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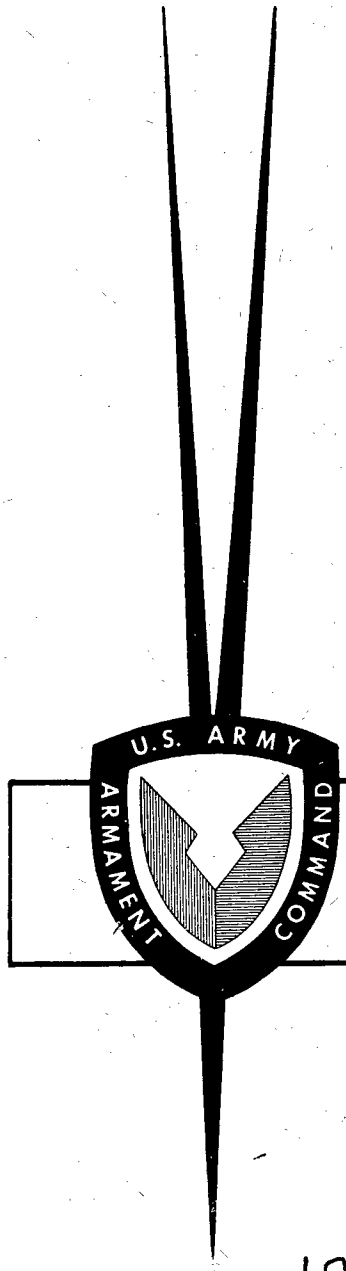
WEAR CHARACTERISTICS OF SPUTTERED VS. ELECTROPLATED CHROMIUM ON ALUMINUM

by

ANDREW CROWSON

January 1977



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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

The wear characteristics of sputtered chromium coatings on aluminum 7075 substrates were investigated and compared with electroplated chromium coatings. Both types of coatings were tested on a LFW-1 machine of the flat-on-cylinder variety under oscillating contact conditions. The structural characteristics of each type of chromium coatings have a definite relationship to the type of wear observed. The electroplated coated rings exhibited negligible wear during

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20. ABSTRACT (cont)

the initial cycle but eventually failed catastrophically by flaking and shearing of the deposits owing to its residual stress and cracked microstructure. The sputtered coatings, on the other hand, were found to be devoid of the residual stresses and cracked microstructure prevalent for the electroplated coatings. A conical-or columnar- shaped microstructure was exhibited. A uniform wear rate and lower overall wear of the sputtered coating was attributed to this microstructure and absence of gross defects. Evidence of abrasion and localized plastic flow on the sputtered wear surface was noted. (U) (Crowson, A.)

FOREWORD

The evaluation of the sputter coating process for weapon components was undertaken by Battelle Pacific Northwest Laboratories under MIPR M5-4-P0048-01-M5-W3. The results of this effort was published in Technical Report R-TR-75-042.

This report covers a segment of that effort involving wear characteristics of sputtered chromium as compared to electrodeposited chromium. This evaluation was performed by Rock Island Arsenal. This work was authorized as part of the Manufacturing Methods and Technology Program of the U.S. Army Materiel Development and Readiness Command and was administered by the U.S. Army Industrial Base Engineering Activity.

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INTRODUCTION

Chromium is the most extensively used wear coating applied to gun tubes. Its properties of high hardness, low coefficient of friction, and wear, corrosion and heat resistance make electrodeposited chromium an attractive material for the protection of internal bore surface of gun tubes. Electrodeposited chromium is also used extensively in other applications where sliding contact occurs between weapon components. However, with the impedus provided by the space exploration effort, newer methods to deposit improved coatings have been developed. Among the new coating methods, sputter coatings have been applied for electrical contacts, bearing and wear surfaces and for high reflectivity purposes. This report covers the evaluation of sputtered chromium as compared to its electrodeposited counterpart. This work was part of a project involving the evaluation of sputter coating techniques for processing weapon components.[1]

Electroplating of industrial or "hard" chromium resulted principally from the work of C. G. Fink and C. H. Eldridge.[2,3] Currently, hard chromium deposits are obtained by the electrolysis of standard solutions of chromic acid (CrO_3) and sulfuric acid (H_2SO_4) in self-regulating, high speed (SRHS) baths. Controlling factors of current density, solution temperature, chromic acid concentration, sulfuric acid concentration, and trivalent chromium affect the final properties of the deposit. In the electrodeposited condition the structure is much finer than in the cast or wrought form, and the crystal structure contains many faults and built-in oxide or hyride complexes.[4] Because of this complex structure and built-in stress pattern, a high hardness is developed. The resistance to wear of plated chromium is based on this hardness and on the fact that the deposited metal has a low surface energy.[5] The maximum hardness of a chromium coating is obtained only when applied to a thickness of several mils (0.003 to 0.015 in.) on a sufficiently hard base.[6]

---

1 Jones, R. H., Moss, R. W., McClanahan, E.D. and Butts, H. L., "The Sputter Deposition and Evaluation of Tungsten and Chromium Coatings for Use in Weapon Components," Technical Report R-TR-75-042, October 1975.

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Sputtering of chromium is carried out in an inert atmosphere at a low gas pressure. The atmosphere generally used is either argon or krypton. In this operation, the chromium metal is made cathodic and is bombarded by positive ions (formed by applying a high potential to the inert gas). As a result, chromium atoms are driven off into an adjacent gas phase towards the confining solid surfaces. A strategically placed substrate will collect expelled chromium atoms, which will eventually build up to form a thin coating. Conditions which influence the deposition rate are pressure and temperature of discharge gas; cathode fall and current density; cathode and collector geometry; and the nature of cathode material and residual gas.[7] Successful deposits of sputtered chromium up to 0.6 mils at deposition rates at 0.3 to 0.7 mil/hr. have been obtained.[8]

In this investigation, the wear characteristics of sputter versus electroplated chromium will be compared. Evaluations will be made to see if structural differences of the chromium deposited by these two processes play an important role in the wear phenomenon.

PROCEDURE

Wear measurements were determined with the LFW-1 friction and wear testing machine equipped with an oscillating drive. In this test, a stationary block is loaded against a ring, as shown in Figure 1. With an oscillating drive, the variable high-speed model of the machine is capable of oscillating the ring through a fixed arc, which may range from 0 to 90 degrees, at frequencies from 0 to 600 cycles per minute. An arc of 90 degrees at 200 cycles per minute was used in this investigation.

Standard size aluminum rings were either sputtered or electroplated with chromium. Sputtered chromium coatings on 1.375-inch diameter hollow cylinders of 7075-T6 aluminum alloy were provided by Battelle Pacific Northwest Laboratories (see Figure 2). The coatings were produced by a high-rate sputter deposition in a dc-triode system. Electroplated chromium coatings on 1.375-inch diameter aluminum rings were deposited following the conventional zinc immersion-copper strike surface preparation for aluminum metal.

Tests on the sputtered and electroplated chromium coatings were conducted at 10- and 20-pound loads against a standard tungsten carbide block. These loads were applied under dynamic conditions in incremental steps from zero to the desired test load to avoid abrupt load application. The total weight loss and wear scar length were measured after 3000, 6000, and 12,000 cycles.

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7 Holland, L., Vacuum Deposition of Thin Films, John Wiley & Sons, New York, 1960.

8 See Reference 1.

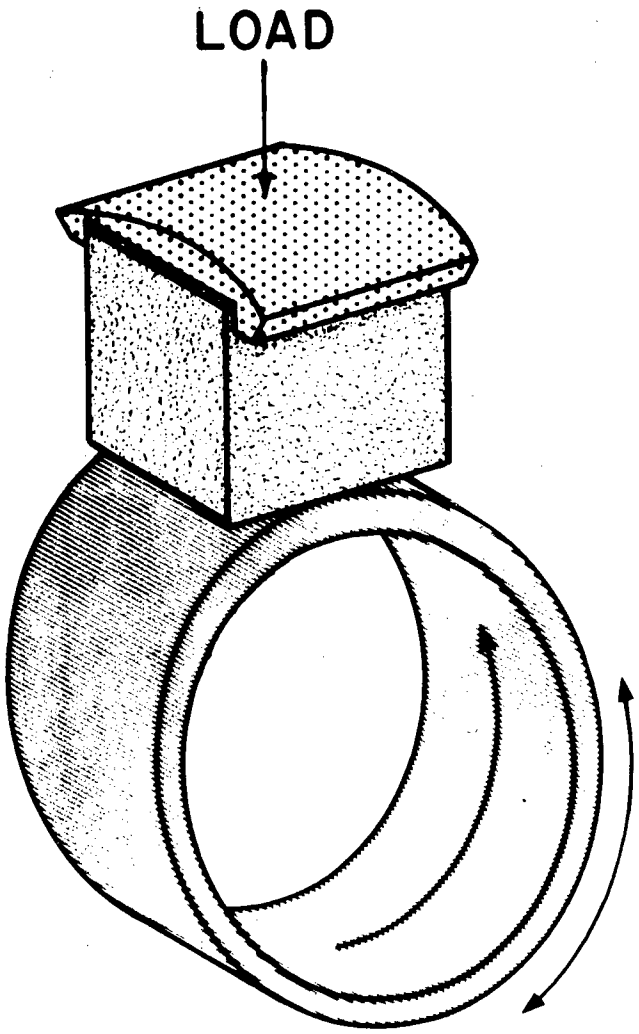
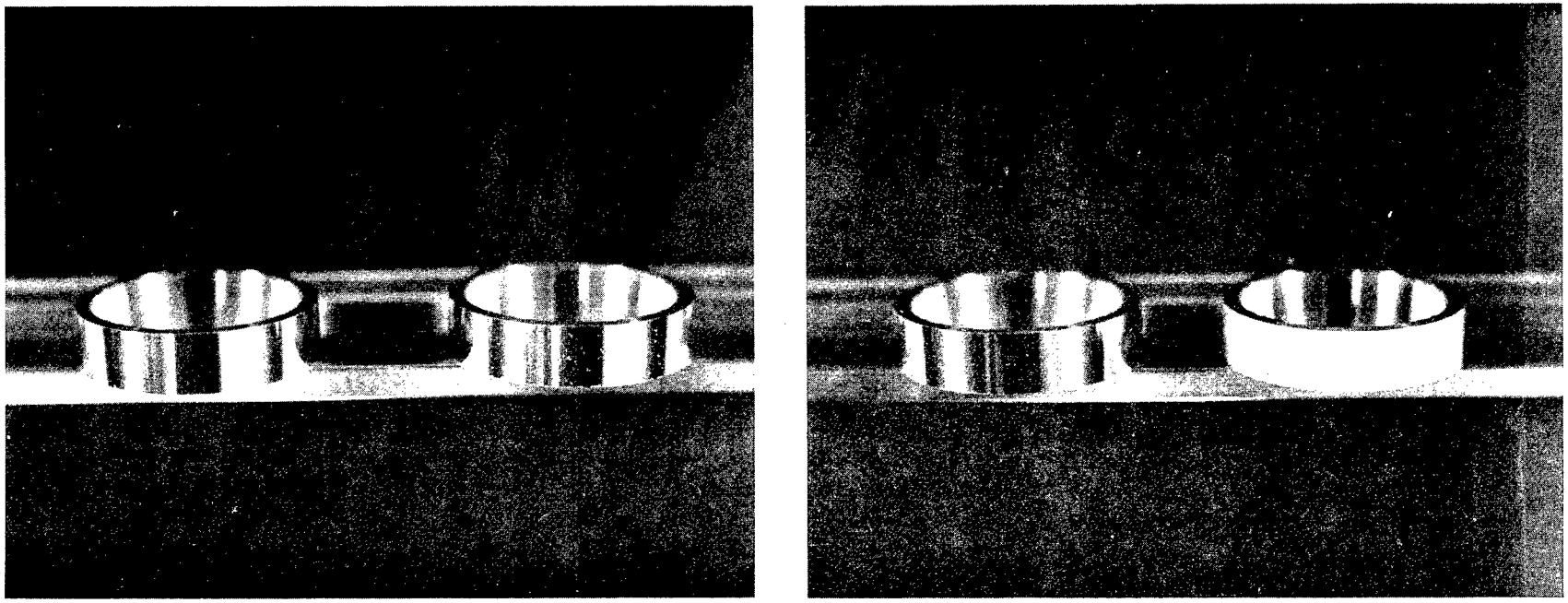


FIGURE 1 Test Configuration for LFW-1 Wear Tester.



Specimen No. 2A 1A 4A 3A

FIGURE 2 Typical Sputtered Chromium Deposits on 7075 Aluminum Alloy Rings

The chromium deposits were structurally evaluated by optical and scanning electron microscopy (SEM). Optical metallography of transverse and longitudinal sections was performed on the chrome-plated surface before and after wear to examine grain size and shape, deposit thickness, structural defects, and topography. SEM examinations were made on cross section areas using an eleven degree-taper through the wear surface with respect to the surface to be polished. This procedure facilitated simultaneous focusing on the wear and polished surface during SEM observation.

RESULTS AND DISCUSSION

Typical surface finishes of the sputtered and electroplated deposits used in this investigation are shown in Figure 3. The physical appearance of the sputtered chromium deposits (1-6) depended on the sputtering conditions used during deposition. A variety of surface finishes were obtained for the sputter deposits; ranging from a dull appearance to a bright finish with pitting noted in some of the samples. The hardness values of the plating were found to vary with the surface finish (see Table 1). Two different electroplated chromium finishes were used. Deposit Number 7 in Figure 3 shows a coarse grainy appearance whereas Number 8 shows a smooth bright finish. Diamond pyramid hardness values for these two coatings were approximately the same ($\sim$ 900).

SEM photomicrographs of the electroplated and sputtered chromium surfaces are shown in Figures 4 and 5. The chromium surface shown in Figure 4 has the characteristic dome-like projections prevalent for electroplated deposits. An inherent network of cracks is exhibited in the deposit. The pattern consists of crack-free areas ("plateaus") with fine grains. The basic cause of the cracks in the deposit was shown by Snavely to be related to the formation of unstable chromium hydrides of variable composition during the plating operation.[9] Figure 5 shows the characteristic surface of a typical sputtered chromium deposit. Unlike the uneven-crack surface of the electroplated deposits, a relatively smooth, powdery surface is exhibited by the sputtered deposits. Small crater-like defects are noticeable throughout the surface. The presence of particulates or perturbations on the substrate surface can cause the formation of such defects. Careful cleaning and handling of the substrates and target materials before sputtering deposition, however, help to minimize the number of these structural defects.[10]

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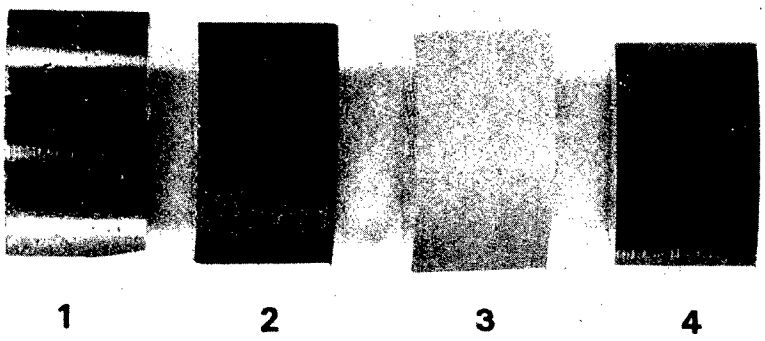
[9] Snavely, C. A., Trans. Electrochem. Soc., 71, 313 (1949)

[10] C.M. Jackson, J.G. Kura, J.F. Shea, V.D. Barth, A.G. Imgram, C.E. Sims and C.B. Voldrich, "Physical Vapor Deposition," Technical Report RSIC-574, March 1966.



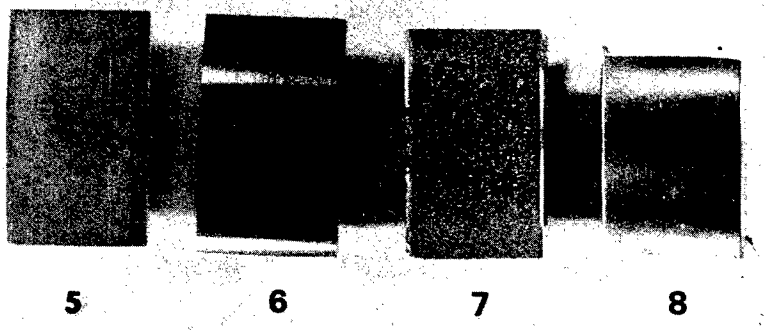
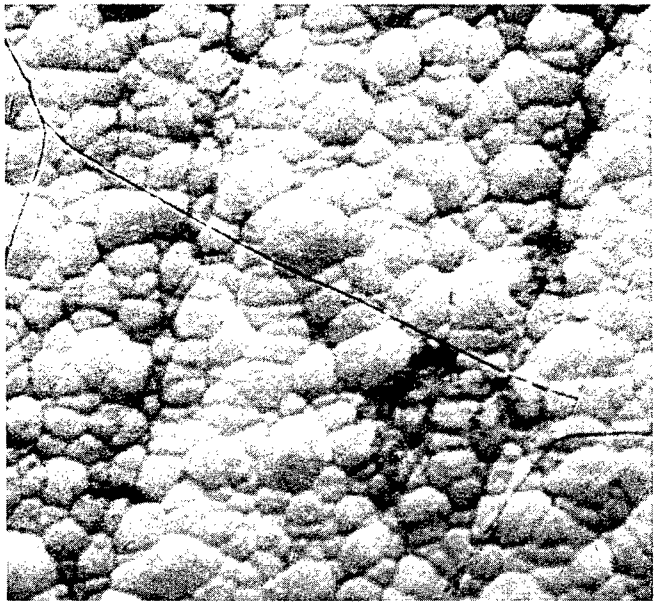


FIGURE 3 Surface Finishes of Sputtered (1-6) and Electroplated (7-8) Chromium Deposits



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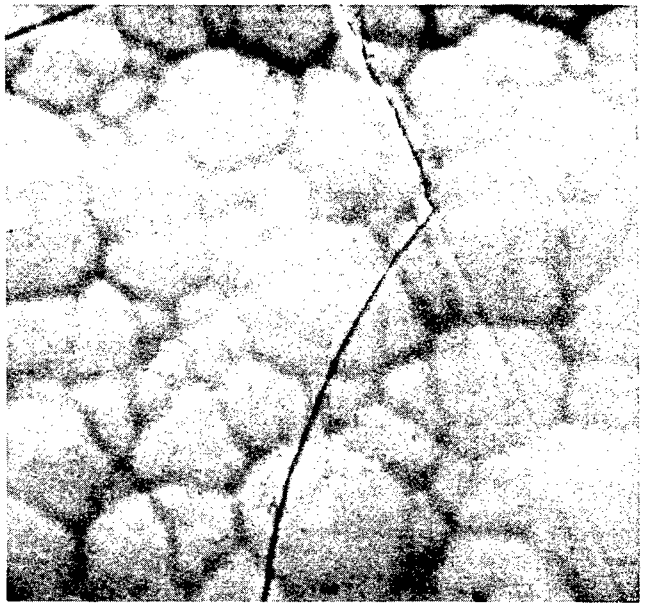

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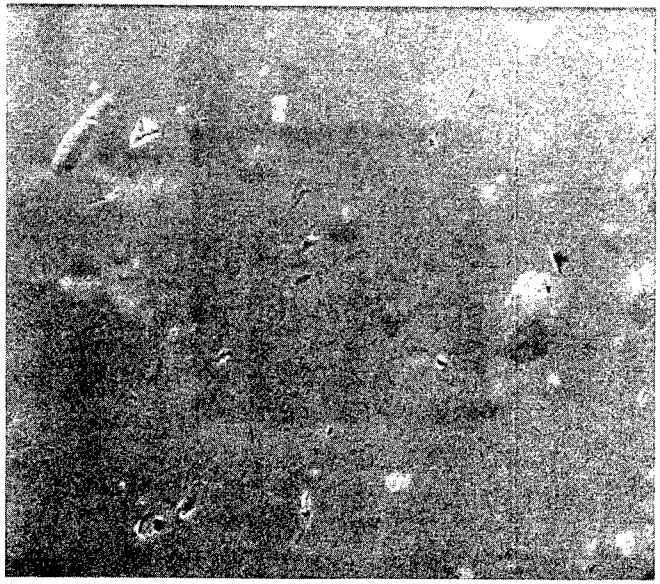
FIGURE 4 Surface of Electroplated Chromium Deposits



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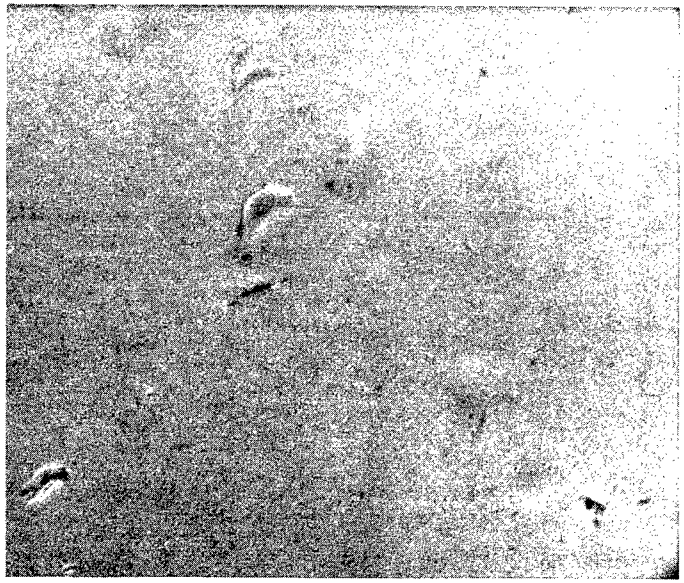

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FIGURE 5 Surface of Sputtered Chromium Deposits

Standard optical metallographs of electroplated and sputtered chromium coating cross sections are shown in Figure 6. The texture of the electroplated chromium deposit exhibits a characteristic small grain size. The grains formed by the sputtered chromium were conical- or columnar-shaped with a fiber texture perpendicular to the deposit plane. Inclusions and defects can be seen at the coating-surface interface for the sputtered deposit.

LFW-1 wear tests on the sputtered and electroplated chromium deposits are shown in Table 1. Wear may be defined as the removal of material from solid surfaces as a result of mechanical action. In general, it is a characteristic feature of the wear process that the amount of material removal is quite small.[11] In the case of the wear test data for the sputter and electroplated deposits this was found to be valid. Wear rates for the sputtered deposits ranged from 1.23 to 6.71 $X10^{-8}$ cm^3/cm kg. No relation between the hardness of the deposit and the wear rate could be ascertained. The cause for the difference in the wear rates for the various samples probably relates to the surface defects present in the deposit. Electroplated wear rates were found to range from 4.32 to 5.71 $X10^{-8}$ $cm^3/cm/kg$. A summary of the wear rates for the respective deposits is shown in Table 2 for comparison. In general, the electroplated chromium deposits had a higher wear rate than the sputtered deposits.

Explanation of the differences noted in the wear rates between electroplated and sputtered chromium deposits can be best understood after examination of the wear surfaces. In Figure 7, typical wear scars formed on electroplated and sputtered chromium deposits are shown. The sputtered chromium wore evenly throughout the wear cycle, i.e., layer by layer, whereas the electroplated chromium was susceptible to catastrophic failure by shearing and flaking. Closer examination of a typical section in the electroplated deposit where flaking occurred, reveals underlying, exposed aluminum (see Figure 8). SEM micrographs of a tapered section through the wear cross section are shown in Figure 9. Again, shearing and flaking of chromium plates are evident during the wear process.

Upon examination of a tapered cross section of sputtered chromium deposits, an absence of shearing and flaking during the wear process is revealed (see Figure 10). Evidence of abrasion and localized plastic flow on the wear surface is shown in Figure 11. This type of wear mechanism is consistent with the uniform rates measured.

CONCLUSIONS

Normal electroplated chromium deposits contain a network of cracks with resultant residual tensile stresses in the as-plated condition. Because of the complex structure and built-in stress pattern, cracks are

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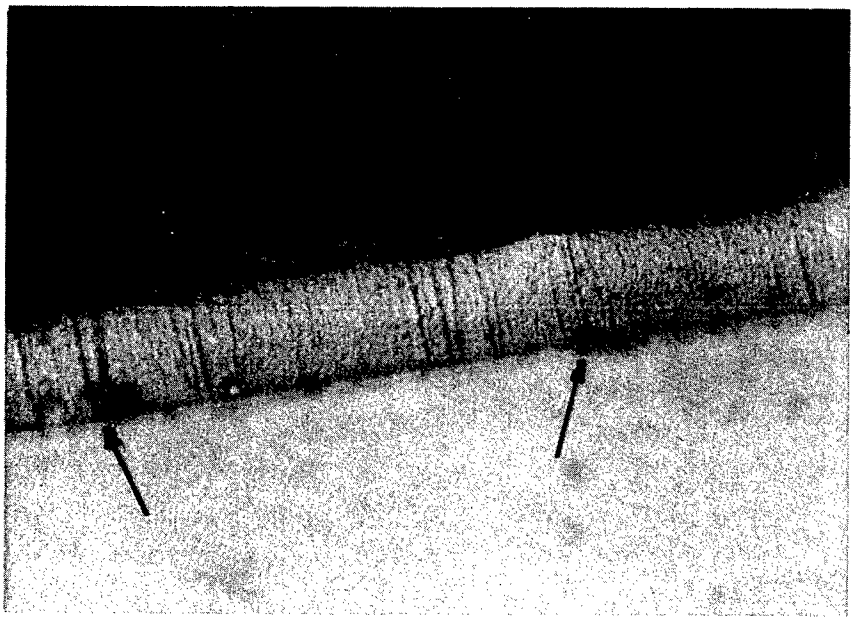
[11] Rabinowicz, E., Friction and Wear of Materials, John Wiley & Sons, New York, 1965.

CrO_3 Etch 500X

Electroplated Chromium



CrO_3 Etch 500X

Sputtered Chromium

FIGURE 6 Etched Cross Sections of Electroplated and Sputtered Chromium Deposits

The arrows indicate areas of particulate defects in the sputtered chromium deposits.

TABLE 1

LFW-1 WEAR TESTS FOR CHROMIUM PLATED 7075 ALUMINUM RINGS

Deposit[a]	Deposit Hardness (DPH)	Wt. Loss (mg)	Wear Scar (mm)	Load[b] (psi)	Cycles	Wear Rate (10^{-8} cm^3/cm-kg)
1A	677-732	96	26.3	37.7	6018	1.78
1A	------	120	26.5	35.8	6021	2.19
1B	------	110	26.5	37.9	6600	1.90
2A	572/677	84	26.1	37.5	6330	1.54
2B	------	144	27.5	33.9	6420	1.23
3A	775/836	316	27.2	35.5	12,540	2.79
3A	------	200	27.0	34.1	6480	3.45
3B	------	229	27.0	37.9	6420	4.00
4A	513/545	74	25.7	39.8	6660	1.30
4A	------	140	27.4	35.6	12,180	1.26
5A	710/774	342	26.5	36.2	6360	6.10
5A	------	374	27.4	35.6	6120	6.71
6A	753/777	235	27.7	40.8	6420	3.99
6A	------	256	27.7	39.7	6300	4.42
6B	------	249	27.1	39.7	6480	4.27
7	871/958	473	25.8	34.7	12,780	4.32
8	------	246	25.3	38.6	6240	4.71
9	------	310	25.5	37.2	6420	5.71

[a] Deposits 1A, 2A, 3A, 4A, 5A, 6A - Center sections of sputtered aluminum tubing
Deposits 1B, 2B, 3B, 4B, 5B, 6B - Off-center sections of sputtered aluminum tubing
Deposits 7, 8, 9 - Electroplated chromium deposits

[b] Tungsten Carbide block used - $R_c 75$

TABLE 2

SUMMARY OF WEAR TEST RESULTS

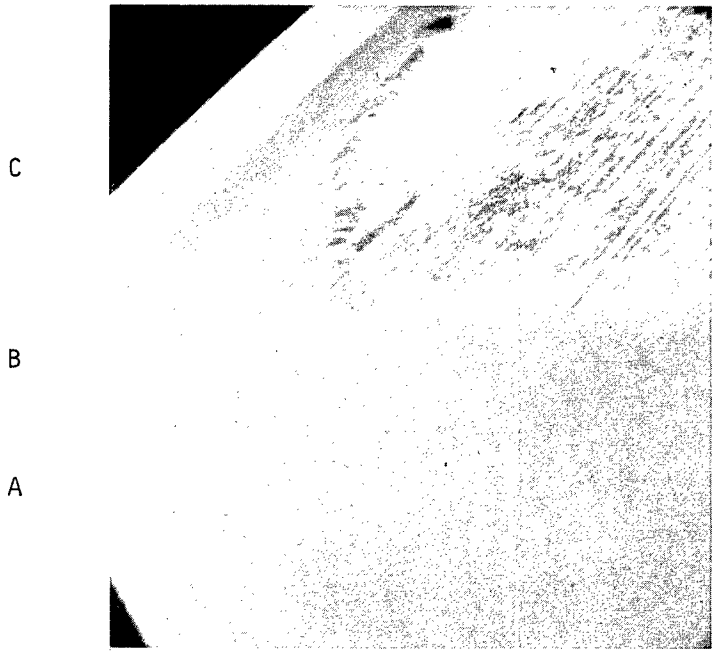
Deposit No.	Wear Rate (10^{-8} cm.^{3}/cm-kg)
1	1.96 ± 0.16
2	1.39 ± 0.16
3	3.41 ± 0.42
4	1.28 ± 0.02
5	6.41 ± 0.30
6	4.23 ± 0.16
Electroplated	4.91 ± 0.53

A B

FIGURE 7 Wear Scars on Electroplated (A) and Sputtered (B) Chromium Deposits

20X

FIGURE 8 Electroplated Chromium Flaking Observed During Wear



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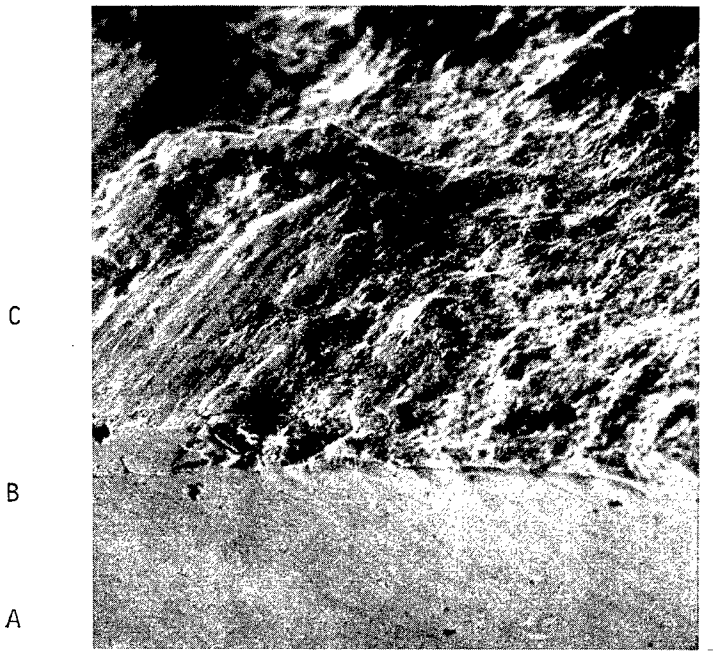

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FIGURE 9

SEM View of Electroplated Wear Sample Showing Substrate (A), Chrome Layer (B), and Wear Surface (C).

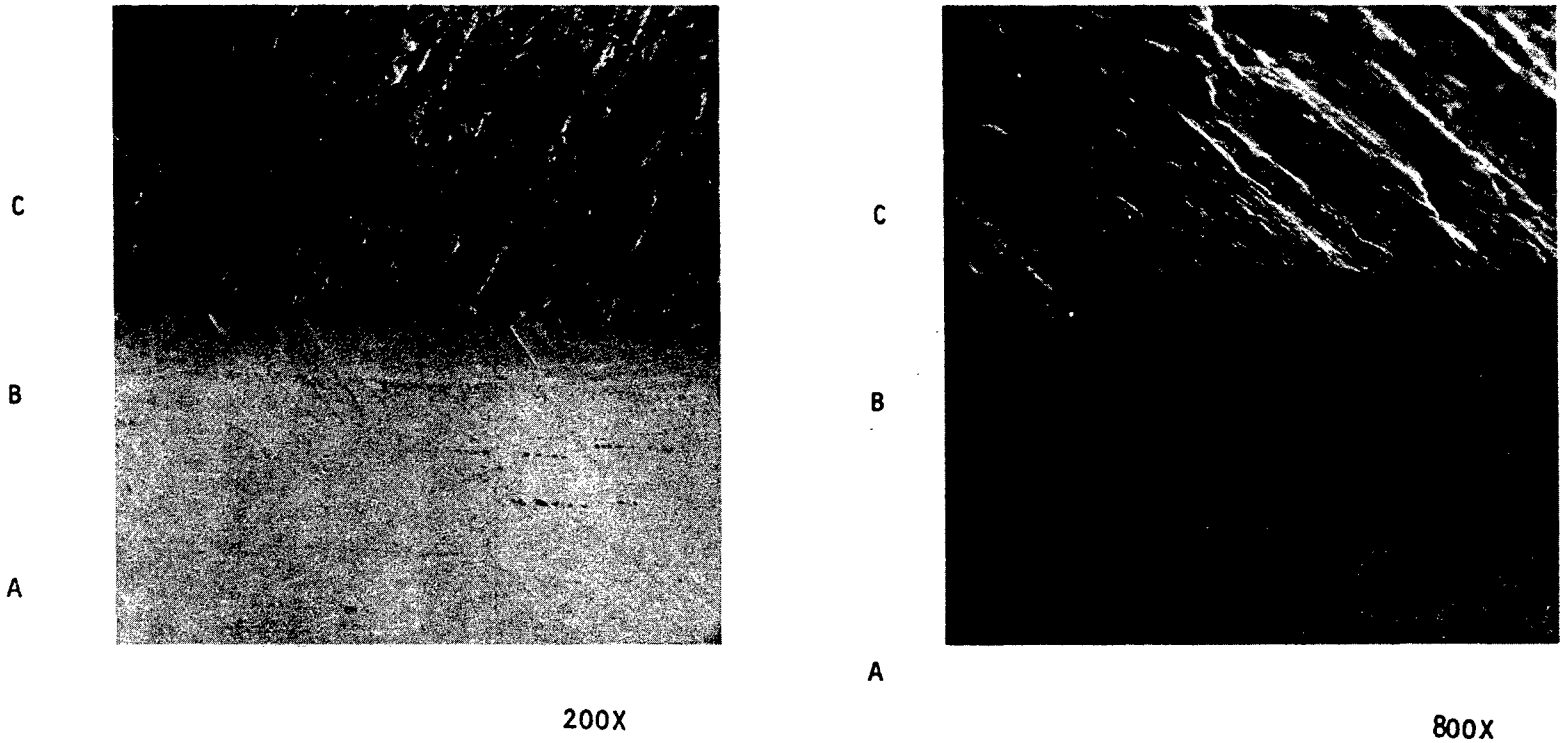

FIGURE 10 SEM View of Sputtered Wear Sample Showing Substrate (A), Chrome Layer (B), and Wear Surface (C).

FIGURE 11 Typical Wear Surface of Sputtered Chromium Deposits

FIGURE 9 Substrate (A), Chrome Layer (B), and Wear Surface (C). of Electroplated Wear Sample Showing

propagated early during wear with eventual crack branching and removal of chromium plates. As the plates gradually become detached from the surface, wear debris is formed and further enhances the wear rate of the chromium plating. Wear eventually occurs predominately by an abrasive wear mechanism.

Sputtered chromium deposits do not contain the residual tensile stresses nor the crack network found in electroplated chromium. Conical- or columnar-shaped grains with a fiber texture perpendicular to the deposit plane is exhibited. The wear mechanism which follows is based on abrasion and localized plastic flow. The wear is generally uniform, and a slower overall rate of wear occurs.

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