specimen to the ball or from the ball to the coated specimen. This effect is expected to be due mainly to the preferential softening of the 52100 ball at high temperature with relatively little change in mechanical properties of the thermally stable nitride and plated coatings.

Manganese phosphate is one of the predominant coatings for steels used in small arms. In the as-deposited state and when combined with a liquid lubricant, it provides for low friction operation, improved wear resistance, and excellent corrosion resistance. The BOTD tests reported herein show that the coefficient of friction of manganese phosphate is about 30% of that of 4140 steel at room temperature and about 15% of steel's coefficient of friction at 220° C even in the absence of lubrication. This decrease in friction is related to the properties of manganese phosphate since the observed coefficient of friction of 4140 steel was about the same at the two temperatures. For these operating conditions the manganese phosphate reduces the abrasive wear by about one-third and virtually eliminates adhesive wear at elevated temperature. The greater impact on adhesive wear is not a surprising result given the modest hardness of manganese phosphate. Overall, manganese phosphate is a good example of the improvement that can be obtained relative to equation 1 by modestly increasing the hardness and lowering the coefficient of friction of a coating.

The nickel boron coating examined in this study is an example of a coating that uses higher hardness relative to equation 1 to achieve greater wear resistance. Electroless nickel is applied by an autocatalytic process and is a historically used wear and corrosion resistant coating, and work in the early 1970's showed that the coating's hardness could be substantially improved by incorporating 5% to 6% by weight of boron. Nickel boron is an alternative to hard chrome, and it has a higher hardness than hard chrome. ASTM G65 testing indicated that abrasive wear was reduced by use of nickel boron by over 60% relative to hard chrome [7]. The BOTD tests reported herein show that the abrasive and adhesive wear rates are reduced by over 70% and 80% respectively, which shows that the higher hardness of nickel boron is an effective strategy to reducing both adhesive and abrasive wear as predicted by equation 1.

Diamond chrome is an example of a composite coating in that micro-size hard diamond-like particles are held in a more ductile matrix, which is chrome in this case. Due to the presence of the hard diamond particles in the composite, diamond chrome should provide some of the higher hardness and increased toughness discussed for equation 2, and the published data indicate the hardness is significantly greater than for hard chrome, 1960 HV for diamond chrome as compared to ~600-1000 HV for hard chrome. The data in Figure 1 clearly show the higher hardness and toughness gained by incorporating the nano-particles to be beneficial. Diamond chrome reduces the abrasive wear rate of 4140 by 97% and the

adhesive wear rate is reduced by 95%. The coefficient of friction is also reduced by about 25% at room temperature and by almost 90% at 220°C.

One other aspect worth noting about the diamond chrome results is the difference in the abrasive wear rates in Table 1 for the 1  $\mu\text{m}$  coating and the 10  $\mu\text{m}$  coating. The order of magnitude higher wear rate for the 1  $\mu\text{m}$  coatings likely reflects what is widely reported in the literature about the occurrence of higher wear rates when the hardness of the coating is substantially less than that of the abrasive particles and when the thickness of the coating is less than the size of the abrasive particles, which is the case for the 1  $\mu\text{m}$  diamond chrome coatings. It has also been shown that the surface hardness of softer coatings increases as the thickness increases, which is why the abrasive wear rate for the 10  $\mu\text{m}$  diamond chrome coating is substantially less than for the 1  $\mu\text{m}$  thick coatings [8].

Nickel boron and diamond chrome both exhibit high hardness and composite structures. Cross-sectional micrographs of the as-deposited coatings are given in Figure 2.

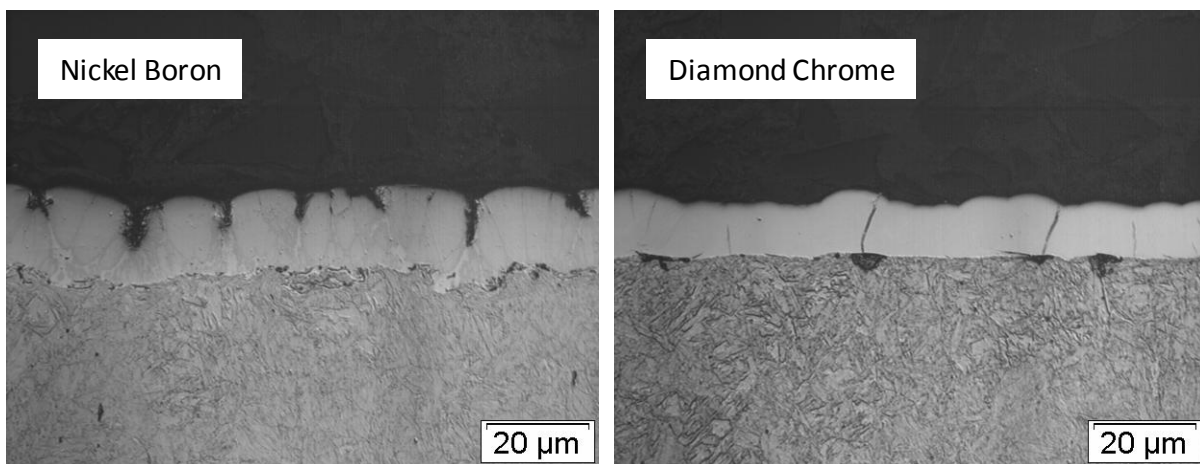


Figure 2 – Cross-sectional micrographs of as-deposited nickel boron and diamond chrome layers (1000X)

The AlCr family and the TiAlN family are comprised of materials examples of nano-layered coatings in which nano-thickness layers increase the hardness, stop crack propagation, and relax stresses. The TiAlN coatings are an older design, which uses the multilayer structure to maximize the hardness of the coating. The AlCr coatings are a newer design that optimizes the multilayer structure to improve toughness as well as hardness. Unlike nano-composite coatings such as diamond chrome where both abrasive and adhesive wear were reduced about equally, the nano-layered coatings reduce the abrasive wear rate by over 97%, but the adhesive wear rate is reduced far less, only 60% to 80%. As discussed in the “Theory” section, this demonstrates that the crack propagation inhibition of multilayer coatings is most valuable for preventing abrasive wear where the hard particles introduce cracks that then propagate and less valuable for adhesive wear where micro-welding between the coating and counter-surface constantly pulls out material. The coefficients of friction are relatively high for the AlCr and TiAlN families, which, as discussed in the “Theory” section, reflects that friction is highly influenced by the deformation of asperities and the high shear strength of hard coatings inhibits plastic deformation of the contacts and leads to increased friction. The AlCr family has a lower coefficient of friction than the TiAlN family, and this is especially true at the elevated temperature.

The images of the as-deposited coatings in cross-section for AlCrN and TiAlN are shown in Figure 3. Unlike the nickel boron and diamond chrome coatings, AlCrN and TiAlN do not show any extent of

cracking or porosity in the as-deposited condition, which indicates the potential for improved toughness.

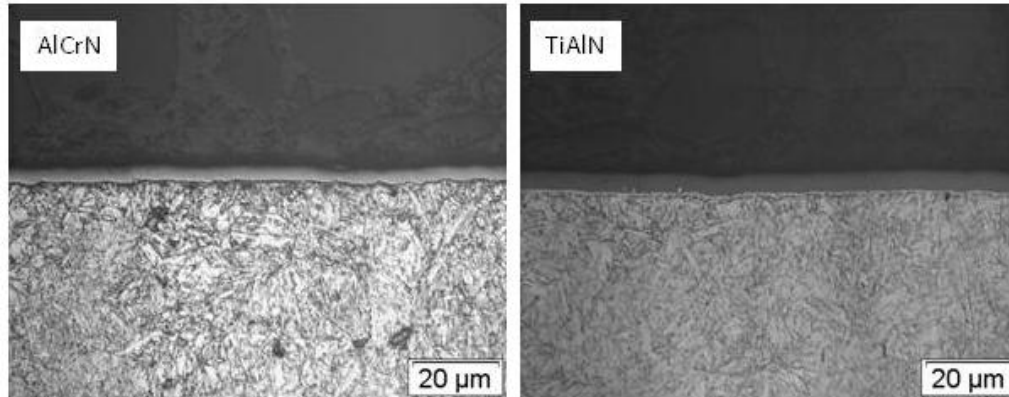


Figure 3 - Cross-sectional micrographs of as-deposited AlCrN and TiAlN (1000x)

The AlCr family and the TiAlN family merit further dissection because of the composition and structural differences that exist in the members of these families. The results for the individual members of the AlCr family and the TiAlN family are shown in Figure 4.

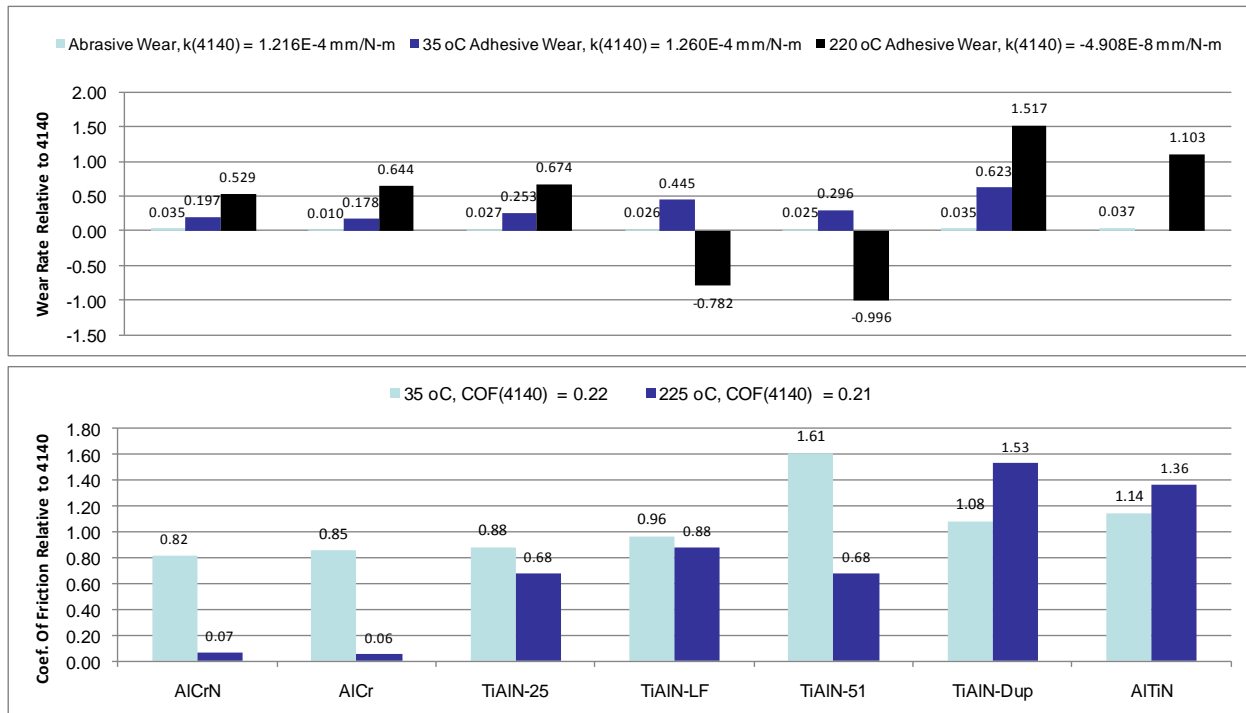


Figure 4 - Comparison of Sputtered Nitride Nano-Layer Coatings

There are four TiAlN coatings and one AlTiN coating, and these will be considered first. The TiAlN coatings contain alternating layers of TiN and TiAlN, with each layer being about 120nm thick. This provides abrupt interfaces that increase hardness and TiN is relatively soft (2300 HV) by sputtered nitride standards, so the TiN layers provide some stress relief. The TiAlN-25 and TiAlN-51 are exactly the same coating in two different thicknesses, and both the abrasive and ambient temperature adhesive

wear rates are about the same. The TiAlN-LF specimen is also similar, but has a low friction top coating that would not be expected to alter the abrasive wear, and this coating has essentially the same abrasive wear as the TiAlN-25 and TiAlN-51 coatings. Although the elevated temperature adhesive wear rates are different in that the TiAlN-25 specimen shows transfer of material from the coated specimen to the ball and TiAlN-51 shows transfer of material from the ball to the coated specimen, this is an artifact of the test. Breakthrough of the coating occurred for TiAlN-25 and in testing of the uncoated 4140 substrate, transfer of material from the 52100 ball to the 4140 disk was observed, so this is likely what is also being observed in the case of TiAlN-25. In contrast, breakthrough did not occur for TiAlN-51 so its behavior would be expected to be different even though the coating for this specimen is the same as for TiAlN-25. Regardless of which way material transfers at 225° C, the adhesive wear rates at this elevated temperature are very low.

The Dup-TiAlN specimen is considered a type of duplex coating. The duplex coating in this case is one in which the substrate is hardened prior to deposition of the hard coating under the assumption that an increase in hardness of the substrate will lead to less potential for elastic and plastic deformation and subsequent delamination of the coating. This improvement should thus result in better overall performance [9]. Gas nitriding was used to harden the 4140 substrate in this study, and 4140 shows a significant increase in hardness due to gas nitriding. The test results for the duplex coating reported in Figure 2 contradict the expectation of better performance for both adhesive and abrasive wear testing [9, 10]. The abrasive wear rate of the duplex coating is slightly higher than for the non-duplex specimen of the same coating, and the room temperature adhesive wear rate is substantially higher for the duplex coatings. The reality is that the underlying duplex coating does not always provide improved performance and that the behavior will be highly dependent on the testing conditions. Compared to the study reported herein, the published adhesive wear testing from reference 10 of duplex TiAlN used much higher loading (60 N) where the increased hardness of the substrate would provide more value by limiting elastic and plastic deformation of the substrate. There are many applications with much lower loading and contact stresses such as sliding components in small arms, so in order to better represent these applications, the load in the current study was much lower, 3.34 N. The same holds true for the abrasive wear testing, where in the case of reference 9, the counterface is a hard 52100 steel ball where in this case, the counterface is a soft neoprene. This would result in again much lower contact stresses and less of a propensity for deformation.

The contact stresses in the BOTD abrasive wear test are only several hundred psi because the rubber ball deforms and the sand forms a socket that carries some of the load and substantially increases the contact area between the ball and specimen. Hardening the substrate would therefore not be expected to have much impact on the abrasive wear resistance, and that is what was observed in the BOTD abrasive wear test. In contrast, the initial Hertzian contact stresses in the BOTD adhesive wear test are much larger, about 110,000 psi, because there is no sand to carry the load and the steel ball does not deform as much as the rubber ball. Substrate hardness would be expected to have a bigger impact in this case, and the BOTD adhesive wear rate for the duplex coating suggests that it does have a negative impact. At these contact stress levels, the higher hardness and modulus of the nitrided substrate act to locally increase the contact stresses by reducing the contact radius. The substantially higher adhesive wear rate is attributed to this phenomenon.

Increasing the aluminum content of TiAlN coatings has been reported to increase the wear rate, and a slight increase in the abrasive wear rate is observed in Figure 4 for the AlTiN coating [11]. The BOTD testing also suggests that increasing the aluminum content increases the ambient temperature adhesive wear rate, which is due to the fact that increasing aluminum content increases chemical reactivity and thus adhesive wear between surfaces [12]. The TiAlN-LF specimen contains an amorphous carbon top



coat that should provide lower friction and that would also be expected to reduce adhesive wear, but neither of these expectations is observed in the BOTD test results. The relatively high contact stresses of the BOTD test combined with some deformation of the relatively soft 4140 substrate likely cause the amorphous carbon top coat to be quickly removed. None of the TiAlN and AlTiN specimens show low coefficients of friction, and increasing the aluminum content appears to increase the coefficient of friction. This reflects the fact that the deformation is important in determining the coefficient of friction and harder materials exhibit far less deformation than more ductile, softer ones. These deviation of the bench-scale test results from the expected performance based on fundamental materials properties demonstrate why it is important to not rely on the fundamental properties (if they are even available) and why efficient bench-scale testing, with representative testing conditions tailored to the specific application is so valuable.

For the testing conditions in this study, the best performing coatings are the AlCrN-based ones, which actually have a lower published hardness (3000 to 3200 HV) than do the TiAlN's (3400 HV), which further shows that hardness is not the only important property in determining wear resistance. The composition and nano-layer structure of these coatings is claimed by the manufacturer to provide higher shear strength and more toughness, which would explain why the AlCr based coatings perform better than the harder TiAlN ones. The manufacturer also claims that the AlCr based coatings have less of a tendency for adhesion than their AlTi and TiAl based counterparts, which is also seen in the BOTD test results. The published values of coefficient of friction are given as 0.25 for the AlCr-based coatings, as compared to 0.35 for the TiAlN coatings.

There is an important difference between the two AlCrN-based coatings and this difference sheds light on the crack growth inhibition provided by multilayer coatings. The AlCrN coating consists of a monolayer, whereas the AlCr (really AlCrN-based) coating consists of multilayers that offer better resistance to crack growth, Figure 5. The result of this difference is seen in the abrasive wear data, where it would be expected because of the better crack growth inhibition of a multilayer structure, than for the monolayer AlCrN coating. The abrasive wear rate of the multilayer coating is almost 75% less than that of the monolayer coatings. In contrast, the adhesive wear rates, which would not be expected to depend on crack growth inhibition since adhesive wear is driven by micro-welding between the two surfaces and pullout, are similar for the monolayer and multilayer coatings.

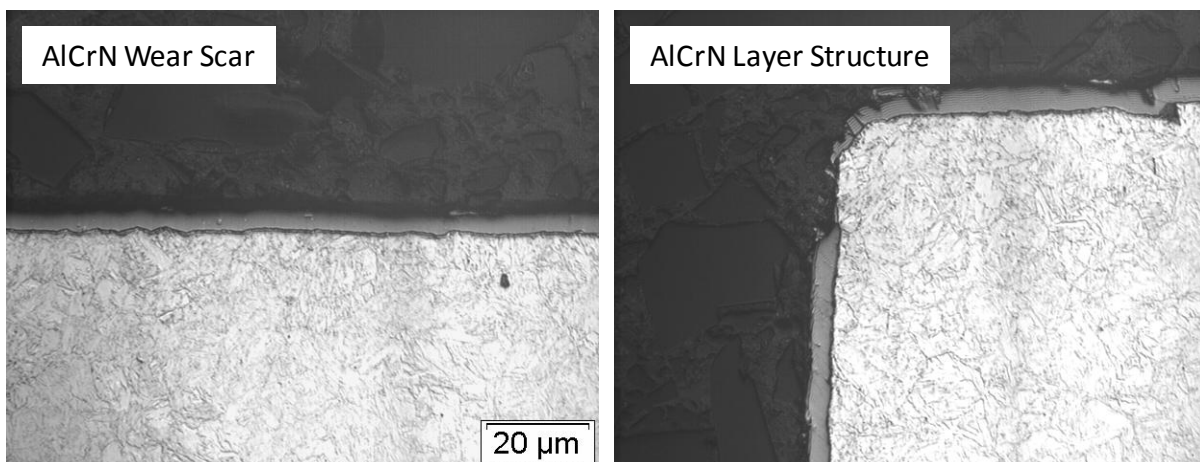


Figure 5 - Cross-sectional micrographs of as-deposited AlCrN and AlCr based coatings (1000x)



## 6. Conclusions

Ball-on-three-disk abrasive and adhesive wear measurements are extremely valuable for demonstrating the effectiveness of various coating design strategies and for generically comparing the performance of various coatings. The work described herein demonstrated the performance benefits of all of the modern coating strategies including superlattices and nano-composite coatings. These studies also demonstrated how the impact of the various coating design strategies differs for abrasive wear and adhesive wear.

This study demonstrated that the hard reactively sputtered nitride coatings provide the greatest wear resistance, but an innovative chrome coating that contains hard nano-particles equals the performance of the sputtered nitrides as long as the thickness is adequate and provides lower friction. The study also showed that recently developed AlCrN coatings that are claimed to have superior toughness as well as hardness do indeed provide greater wear resistance, with the multilayer version of the coating providing more wear resistance than the monolayer version of the coating. Nickel boron, which is a coating that relies almost solely on hardness, does not provide the same level of abrasion and adhesion resistance. In addition, it gives a significantly higher coefficient of friction than either the baseline material or uncoated steel.

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