

**Continued Development in Chrome-Free Anodic Oxide Finishes for Aluminum:
Evaluation of Selected Mechanical Properties¹**

by

Jude M. Runge

Argonne National Laboratory
Chemical Technology Division
9700 S. Cass Ave.
Argonne, IL 60439

Phone: (630) 252-3430
Fax: (630) 252-9917
e-mail: runge@cmt.anl.gov

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**Continued Development in Chrome-Free Anodic Oxide Finishes for Aluminum:
Evaluation of Selected Mechanical Properties**

**Jude M. Runge, Ph.D., Argonne National Laboratory, Argonne, IL
Aaron J. Pomis, University of Illinois at Chicago, Chicago, IL**

In an effort to establish the composite anodic finish as an alternative to chromic acid (Type I) anodizing, comparative testing of Type I finished aluminum components with equivalent components finished with the composite anodic finish for aluminum has continued. This study compares the performance of components finished via typical Type I, II and III anodization processes to the performance of components finished with the alternative formulation under four (4) types of mechanical tests: abrasion resistance, fatigue, friction and thread torque. Differences in test performance show that the composite finished components meet or exceed the mechanical performance of components finished with the conventional anodic oxide finishes, regardless of type. The results indicate that the composite finish offers a chrome-free alternative for aluminum components that require finishing.

For more information, contact:
Jude M. Runge
Argonne National Laboratory
Chemical Technology Division
9700 S. Cass Ave.
Argonne, IL 60439
Phone: (630) 252-3430
Fax: (630) 252-9917
e-mail: runge@cmt.anl.gov

Introduction

The anodizing of aluminum is a sturdy fixture in the mind of the light metals industry. By capitalizing on the natural phenomenon of passive film formation on aluminum in a production environment, the anodization process has become synonymous with surface protection and durability of aluminum substrates. Anodizing is the deliberate, electrochemically controlled oxidation of the aluminum surface in aqueous solutions of sulfuric, chromic or oxalic acid. The resultant films are uniform, continuous and exhibit unique microstructures. Through its three most widely used variations, Types I, II, and III, anodic oxide finishes are used in various industries to impart protection, durability and decoration to aluminum component surfaces [1].

Of the three types of anodic oxide finishes most commonly specified, Chromic Acid Anodizing, designated by finishing specification MIL-A-8625 F as Type I, has been used in the United States, Great Britain and Russia principally for the treatment of aircraft parts because it imparts corrosion resistance, provides an excellent surface for subsequent coatings without affecting component tolerances and does not adversely affect component fatigue strength [2]. The durability of the Type I finish is a function of impact and scratch resistance.

The main disadvantage with Chromic Acid or Type I anodizing is the electrolyte, because of environmental and health concerns related to the discharge of hexavalent chromium in wastewater, air and solid emissions. There is a growing interest within the metal finishing industry and a tremendous effort among formulators within the chemical industry to develop an alternative formulation to the chromic acid electrolyte. The alternative must yield an anodic finish with attributes of corrosion and abrasion resistance at thicknesses that do not impact component tolerances or fatigue life. Types II and III, produced in a sulfuric acid electrolyte, are not appropriate for aircraft/aerospace applications because they are too thick as-finished and therefore affect component fatigue resistance.

Other chrome-free anodizing formulations that are available yield the Type IIB or "thin"

sulfuric acid anodic oxide finish and the boric acid – sulfuric acid anodic oxide finish. The former finish, produced at the common Type I thicknesses, does not exhibit comparable corrosion resistance to a chromic acid anodic oxide finish. Some metal finishers utilize the boric acid – sulfuric acid formulation as a substitute for the chromic acid formulation.

A new promising formulation of a chromium-free electrolyte utilizes an electroactive polymer additive to a sulfuric-acid based electrolyte. Studies have determined that at thicknesses typical for a Type I finish, the resultant composite anodic oxide exhibits superior corrosion and abrasion resistance as well as the ability to function as a dual-phase transition layer for polymer-metal bonding [3]. Extensive comparative mechanical testing has enhanced understanding of finish wear at the microscopic level. This study compares the performance of components finished via typical Type I, II and III anodization processes with the performance of components finished with the alternative formulation under four (4) types of mechanical tests: abrasion resistance, fatigue, friction and thread torque.

Scientific Background

Process and Microstructure

Extensive Transmission Electron Microscopy (TEM) has been performed on a variety of anodized finishes, processed in various ways. Analysis results consistently show that process modifications, whether chemical (to the electrolyte) or electrical (to the electrical input) impart distinct microstructural changes to the anodic oxide compared with the microstructures of anodic oxides formed through conventional processing.

By virtue of these changes and the predictable manner in which they can be achieved, the Constraint Concept of Film Formation was proposed. This theory explains how various oxide microstructural characteristics are achieved through electric field effects, as well as diffusion and mass transport that occur within the anodic oxide during anodizing, and how they change through modifications to the process [4].

The kinetics of anodic oxide film formation are governed by (1) the thermodynamics at the

surface and (2) diffusion and mass transfer across the oxide layer as it forms [5].

The columnar structure of the anodic film is the result of lateral film growth following surface reconstruction during early stages of the oxide growth process. As the “infant oxide” flakes impinge on one another, the repulsive forces of the similarly charged oxide flakes foster outward growth of the finish. Pores in the oxide finish are formed through repulsive field effects on the “inside” surface of the flakes, as the oxide flakes impinge and grow outward. It is apparent that diffusion occurs across the column wall, “knitting” the structure together. The stability and robustness of the final structure appear to depend on this stage of the film formation because there is no dynamic flux or ion flow that can disturb the formation of the final aluminum oxide species as in the pores. Consequently, the mechanical and chemical integrity of the finished film often is based on the integrity of the knitlines. See Figure 1.

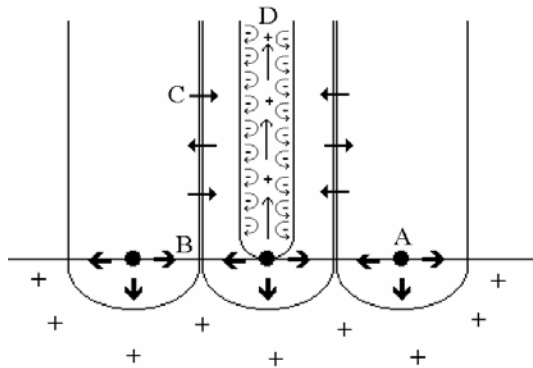


Figure 1: The Constraint Theory of Anodic Oxide Formation. A: Preferential nuclei form base of pore. B: Repulsive forces between similarly charged oxide “flakes” foster outward growth. C: Mass transport and diffusion across column walls form intercolumn “knitlines”. D: Repulsive forces within each pore maintain the erect nature of the column and dynamic flow of the electrolyte.

Pore wall reactivity enables adsorption of the electrolyte counter-ions and additives. In situations where the additive is not electroactive, adsorption occurs after substrate consumption is complete; however, it is apparent that counter-ion adsorption occurs throughout anodic film formation.

Reactivity/passivity of the adsorbed counter-ion dictates film thickness. An adsorbed reactive counter-ion facilitates charge transfer along the

inside surface of the oxide flake. The charge transfer fosters the oxidation reaction such that the repulsive forces of the ions promote pore formation and outward growth of the columnar microstructure. An adsorbed passive counter-ion, such as a chromate ion, will not facilitate charge transfer along the inside surface of the oxide flake and outward growth of the oxide layer does not occur, instead, would-be columns collapse on one another. Therefore, unlike conventionally processed Type II and Type III anodic finishes that exhibit a columnar microstructure, the Type I microstructure is coarse and disordered. See Figure no. 2.

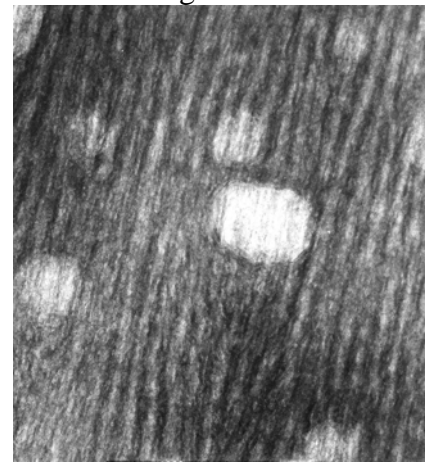


Figure 2: X100,000 TEM micrograph documents the microstructure of a Type II anodic finish. Note the unidirectional nature of the columnar microstructure.

The Type I microstructure exhibits a dense, random microstructure with a heavy interfacial reaction layer. The finish is thin, on the order of 1.5 to 2µm. These features are the result of the high temperature, high current density process parameters in an electrolyte that contains passive metal ions. From an engineering perspective, the random microstructure imparts the corrosion resistance of the Type I finish; the process parameters as well as the passive nature of the chromate ion limit finish thickness which in turn preserves component tolerances and fatigue resistance. See Figure 3.

Analysis of the composite anodic oxide finish produced in the subject chromium-free electrolyte determined it exhibits a unique hybrid microstructure. Instead of long, straight columns as Type II or Type III anodic oxides, or a dense, random microstructure as a Type I anodic oxide, the

composite finish exhibits a more cellular structure. The composite microstructure still has a columnar character but the columns are without a continuous, unidirectional central pore, giving a somewhat cellular appearance. In this respect, the composite microstructure somewhat resembles the Type I microstructure. These results indicate adsorbed electroactive polymer additive maintains pore wall surface reactivity enough to foster outward growth of the anodic oxide finish. See Figure no. 4.

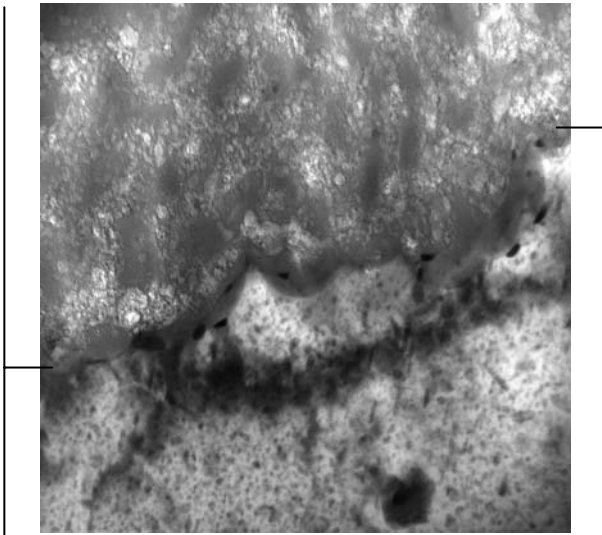


Figure 3: X100, 000 TEM micrograph documents the microstructure of a Type I anodic finish. Area above the central lines in the outer brackets is the anodic finish.



Figure 4: X100,000 TEM micrograph documents the microstructure of the composite finish. Note the differences in the microstructure as compared to the Type I and Type II finish microstructures. The composite microstructure appears as a hybrid of the two finishes.

Engineering Background

Wear and Hardness

Typically, an anodic finish is loaded in shear as well as in compression, as when a threaded fastener is seated or a piston articulates in a ring. The load is translated along the finish surface, and through the anodic oxide microstructure. For a Type II or Type III anodic finish, the shear stress produces angular displacement within the finish, and recovery of the structure depends upon its inherent mechanical properties and the continuity of the columnar microstructure. See Figure 5.

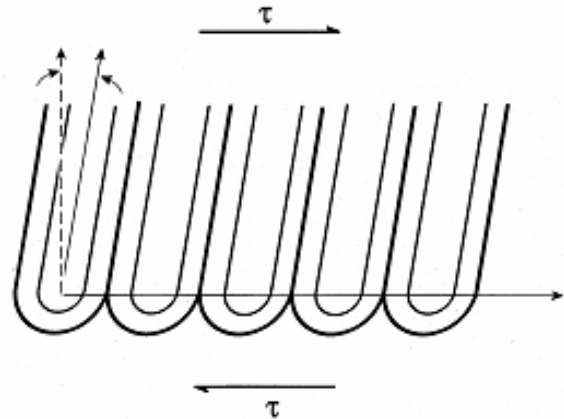


Figure 5: Schematic of the columnar structure of the Type II and Type III anodic finish under applied shear force (τ). The angle represents displacement of the structure through elastic deformation.

A material with a random microstructure, such as a Type I anodic oxide finish, will tend to smear or gall in shear while the same material with an ordered microstructure, such as a Type II or Type III anodic oxide finish, will exhibit some elastic recovery if the resolved shear force remains below the yield strength of the material. A material with a more ordered microstructure may even crack or spall if the yield strength of the material is exceeded. The random microstructure of the Type I finish does not have the mechanical capability to constrain shear force, as does the columnar microstructure of the Type II or Type III anodic finish.

Material failure due to excessive elastic deformation is controlled by the modulus of elasticity, and not by the strength of the material. Little metallurgical control can be exercised over the elastic modulus. Therefore, the most effective way to increase the stiffness of a component is to change its shape and/or increase the dimensions of its cross section. [6]. Because the anodic finishes, regardless of type, are chemically the same [7], the modulus of elasticity for the various types is also the same. Finish resistance to shear forces in wear is therefore partially governed by the robustness of the structure – in other words, by the ordered microstructure and thickness of the column walls. Clearly it follows that a Type III finish would exhibit superior wear resistance to Types I and II anodic oxide finishes.

Cohesive strength (the ability of a material to “hold together”) seems to greatly enhance the mechanical properties of a material in shear. Increased cohesion increases the ductility of the microstructure and therefore its resistance to fracture, making the material more “fracture tough”. For a material to have high strength and high toughness, other material conditions, such as hardness, are often compromised. This concept is sometimes counterintuitive as one imagines that harder means stronger. However, harder can also mean more brittle with lower cohesive strength.

Hardness of a material is a poorly defined term that has many meanings depending upon the experience of the person/people involved. In general, hardness usually implies a resistance to deformation. For metals the property is a measure of the resistance to permanent or plastic

deformation; for minerals and ceramics, hardness is measured as a function of how the materials abrade or scratch one another. There are a variety of standardized tests; each provides comparable data within the auspices of the specific test procedure.

The hardness of anodic oxides is typically obtained through microhardness testing. Metallographic cross sections are prepared and indented with Vickers indenter under a fixed applied load. The measurements are limited by the size of the indenter, and values are reported for Type II and Type III finishes which are thick enough to accommodate a meaningful indentation (3X the length of the long axis of the indenter). The typical microhardness measured for a Type III finish ranges from high 300 to about 600 Vickers Hardness Numbers (HV).

A note of caution is often sounded about the significance of hardness testing. When it comes to wear resistance, the hardness measured by microindentation methods does not always represent the performance of the material because morphological and microstructural characteristics impact the performance of the finish [8].

Substrate surface features can impact the surface morphology of the anodic finish. Interfacial and substrate defects such as burrs and laps on the macroscopic level, and grain boundaries and inclusions on the microscopic level can create knitline defects within the anodic oxide microstructure. Nonmetallic inclusions will not anodize and, as the adjacent substrate material is anodized the nonmetallic particles will be included within the finish. At the atomic level, defects such as vacancies and dislocations can “pile up”, leading to discontinuities in the anodic finish. Worst-case substrate defects can lead to discontinuities in the finish surface that serve as crack equivalents [9]. When the component is loaded in shear, defects in the surface and throughout the microstructure of the anodic oxide will act as stress raisers and exacerbate abrasive wear; these same features also detrimentally impact the fatigue resistance. See Figure 6.

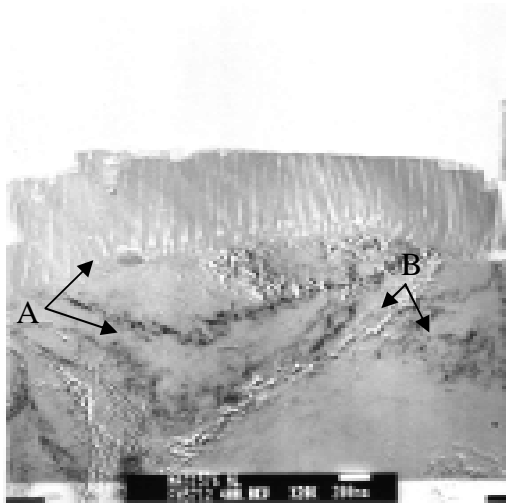


Figure 6: Crystallographic defects impact the way the anodic oxide finish nucleates and grows. A arrows indicate inclusions at the interface and in the finish. B arrow indicates an area with a grain boundary and dislocation “pile-up”.

Surface Effects and Fatigue

Practically all fatigue failures originate at a surface. The factors that affect the surface of a fatigue specimen can be divided into three categories; 1) surface roughness or stress raisers at the surface, 2) changes in the fatigue strength of the surface metal, and 3) changes in the residual stress condition of the surface. Surfaces are also subjected to oxidation and corrosion and these changes can also impact the fatigue resistance of a specimen. When corrosion and fatigue occur simultaneously, the chemical attack greatly accelerates the rate at which fatigue cracks propagate [10]. Therefore, protecting an aluminum surface that is subject to cyclic loading from the environment by anodizing is common practice.

As Type II and Type III anodic oxide fails in shear it chips and spalls, and the finish breaks apart. Surface discontinuities, such as crazing cracks, on the anodic finish offer fatigue crack initiation sites. As tiny pieces of the finish are cracked from the anodic oxide and incorporated into the wear debris, the film wears rapidly. Oxide chards exacerbate wear at edges and asperities within the microstructure. Thicker, ordered Type II and Type III coatings tend to be more prone to this type of degradation. Therefore, the thin, less ordered Type I anodic oxide is the finish of choice in applications where the aluminum surfaces will be cyclically loaded, experience little to no abrasive wear,

corrosion protection is necessary and the possibility of decoration exists.

Experimental Procedures and Results

Except as designated, comparative testing was performed following standard test procedures for anodic finishes for aluminum and aluminum alloys per MIL A 8625 F. Quantitative data for Type II and Type III finishes and equivalent data for the composite finish is provided for comparison for tests where the thickness of the Type I finish precluded testing, or only qualitative data for the Type I finish was available (e.g. abrasion resistance and pin-disk testing). We believe the results are important and germane to the use of the composite finish as a substitute for a Type I anodic oxide finish.

Abrasion Resistance

Comparative abrasion resistance was performed on coupons anodized with a Type I (chromic acid) finish and coupons anodized with the composite finish to the same 2 μm thickness. The testing was based on ASTM B571, “Adhesion of Metal Coatings to Metal Substrates”, paragraph 4, “Burnishing Test”. The qualitative results indicated that the composite finish exhibited increased abrasion resistance; the Type I film was easily scratched, exposing the aluminum substrate, while the composite finish was not scratched with the same tool [11].

Fatigue Strength

Reverse bending fatigue test bars were precision machined per ISO 1143-1975 (E). The test bars were machined from one rod each of aluminum alloy 2024 T4 and 7075 T6. Five samples were left unfinished, six were anodized in the gage (test) area with a chromic acid (Type I) anodic finish to a thickness of 1.5 to 2 μm (.00006 to .00008 inches), and six were anodized with the same thickness of the composite finish. The endurance limit for each alloy group was established through reverse bending fatigue testing of the unfinished test bars for $N = 107 - 108$, at a frequency of 1,000 to 9,000 cycles per minute. The finished bars for each alloy group were tested following the same profile.

The fatigue data was collected for each group and plotted as S-N curves (stress (ksi) vs. number of cycles). Neither the Type I nor the composite finish adversely affected the fatigue strength of the 2024 alloy. Type I anodizing appeared to reduce the fatigue strength of the 7075 material at higher stress levels; the composite finish did not adversely affect the fatigue strength of the 7075 alloy. See Figures 7 and 8.

2024 CANTILEVER ROTATING BEAM

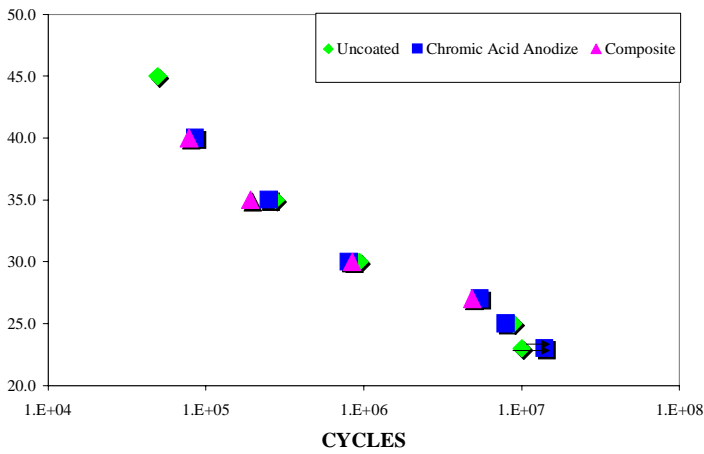


Figure 7: S-N curve for alloy 2024 T4 samples

7075 CANTILEVER ROTATING BEAM

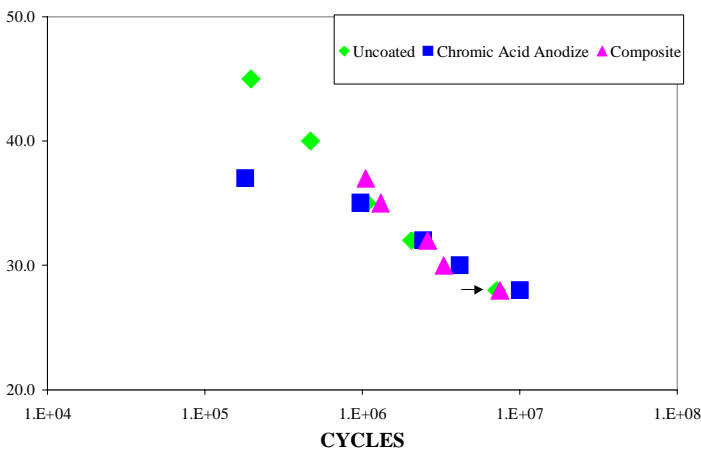


Figure 8: S-N curve for alloy 7075 T6 samples

Friction Testing

It is important to realize that interfacial friction values are determined per material system. Comparison can be made between wear couples only when a value for the coefficient of friction, μ , has been established for a material system control. In the case of determining the coefficient of friction for a coating, it is imperative that one also realizes the value for μ does not depend only on the coating; there are also substrate considerations. In determining what finish performs best it is necessary to evaluate precision within a specific material system and to compare that precision as well as the values for the coefficient of friction between the different finish groups.

For the following tests, the parameters were varied as follows. For the pin-disk tests, weight loss and μ values were determined and compared with abrading pairs in which the same finishes were wearing against one another under the same external test conditions. For the torque tests, μ values were determined individually for different finishes under the same test conditions and then compared.

Pin-Disk Testing

Tribological characteristics of Type II, Type III and corresponding composite anodic finished aluminum samples were determined by way of pin-disk abrasive wear/friction testing. The samples were tested under standard laboratory conditions at 23°C and 50% relative humidity throughout.

The pin tip radius was precision machined to 40 μ m. The test plates were 10 cm X 10 cm sheet samples machined from 6061 T6 aluminum alloy. Four sets of the two sample groups (the pins and the “disks”) were finished as described above. The Type II and composite samples were finished to a thickness of 20 μ m; the Type III and hard composite samples were finished to a thickness of 40 μ m. The test program was set up such that the pin-disk sample pairs were finished identically.

The test apparatus was set up such that a finished pin rotated at a rate of 0.3 m/s for 1 hr against a finished plate. A normal force of 5 Newtons was applied. Mass loss of the pin and the disk were determined with an analytical balance. The depth of the wear track was measured with a

laser profilometer. Coefficient of friction values were calculated by dividing the normal force by the frictional force.

The weight loss data for the pin-disk test, both the mass loss of the disk and the mass loss of the pin determined the Type II finish exhibited the greatest weight loss. The composite finish performed significantly better than the Type II films. Type III and hard composite finishes exhibited the lowest weight loss. Additional testing consistently showed that the hard composite finish exhibited the lowest weight loss with corresponding shallower wear tracks. See Figure 9.

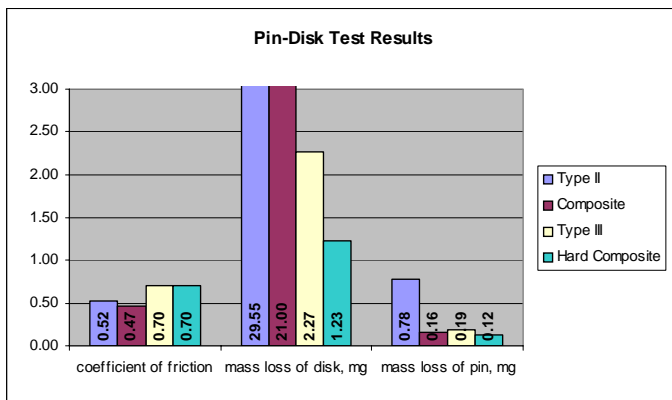


Figure 9: Pin-Disk Test Results. Note the range in mass loss values for the finished disk samples and correspondence to the mass loss of the pin.

The coefficient of friction for pin-disk pairs with the same finish was consistent within sample groups. The Type II samples exhibited an average μ value of 0.52; for the composite finish samples, an average μ value of 0.47 was determined. Type III and hard composite finishes were determined to have approximately the same μ value, 0.70 [12].

Torque Testing

Torque testing was performed per the German Industrial Specification DIN 946 “Determination of Coefficient of Friction of Bolts and Nuts under Specified Conditions” in order to determine and compare the coefficient of friction of various types of finishes on aluminum fasteners.

The applied load utilized to insert a fastener depends upon the coefficient of friction within the threads. This applied load ultimately governs the integrity of the bolt-nut joint. An over-torque condition damages components by galling of the

threads and may exceed the yield strength of the bolt base material. Under-torque conditions lead to fatigue problems within the joint. Therefore, the assurance of reliable wear characteristics of a given finish will reduce the likelihood of galling within the joint and help achieve a precision load for a given torque.

Comparative testing with threaded aluminum fasteners was performed. Alloys tested were aluminum alloy 7075 and 7278; finishes tested were the anodic composite finish, standard sulfuric acid anodizing (Type II), and yellow dichromate conversion coating. The average finish thickness for the anodized coatings was 15 μm . Steel nuts (alloy 34CrMo4), were used as test mates. One of three lubricants was used on the nuts: MoS₂, cetyl alcohol, or lanolin. Each bolt was tightened to a fixed load, removed, and retightened ten (10) times. Load versus thread torque was charted. The coefficient of friction in the threads was determined as the slope of each charted excursion. Precision could be directly observed on the curves; overlapping data indicated an absence of finish galling for the applied load.

The load-torque curves for the fasteners finished with the yellow dichromate conversion coating exhibited increased thread torque values for each tightening. This progression suggested that the finish galled with each insertion. The load-torque curves for fasteners finished with the standard anodized finish also exhibited increasing thread torque values, but to a lesser degree. The composite finish yielded precise load torque curves, with the slope (coefficient of friction at the threads) decreasing slightly with each tightening.

A direct comparison of the change in friction values of Type II anodized threaded fasteners and composite anodized fasteners with a MoS₂ lubricant on the steel nut showed that $\Delta\mu_{\text{type II}} = 0.08$ with μ values increasing with each load excursion, and $\Delta\mu_{\text{composite}} = 0.04$ with μ values decreasing with each load excursion. These results indicate that the composite finish exhibits antigalling characteristics superior to the other finishes tested. See Figure 10[13].

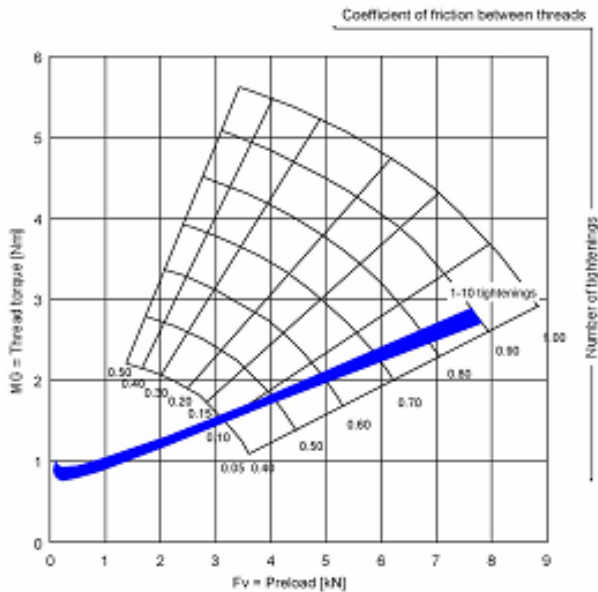


Figure No. 10: Torque test data for composite finished aluminum fastener, MoS_2 lubricant and steel bolt. The data band represents all ten load excursions. The data overlap indicates the finish exhibits good antigalling properties.

Conclusion

A promising chromium-free electrolyte formulation for aluminum anodizing utilizes an electroactive polymer additive to a sulfuric acid based electrolyte. Previous studies have determined that at thicknesses typical for a Type I finish, the resultant composite anodic oxide exhibits superior corrosion resistance as well as the ability to function as a dual-phase transition layer for polymer-metal bonding. Extensive comparative mechanical testing has enhanced understanding of finish wear at the microscopic level. This study compared the performance of components finished via typical Type I, II and III anodization processes to the performance of components finished with the composite formulation under four (4) types of mechanical tests: abrasion resistance, fatigue, friction and thread torque.

Regarding abrasion resistance, at thicknesses comparable to a typical Type I anodic oxide finish, the composite finish could not be scratched or abraded to the point of exposing the aluminum substrate. Similarly, in torque testing, the composite finish exhibited superior antigalling characteristics as compared to other finishes, even conversion coatings, against the same type of bolt and lubricant. In pin-disk testing, the composite finish exhibited weight loss consistent with the

Type III or “hard” anodic oxide finish, although it had been processed at Type II parameters.

It is believed that the ability of the composite finish to withstand abrasion and galling at all thicknesses lies in the ordered-disordered hybrid microstructure. When subjected to shear forces, the composite structure is ordered enough to constrain the force and deform elastically, yet it is disordered enough not to chip or spall. The level of disorder appears to impart a level of compliance to the microstructure that does not impact the fatigue strength of the base material when the composite finish is at thicknesses comparable to Type I anodizing.

The preceding experimental results indicate that in specifying finishes for applications normally relegated to Type I finishes, serious consideration can be given to the composite finish. Should actual application tests determine the composite finish performs comparably to Type I, added benefits can be derived in the form of energy savings, as the oxide formation current density is lower and the process temperature is ambient for the composite finish. In addition, the chrome-free electrolyte avoids pollution hazards and waste treatment costs associated with Type I anodizing processes.

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