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Abstract

There is a continuing requirement for high-performance tribological coatings in both commercial and military applications. To maximize system performance, corresponding improvements in wear resistance, high-temperature stability, corrosion behavior, and bearing durability must be realized. In our ongoing study, a number of different coatings were applied to 52100 bearing steel, 4340 steel, Inconel 718, and Ti-6A1-4V to improve wear characteristics, corrosion resistance, and rolling contact fatigue behavior. This report deals with CrN, TiN, W, and Ta coatings deposited by magnetron sputtering. Data on corrosion, Falex annular wear, ball-on-disk, and rolling contact fatigue are presented.

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1. Introduction

Military applications are constantly evolving, requiring ever-increasing performance requirements for single components and complete weapons systems. Additional environmental and budgetary concerns have further compounded the need for components that are benign to the environment and have low maintenance costs. For these reasons, the Department of Defense (DOD) and the U.S. Army Research Laboratory have pursued development of alternatives to hard electroplated chrome. Hard electroplated Cr is used widely throughout DOD and industry to provide a wear- and erosion-resistant coating on a wide spectrum of components, including bearing materials, large-caliber gun barrels, actuator tubes, etc. Hexavalent chrome (Cr^{+6}) is a known human carcinogen and is present in the electroplating and rinse baths. Both environmental and tribological improvements must be realized to meet the Army's future needs. Therefore, this work examines several magnetron-sputtered coatings as potential replacements for hard chrome electroplate.

2. Materials and Experimental Procedures

2.1 Sample Fabrication. Round flats, 2.50 cm in diameter and 0.63 cm thick, and round disks for Falex annular ring wear testing were machined from bar stock of each material tested to a standard surface grinding finish of between 3.2 μm and 1.6 μm. Rolling contact fatigue (RCF) specimens were finished to a diameter of 0.952 cm and surface roughness of 0.05 μm to 0.1 μm prior to coating application. See Glover [1] for more detail concerning the RCF sample dimensions.

2.2 Coating Application. Flat samples were coated using a single cathode magnetron sputtering process, while the RCF specimens were coated using a dual cathode in combination with a two-fold rotation device. The coatings were applied under the conditions in Table 1 (R - gas pressure × 10^{-4} torr, kW - DC power in kilowatts, V_S - substrate bias in volts, A_B - bias current in amperes):

Table 1. Coating Application Conditions

Coating	Gas Pressure $\times 10^{-4}$ (R) (torr)	DC Power (kW)	Substrate Bias (V_S) (V)	Bias Current (A_B) (A)
TiN	2.0	5	100	0.71
CrN	0.8	5	100	0.38
Ta	—	5	70	0.64
W	—	5	100	0.50

2.3 Corrosion Testing. Flat samples of coated 4340 were run through several cycles of a cyclic salt spray test that included intermittent salt spray, drying, humidity, and ambient cycles [2]. Uncoated portions of the specimens were masked to prevent premature attack of these regions, and visual observations were made throughout the test. A control specimen electroplated with a 25-mm hard chrome coating was also included in the corrosion testing.

2.4 Friction Coefficient Measurements. A ball-on-disk tribometer (Implant Sciences Corp. ISC-200PC) with a 440C 1.25-cm-diameter steel ball under a 100-g load (1 N), corresponding to a Hertzian pressure of 0.265 GPa and an average sliding rate of approximately 4 cm/s, was used to determine the unlubricated sliding coefficient of friction μ and to assess the wear rate.

2.5 Annular Ring Wear Testing. A Falex tribological machine was also used to perform wear testing. All samples were tested against a 1.25-cm-diameter Si_3N_4 ball at a sliding speed of 622.4 mm/s. A total of 20 ml of 15W-40 lubricant was applied to the test chamber before each test. Loads of 222, 444, and 888 N were applied. Specimens were typically run for 1 hr unless a sudden increase in friction was observed. A profilometer was used to determine the wear volume at four equally spaced locations across the wear track. The wear coefficient reported is given by:

wear coefficient = wear volume/(sliding distance * load).

2.6 RCF. All RCF testing for the present effort was performed on a ball/rod rig (developed by Federal-Mogul and now produced by NTN) [1], under the following conditions:

Hertzian stress	=	5.42 GPa (772,000 psi)
Rotational speed	=	3,600 rpm
Lubrication supply	=	8–10 drops per minute
Lubrication type	=	MIL-L-23699
Specimen length	=	76.2 mm +0.025/-0.000
Specimen diameter	=	9.52 mm +0.00000/-0.00005
Surface finish	=	0.10 to 0.05 μm. AA
Temperature	=	20–25° C

Four stations of the RCF rig were operated simultaneously. At least three wear tracks and associated fatigue spalls were obtained for each specimen condition, and the specimens were alternated among the test stations to minimize any systematic experimental error.

2.7 Hardness Testing. Nano-indentation tests were performed to determine the hardness of the applied coatings. An instrumented Vicker's indentor was used to record load-displacement curves for maximum loads ranging from 30 mN to 100 mN. The plastic hardness (Hp) was calculated for each maximum load. Each point represents an average of at least eight readings.

Hardness testing of the RCF specimens was done under the following conditions: 150-kg major load and a Rockwell "C" Brale indentor. The hardness data are shown for the RCF specimens in Table 2. Significant softening of the 52100 substrate occurred during the sputtering process, making the 8.0-μm W-coated and Ta-coated specimens too soft for RCF testing.

The IN-718, Ti-6A1-4V, and 4340 steel substrates were respectively 45.1, 40.9, and 38.6 HRC. This is too soft to attempt any RCF tests at typical loads for bearing materials. It should be noted that the 4340 samples were incorrectly heat-treated prior to machining, resulting in the substandard hardness.

Table 2. Rockwell Hardness of 52100 Steel RCF Specimens

Specimen	HRC
Uncoated	60.9
0.25-μm TiN	55.0
1.0-μm TiN	46.2
0.25-μm CrN	56.0
1.0-μm CrN	54.0
2.0-μm W	50.9
8.0-μm W	39.6
2.0-μm Ta	54.7

3. Results and Discussions

3.1 Nano-Indentation. The hardness of each coating tested is given in Table 3. Note the high hardness achieved by the W and Ta coatings. The 0.25-mm-thick nitride coatings were not tested due to the fact that the penetration depth at 30 mN exceeded the coating thickness.

3.2 Corrosion Results. The samples of coated 4340 showed very little resistance to pitting corrosion under the cyclic conditions. In less than two complete cycles, all of the coatings allowed significant pitting corrosion, including the specimen coated with electroplated chrome. The 8-km Ta coating performed slightly better, showing only a few isolated pits. It is evident that pinholes still exist in all of the magnetron-sputtered coatings evaluated. However, as the electroplated chrome did little to retard the pitting corrosion, this should not be viewed negatively.

3.3 Annular Sliding Wear. The wear coefficients determined from the annular ring testing are given in Table 4. As would be expected, the 52100 specimens consistently outperformed the other materials due to its higher substrate hardness. The metal nitride coatings improved the wear resistance of 52100 at all load levels, except for the 1.0-μm CrN coating at 444 N. The metallic coatings provided mixed results, but the 2.0-μm W coatings performed well at 222-N and 444-N loads. The wear coefficient of 1.31E-12 for 2.0-μm W on 52100 at 222 N was the lowest recorded in this study.

Table 3. Plastic Hardness of Coatings (Nano-Indentor)

Substrate	Coating	Penetration Depth (μm)	Hp (GPa)
4340	Ta - 8 μm	0.28±0.02	22±4
4340	Ta - 2 μm	0.29±0.02	17±2
4340	W - 8 μm	0.22±0.02	20±8
4340	W - 2 μm	0.25±0.01	22±2
4340	CrN - 1 μm	0.31±0.02	13±1
4340	TiN - 1 μm	0.34±0.02	11±2
52100	Ta - 8 μm	0.27±0.01	24±4
52100	Ta - 2 μm	0.27±0.03	24±10
52100	W - 8 μm	0.22±0.01	27±4
52100	W - 2 μm	0.24±0.02	26±7
52100	CrN - 1 μm	0.28±0.02	17±4
52100	TiN - 1 μm	0.25±0.02	24±6
IN-718	Ta - 8 μm	0.26±0.01	24±5
IN-718	Ta - 8 μm	0.26±0.02	19±3
IN-718	W - 8 μm	0.20±0.02	28±7
IN-718	W - 2 μm	0.22±0.02	23±6
IN-718	CrN - 1 μm	0.30±0.01	12±3
IN-718	TiN - 1 μm	0.27±0.03	16±5
Ti-6A1-4V	Ta - 8 μm	0.34±0.02	9.4±1.4
Ti-6A1-4V	Ta - 2 μm	0.33±0.02	11±2
Ti-6A1-4V	W - 8 μm	0.21±0.02	25±7
Ti-6A1-4V	W - 8 μm	0.21±0.02	25±7
Ti-6A1-4V	W - 2 μm	0.24±0.02	22±4
Ti-6A1-4V	CrN - 1 μm	0.31±0.01	13±2
Ti-6A1-4V	TiN - 1 μm	0.34±0.02	10±1

The greatest improvements were seen for the Ti-6A1-4V specimens when coated by the metal nitrides. Uncoated specimens of Ti-6A1-4V were generally off the scale, with wear coefficients in excess of 10^{-6}. The CrN and TiN coatings drastically improved the wear resistance, even to the point of approaching the behavior of uncoated 52100 at 444 N (Figure 1).

Comparison of the W coating to the Ta coatings generally shows better wear resistance for the W coatings, probably as a result of their higher hardnesses. No general statements can be made about thickness variations in these coatings.

Table 4. Annular Ring Sliding Wear Results

Wear Coefficient at 222-N Load				
Coating	521000	Ti-6A1-4V	IN-718	4340
Uncoated	1.10E-11	Off Scale	8.20E-09	4.50E-11
TiN - 0.25	1.03E-11	1.92E-11	2.41E-11	3.91E-11
TiN - 1.0	3.94E-12	2.35E-11	1.69E-11	5.67E-11
CrN - 0.25	5.95E-12	1.68E-11	2.69E-11	4.82E-11
CrN - 1.0	2.49E-11	1.69E-11	NA	4.07E-11
Ta - 2.0	1.71E-09	Off Scale	8.67E-09	1.41E-09
Ta - 8.0	1.33E-09	Off Scale	1.55E-09	1.23E-09
W - 2.0	1.31E-12	Off Scale	2.91E-11	5.54E-11
W - 8.0	1.89E-11	2.04E-11	2.42E-11	6.92E-11
Wear Coefficient at 444-N Load				
Coating	52100	Ti-6A1-4V	IN-718	4340
Uncoated	9.40E-12	1.31E-06	4.97E-09	9.13E-11
TiN - 0.25	4.97E-12	2.56E-11	2.86E-11	8.16E-11
TiN - 1.0	6.46E-12	2.24E-11	3.43E-11	5.44E-11
CrN - 0.25	7.38E-12	2.34E-11	3.76E-11	7.31E-11
CrN - 1.0	1.14E-11	2.27E-11	3.45E-11	8.43E-11
Ta - 2.0	1.68E-10	Off Scale	1.58E-07	2.47E-08
Ta - 8.0	7.85E-10	Off Scale	3.46E-09	8.41E-10
W - 2.0	4.81E-12	9.94E-07	4.23E-11	9.23E-11
W - 8.0	6.04E-11	9.74E-07	3.72E-11	8.58E-11
Wear Coefficient at 888-N Load				
Coating	52100	Ti-6A1-4V	IN-718	4340
Uncoated	1.41E-10	6.91E-07	1.29E-08	1.11E-09
TiN - 0.25	4.30E-11	5.11E-08	7.05E-11	1.02E-10
TiN - 1.0	6.07E-12	6.60E-11	6.41E-11	1.94E-10
CrN - 0.25	2.09E-11	4.82E-08	7.81E-11	1.46E-10
CrN - 1.0	1.12E-11	3.11E-09	7.14E-11	1.55E-10
Ta - 2.0	2.36E-10	2.83E-07	8.71E-08	1.47E-08
Ta - 8.0	6.49E-10	3.97E-07	2.73E-09	1.52E-09
W - 2.0	1.35E-10	2.01E-07	4.04E-10	2.23E-10
W - 8.0	5.65-11	4.08E-07	9.24E-09	5.14E-10

3.4 RCF. The RCF data in Table 5 show that the uncoated specimens had the highest lifetimes. The degradation in RCF life was caused by undesirable tempering of the substrate during the deposition of the coatings, as seen in the hardness data in Table 2. However, comparisons between the various coatings can be made. The B10 and B50 lifetimes correlate well with the Rockwell hardnesses. The slope of the Weibull plot shows that the 1.0-mm TiN and 2.0-mm W had the least variability in lifetime, which suggests that had the substrate not been tempered, these two coatings might have improved the wear resistance of the steel. Previous

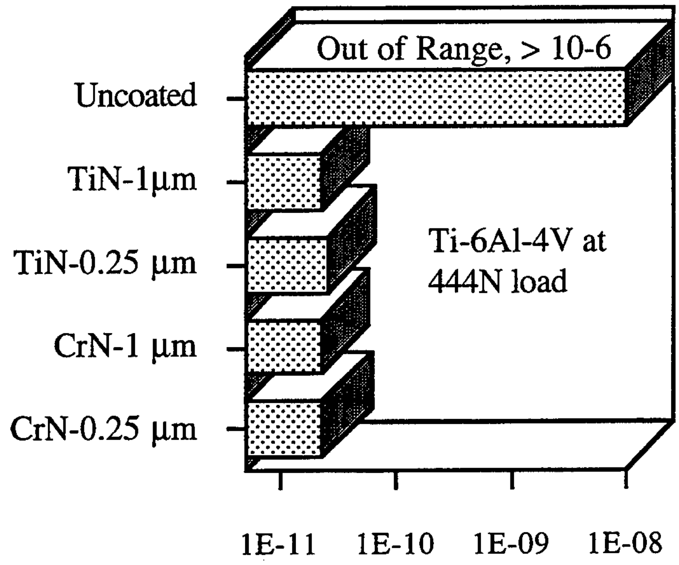

Figure 1. Annular Ring Wear Data for Ti-6A1-4V at 444-N Loading.

Table 5. RCF Lifetimes

Materials	B10 ($\times 10^6$)	B50 ($\times 10^6$)	Weibull Slope
52100 uncoated	4.70	27.92	1.06
52100–0.25 CrN	0.51	2.43	1.20
52100–1.0 CrN	0.34	0.92	1.91
52100–1.0 TiN	0.25	0.41	3.75
52100–0.25 TiN	0.18	0.56	1.62
52100–2.0 Ta	0.13	0.57	1.26
52100–2.0 W	0.07	0.13	3.14

work has shown that RCF lifetimes increased with coatings of TiN and CrN from 1 to 2 mm thick [3–4].

The softening of the substrate may represent a serious barrier to using magnetron sputtering to protect large-caliber gun tubes. Significant heating beneath the surface could allow for recovery of the plastic work that produces residual compressive stresses near the bore surface. It is critical that the Army address this problem at the outset of future gun-related studies.

3.5 Ball-on-Disk Results. The coefficients of friction, μ, vs. ball-travel distance for the 52100 substrates in the unlubricated ball-on-disk rests are shown in Figure 2. The initial μ measurements were similar on the other substrates. No other trends were readily apparent based on the available data. The Ta-coated 52100 steel showed both the smallest friction coefficient and the largest traveled distance before coating failure (Figure 2).

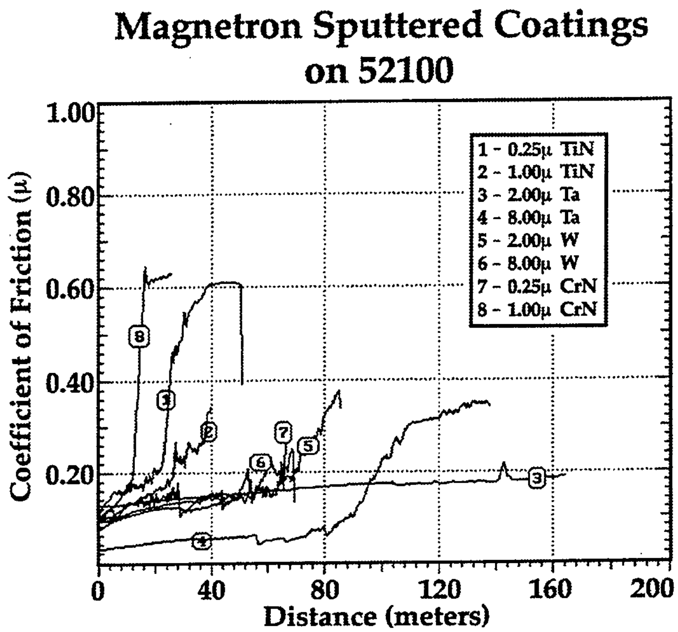

Figure 2. Ball-on-Disk Measurements on 52100 Steel.

4. Conclusions

1. The magnetron-sputtered coating of CrN, TiN, W, and Ta provided a very hard, wear-resistant surface.

2. Substrate tempering during the sputtering process reduced the effectiveness of the tribological coatings, especially in RCF. This could affect gun tube applications as well.

3. Both nitride coatings on Ti-6A1-4V provided greatly enhanced wear properties in the annular ring tests, approaching the properties of uncoated 52100 steel.

4. The unlubricated ball-on-disk tests were inconclusive from a wear standpoint.

5. Pinholes in all of the sputtered samples allow pitting at the specimen surface during corrosion testing. However, the behavior of hard electroplated chrome (25 mm) is similar.

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