Conf-950304--8

SAND94-3141C

PROCESSING AND PROPERTIES OF CHROMATE-FREE CONVERSION COATINGS ON ALUMINUM

R.G. Buchheit, C.A. Drewien, M.A. Martinez
Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185

G.E. Stoner
Department of Materials Science and Engineering
University of Virginia
Charlottesville, Virginia 22903

ABSTRACT

A conversion coating method has been developed based on precipitation of $Li_2[Al_2(OH)_6]_2 \cdot CO_3 \cdot 3H_2O$ from alkaline lithium salt solutions. The process is procedurally similar to chromate conversion coating but does not use or produce hazardous chemicals. The coating that forms is polycrystalline, continuous and conformal. The coating meets the MIL-C-5541E corrosion resistance, electrical contact resistance and paint adhesion requirements for certain aluminum alloys, but does not match the levels of performance exhibited by chromate conversion coatings.

In this paper, methods for producing the coating are described. Corrosion resistance has been characterized using electrochemical impedance spectroscopy and salt spray exposure. The structural, compositional and property changes attending post-coating thermal exposure are discussed. Performance in standardized corrosion, electrical and paint adhesion tests is also presented.

Keywords: hydrotalcite, talc conversion coatings, chromate conversion coatings, electrochemical impedance spectroscopy, aluminum alloys.

INTRODUCTION

Traditional surface finishing technologies for enhancing corrosion resistance of aluminum and its alloys have relied heavily on hexavalent chromium as an active bath agent or pigment. These

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This work was performed at Sandia National Laboratories sponsored by the U.S. Department of Energy under contract no. DE-AC04-94DP85000.

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technologies include conversion coating, hard- and color-coat anodizing, cleaning, deoxidizing, plating, priming and painting. These technologies are mature, trusted and strongly entrenched in manufacturing, field repair and refurbishment operations. However, hexavalent chromium is a known human carcinogen and has been identified as hazardous chemical in major federal regulations including the Clean Air Act, the Clean Water Act, the Pollution Prevention Act, National Emission Standards for Hazardous Air Pollutants (NESHAP), Resource Conservation and Recovery Act (RCRA), the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and the EPA's 33/50 Program. These regulations are being strictly enforced by federal, state and local authorities. It is clear that even under the most favorable circumstances, many traditional surface finishing technologies will be employed only under strictly controlled conditions and with high added cost. For this reason industry-wide efforts are underway to identify, develop and implement simple and cost effective methods for achieving desirable surface properties using means that neither use or produce toxic substances.

We are currently engaged in an effort to develop a high performance, low -cost coating method for aluminum alloys that does not use or produce hazardous chemicals. The process under investigation involves non-electrolytic deposition of an inorganic hydrotalcite, $Li_2[Al_2(OH)_6]_2 \cdot CO_3 \cdot 3H_2O$, (hereafter referred to as "talc") coating by precipitation from an alkaline lithium salt solution. This paper describes methods used to deposit coatings, the physical and chemical characteristics of the coating and the current levels of performance that have been demonstrated in a variety of tests.

EXPERIMENTAL PROCEDURES

Coating and Painting Methods

Talc Coatings. Talc coatings were formed on coupons prepared from 1100 (Al-1.0(Fe,Cu,Si)), 2024-T3 (Al-4.4Cu-1.5Mg-0.6Mn), 5052 (Al-2.5Mg-0.25Cr), 6061-T6 (Al-1.0Mg-0.6Si), and 7075-T6 (Al-5.6Zn-2.5Mg-1.6Cu) sheet stock. Panels were prepared for coating by manually washing using a water-soluble alkaline detergent, degreasing with an alkaline non-etching solution and deoxidizing in an acidic bath typically containing NaBr. Flowing distilled water rinsing was performed in between each step. Details of the nominal coating process are shown in Table 1. Coating was performed by immersion in an aqueous lithium salt solution with a pH of 11.2 to 11.5 at ambient temperatures or at a temperature controlled to 55±3° C. Because the coating forms by a co-precipitation reaction involving aluminate $(Al(OH)_4^-)$, the bath was conditioned by adding 200 ppm aluminate as sodium aluminate $(NaO \cdot AlO)$ or potassium aluminate $(KaO \cdot AlO)$ upon make-up of new bath solution. Once prepared, coupons were immersed for a minimum of 5 minutes to form the coating, removed, rinsed and allowed to air dry. Coupons were allowed to age undisturbed for a minimum of 24 hours prior to any further handling. Some coatings were subjected to isothermal heat treatment in air at temperatures ranging from 25° to 300° C to study the effects of post coating heat treatment on coating structure and properties.

Chromate Conversion Coatings. Aluminum alloy coupons were also prepared using a commercial chromate conversion process sufficient to produce coatings that meet MIL-C-81706 Class 1A (maximum corrosion resistance) requirement. These coupons were tested in parallel to talc coatings for comparative purposes.

Painted Samples. Talc conversion coated, chromate conversion coated and zinc chromate epoxy primed (per MIL-P-23377) 6061-T6 panels were painted using two-coat spray applied high solids white urethane. The top coat solids content was 26 to 33 % by volume and had a thickness of 2.0±0.3 mils (50±8 micrometers). An additional set of talc coated and zinc chromate primed 6061-T6 panels were painted using a spray applied polyurethane enamel 1.0 to 1.25 mils thick with a solids content of 26 to 33 % by volume. These samples were subjected to a variety of paint adhesion studies described at the end of this section.

Characterization Methods

X-ray diffraction (XRD). Grazing incidence XRD was performed using a Siemens D500 powder diffractometer equipped with a receiving beam graphite crystal monochromator and a 2.2kW long focused Cu X-ray tube. A step scan rate of 0.02°/min. was employed. A grazing angle of 0.2° provided signal from 1000 to 5000 Å into the sample surface.

Secondary Ion Mass Spectroscopy (SIMS). Al and C (from carbonate) negative ion and Al and Li positive ion composition depth profiles were determined for talc coated specimens using a Cameca *ims* 4F SIMS instrument. SIMS was performed using an O^- primary ion beam with an accelerating voltage of 10 kV and a beam current of 50 nA. The beam was rastered over a 100 μm by 100 μm area. The field aperture was selected so that ions generated from a 8 μm^2 diameter circular area in the center of the rastered area were admitted into the spectrometer. Ions with energies within ± 10 eV of the peak yield energy were included in the ion count.

Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Plan view secondary electron images of coated surfaces were obtained using a JEOL 6400 SEM operated at 15 kV. To provide further characterization of the coating structure, coating cross sections were prepared for TEM examination. The TEM samples were prepared by cutting 10 mm x 3 mm wide slices from the coated aluminum substrate. The coated surfaces were glued face to face under pressure with Epon 828 epoxy to form a block. After curing, slices were cut, ground through 1 μm diamond to a thickness of 200 μm, and sectioned to 3 mm diameter disks. The disks were dimpled and then ion-milled to perforation. Thin foil samples were examined using a JEOL 2000 FX, operated at 200 kV and equipped with and energy dispersive spectrometer.

Thermal Analysis. Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) spectroscopy were performed to determine points of major weight loss and components lost as a function of increasing temperature. Experiments were conducted using 2.2g of hydrotalcite under flowing N_2. The samples were heated at a rate of 10° C/min. from 25° to 800° C.

Electrochemical Corrosion Testing

Electrochemical Impedance Spectroscopy (EIS). The barrier properties of talc and chromate conversion coatings were evaluated in air-sparged 0.5 M NaCl solutions under free corrosion conditions using EIS. Measurements were carried out in a Princeton Applied Research (PAR) flat cell modified to accommodate a specimen with a 16 cm^2 exposed area. The impedance experiments were initiated after three hours' exposure to solution. Free corrosion potentials were typically in the range of -0.715 to -0.730 V after 3 hours in solution. Data were collected using either a PAR 273 potentiostat/Solartron 1255 frequency response analyzer (FRA) combination, or a Solartron 1286 electrochemical interface/1250 FRA combination. Each system was controlled by ZPLOT impedance software package installed on a personal computer. Typically, measurements were made at frequencies ranging from 65 kHz to 5 mHz by sampling at 10 points per decade frequency using either a 10 or 20 mV sinusoidal voltage perturbation. At any frequency, the measurement signal was integrated to minimize the effects of spurious components to the measured signal. Total resistances were determined either by circle fitting the data plotted in the complex plane, or by integration of the imaginary component of the impedance[1]. Total capacitances were determined from the slope of the $Y''(\omega)$ vs. ω plot.

Performance Testing

Corrosion Resistance. Corrosion resistance was evaluated as prescribed in MIL-C-5541E, "Chemical Conversion Coatings on Aluminum and Aluminum Alloys"[2] which specifies testing by exposure to a 5% salt spray environment for 168 hours per ASTM B117[3]. Coated coupons were given a pass or fail ranking based on the number of pits present. Coated panels failed if more than 5 pits larger than 0.031 inches (0.0787 cm) in diameter were present per 150 square inches (968 cm^2) of exposed area. Otherwise, panels were given a pass ranking. Discoloration of the panels was not a failure criterion.

Electrical Contact Resistance and Surface Resistivity. Low electrical contact resistance is required in certain military and aerospace applications where contacting components of housings and cases must be at the same ground potential. MIL-C-5541E specified contact resistance measurements were performed by impinging a 1.0 in^2 (6.45 cm^2) copper platen on the coated aluminum surface under a 200 lb (91 kg) load[4]. Resistance was determined with an ohm meter accurate to 0.0001 ohms. At least four measurements per specimen were collected.

Four-point probe type resistance measurements were also made on experimental coated panels and uncoated control panels. These measurements were made using a Veeco Instruments, Inc. Four Point Probe Model FPP-100 resistivity tester. Measurements were conducted by impinging four sharp steel pins onto the test surface under an approximate load of 200 lb. (91 kg). Four measurements were made for each sample.

A third type of surface resistivity evaluation was made using a Leighton Electronics, Inc. mercury (Hg) drop probe. In these experiments, a 1 mm diameter Hg droplet was brought into contact with the test panel surface. Electrical contact was also made with a steel platen on an abraded portion on the backside of the specimen. A 50 mV voltage was applied across the specimen and impedance measurements were made at 0, 10^3, 10^4, 10^5, and 10^6 Hz. DC resistance measurements were made using a Solartron 7151 Computing Multimeter. For resistances greater than about $2 \times 10^7\ \Omega$, measurements were made using a Guildline model 9520 teraohmmeter. Impedance measurements were made using a Hewlett Packard model 4270A automatic capacitance bridge. Typically, three measurements were made for each sample.

Paint Adhesion. MIL-C-5541E specifies testing of paint adhesion according to Federal Test Standard 141, Method 6301[5] which was conducted in the following manner. Painted panels were immersed in distilled water for 24 hours, then removed into ambient air. Two parallel scribe marks, one inch apart, were made through to bare metal using a sharp knife. Adhesive tape was then laid perpendicular over the scribes and immediately removed. If any paint was removed as the tape was withdrawn, the panel was given a fail ranking. If no paint is removed the panel was given a pass ranking.

In addition to the above test, dry tape (pull) testing was conducted according to ASTM D3359[6] in which scribes were made in a cross hatch pattern through to the metal substrate. Tape with a minimum adhesive strength of 35 lb. per inch (width) was laid across the crosshatch, and immediately removed. The amount of paint removed was assessed visually and ranked in 20 percent increments, (eg. 40 percent of paint removed from surface). A variant of this test was conducted by exposing crosshatch scribed painted samples to salt spray exposure for 500 h then performing the tape pull evaluation. Performance ranks were assigned in the 20 percent increments described previously.

Paint "creepback" measurements were also made along scribe marks made in painted panels that were subjected to 500 h salt spray exposure. Here, paint "creepback" refers to the distance corrosion (either filiform or blistering) has propagated under the paint away from the scribe. Typically, four measurements were made at equally spaced points along the scribe per sample using an optical microscope.

RESULTS

Coating Structure, Composition and Physical Characteristics

Under macroscopic examination, coated surfaces exhibit a slightly dulled or matte finish compared to an untreated surface. Slight interference coloring or iridescence is observed for surfaces with thin coatings, a distinctly white translucent film is observed for thicker coatings. The surface is not discolored in any other way.

Figure 1 is plan view SEM image that shows the typical morphology of a talc coated surface using the standard deposition process. The coating consists of intersecting crystals that form a continuous layer across the surface. The coating forms in pits and recesses that develop during pre-coating cleaning operations and is therefore continuous and conformal. From examination of a plan view micrograph, it is not clear whether the interstices between crystals penetrate through to the metal surface. However, as shown in the TEM cross section in Figure 2, a dense layer of coating is present below the outer layer, and the interstices do not penetrate through this inner layer. Selected area diffraction of the coating inner layer indicates that it is amorphous or poorly crystalline material. As shown in Figure 3, sputter depth profiles acquired using SIMS suggest that compared to the outer layer, this layer is depleted in carbon (from CO_3^{2}), and rich in Li which may indicate the presence of a lithium aluminate rather than hydrotalcite.

Coating thickness has been observed to depend on the alloy substrate composition[7], coating bath composition and age[8], and immersion time[8]. For 1100 Al and 6061-T6, typical coating thicknesses range from 1 to 3 μm for immersion times of 15 minutes. Coating thicknesses in excess of 5 μm have been observed for coating bath immersion times of 3 to 5 hours.

Corrosion Resistance Determined by EIS

Figure 4 shows representative Bode plots determined by EIS after 3 hours' exposure to 0.5 M NaCl for (1) 1100 Al with a chromate conversion coating formed using Alodine 1200S process[9], and (2) 1100 Al with a conversion coating subjected to a post coating thermal aging treatment (3 hours at 70° C). This comparison shows that the talc coated sample exhibits a larger total resistance than the chromate conversion coating. The heat treated talc coating does not always exhibit better barrier properties than chromate conversion on 1100 or 6061-T6 during impedance testing. However, the performance is usually comparable.

Long term exposure of talc coated 1100 Al to aerated 0.5 M NaCl solution shows that barrier properties of the coating are retained for approximately 80 hours. Figure 5 shows open circuit potential (E_{oc}), total resistance (R_t), and total capacitance (C_t) as a function of exposure time up to 425 hours (17.7 days). After 80 hours' exposure E_{oc} falls from -0.750 V_{sce} to -0.975 V_{sce} while the C_t increases from 5.5 μF/cm^2 to a peak of about 10 μF/cm^2. R_t exhibits a sharp decline between 50 and 125 hours consistent with the onset of coating failure by pitting. The slight increase in R_t after 125 hours is attributed to the formation of a gelatinous hydrated aluminum that was observed to form at the specimen surface. Examination of the exposed surface after testing confirmed that pits had formed during this test.

EIS data from various talc coated and chromate coated alloys exposed to air-sparged 0.5 M NaCl are shown in Table 2. With the exception of chromate conversion coated 6061-T6, total resistance values are lower than 10^7 Ω·cm^2 which has been suggested as a minimum value required to achieve a passing rank in salt spray testing. Notably, many samples prepared identically to those reported in Table 2 passed salt spray testing. Capacitance is highly sensitive to pitting with large values indicating the presence of pits on the surface. EIS experiments were initiated after 3 hours' exposure to solution, and examination of total capacitance indicates that pitting had occurred in some cases. The largest capacitance values are observed for 2024-T3 which has the least intrinsic corrosion resistance among the alloys tested. The talc coating on 2024-T3 offers little if any corrosion protection and is considerably inferior to its chromate conversion coated counterpart. Heat treatment of the talc coating produces a marginal improvement. High total resistances and low capacitances indicating good coating protection are exhibited by talc coated 1100 and 6061-T6. Heat treatment appears to cause increased corrosion protection.

Corrosion Resistance After Thermal Exposure

Corrosion resistance offered by chromate conversion coatings is lost if the coating is exposed to temperatures in excess of 160° F (71° C)[2]. Anecdotal reports attribute this phenomenon to dehydration and crystallization of the coating. This issue is of concern due to increased use of electrostatically applied paints that are consolidated and cured at elevated temperatures on chromate conversion coated surfaces.

Upon heating, talc coatings lose water and experience a phase transformation, but their corrosion protection increases. Figure 6 is a plot of the time derivative weight loss versus temperature for pure isolated hydrotalcite determined by TGA for temperatures ranging from ambient to 800° C, although for practical applications with aluminum only changes that occur below 200° C are of relevance. Four major episodes of weight loss were observed. Episodic weight loss was attributed to loss of H_2O or CO_2 as indicated on the plot. Water is evolved at temperatures greater between 100 and 200° C, and a mixture of water and CO_2 (from carbonate) are evolved at 200 to 270° C. XRD data sets , presented in Figure 7, collected from talc coated Al powder subjected to isothermal heat treatments over the sample temperature range show that the evolution of water is accompanied by the transformation of hydrotalcite to bayerite ($Al(OH)_3$) as indicated by diminishing 002 hydrotalcite reflection at 11.7° 2θ beginning at 115° C. In spite of the compositional and structural changes indicated, no dramatic morphological changes are observed in the talc coating up to 300° C as is illustrated by comparing scanning electron micrographs in Figure 8 (25° C) and Figure 9 (300° C). No cracking of the coating is observed, and at the macroscopic level, the coating is unchanged in appearance after heat treatment at any temperature up to 300° C. Total resistances (R_t) calculated form EIS data show that significant gains in corrosion resistance are expected upon heating at low temperatures (Figure 10). A slight loss in R_t is observed at intermediate temperatures where the water loss rate is greatest, and the slight further gain at the highest temperatures is likely due to a contribution of thermal oxidation. The EIS data are corroborated by salt spray results which show that coated 1100 and 6061-T6 panels subjected to heat treatment achieve passing ranks after 168 h of exposure (Table 3).

Corrosion Resistance, Electrical Contact Resistance, and Paint Adhesion

Corrosion Resistance. The coating industry standard test for corrosion resistance of inorganic coatings on aluminum is an exposure test carried out at 95° F (35° C) using a fog or spray generated from a 5% salt solution. Aluminum alloys with properly formed chromate conversion coatings regularly survive this exposure test without any visible signs of corrosion. Table 4 summarizes salt spray test data for the range of alloys studied. This table reports the minimum coating time used to achieve a passing rank, and indicates the maximum exposure time used to test that particular alloy. Thicker coatings are required to protect alloys with higher copper contents. Coatings can be grown sufficiently thick to protect 7075-T6, but a 14 μm thick coating on 2024-T3 does not survive salt spray testing.

Electrical Contact Resistance. Table 5 summarizes the electrical resistance measurements accumulated for chromate and talc coated 6061-T6. The MIL-C-5541E specification requires that Cu platen contact resistances be less than 5 $m\Omega/in.^2$ for a coated Al surface and less than 10 $m\Omega/in.^2$ for a coated Al surface after 168 h of salt spray exposure. Chromate coated 1100 Al and 6061-T6 specimens easily meet this requirement. Talc coated 1100 Al does not usually meet this requirement, but talc coated 6061-T6 does.

Four point probe tests yield resistance values that were essentially invariant among all samples tested. Since electrical contact is made by impinging sharp probe under load on the coated surface, it is likely that direct metal to metal contact is made in this test. Nevertheless, these data may still be important in that they reflect the tendency of both chromate conversion coatings and talc coatings to spall or break under impinging contact. From the design standpoint, this is significant when electrical continuity is required between joined parts in an assembly.

Among the three coating resistance evaluations, the mercury probe method is the only technique where metal to metal contact by breaking through the coating is avoided. As a result, these resistance data are more likely to reflect the intrinsic electrical properties of the coating. Table 5 shows that Hg drop probe coating resistances are much greater than those measured with the copper platen or the four point probe. Bare 6061-T6 exhibits resistances typical for native Al oxide film approximately 20 Å thick through which electrons can tunnel[10]. Chromate coated 6061-T6 also exhibits a relatively small Hg drop probe resistance (as do chromate coated 2024-T3 and 7075-T6)[11]. Talc coated specimens exhibit Hg drop probe resistances in excess of $10^9\ \Omega$, suggesting a relatively high electronic resistivity for these coatings.

Figure 11 shows a plot of coating impedances determined for 6061-T6 specimens using the Hg drop probe on dry coatings for frequencies ranging from 10^3 to 10^6 Hz. Coated surfaces are expected to act as a parallel resistor-capacitor combination under these test conditions. The negative slope of the log frequency-log impedance plot is consistent with such a model. Figure 11 shows that over the entire frequency range studied impedances for chromate coated specimens were less than for talc coated samples. Lowest impedances were observed for the uncoated surface. After salt spray exposure, impedances for the chromate coated samples increase while those for the talc coated sample decrease.

Paint Adhesion. Table 6 summarizes the paint adhesion data from a variety of standardized tests performed on surfaces primed with a Zn-chromate epoxy (zincate) primer, and chromate conversion coating and talc conversion coating used as primers. MIL-C-5541E, Method 6301 requires that no paint be removed from scribed painted panels after 24 hours' immersion in DI water. 6061-T6 panels given a zinc-chromate epoxy primer (zincated) prior to application of the paint passed this test. No 6061-T6 panels with the chromate conversion coating or the talc coating used as a paint primer have passed this test to date. In the ASTM paint adhesion tests (D3359 variants), talc coated specimens perform at a level comparable to their zinc chromate coated counter parts and could outperform a chromate conversion coating. No paint is removed from a cross hatch scribed regions of talc or zinc chromate coated panels tested in a dry condition. Moreover, no paint is removed from the cross hatch scribed region after 500 h salt spray exposure per ASTM B117. The last row of data in Table 6 refers to paint "creepback" which is the distance away from the scribe that underpaint corrosion penetrates during 500 h salt spray exposure. In this evaluation Zn-chromate epoxy primed 6061-T6 out performs its talc conversion coated counterpart.

Zn chromate epoxy primed and talc conversion coated 6061-T6 surfaces were also painted using a polyurethane enamel top coat and subjected to the same sets of paint adhesion tests. As shown in Table 7, the performance of the talc coated sample is identical to that of the Zn chromate epoxy primed sample.

SUMMARY

A polycrystalline film forms on aluminum by precipitation from an alkaline lithium salt solution. The predominant compound in this coating is $Li_2[Al_2(OH)_6]_2 \cdot CO_3 \cdot 3H_2O$ which belongs to a class of minerals known as hydrotalcites. The coating can be formed using methods similar to existing chromate conversion coatings processes, but does not use or produce hazardous chemicals. The coating offers desirable properties but does not match the performance offered by chromate conversion coatings. The coating does however, meet the corrosion, electrical contact resistance and paint adhesion performance requirements established in MIL-C-5541E. These coatings may have an advantage over chromate conversion coatings in that they retain good corrosion resistance when subjected to elevated temperatures.

ACKNOWLEDGMENTS

This work has also been support in part by Virginia's Center for Innovative Technology and the Center for Electrochemical Sciences and Engineering at the University of Virginia. The authors wish to thank M. Bode, C. Hills, B. McKenzie, M. Gonzalez, T. Neil and T. Tribble for support in electrochemical testing and characterization work.

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Table 1. Generic talc conversion coating process for aluminum alloys.

Process Step	Talc Coating Process	Comparison to Chromate Conversion Coating Process
Wash	commercial alkaline detergent	standard
Alkaline Degrease	commercial product or $Na_2CO_3/NaSiO_3$ (2 minutes at 65° C)	standard
Acid Deoxidize	commercial product or $HNO_3/NH_4(HF)_2$ (< 2 minutes at 22° C)	standard
Coat	7.4 g/l Li_2CO_3 plus 4.9 g/l LiOH pH 11.3 - 11.5 (5 - 15 minutes at 55° C)	low-toxicity replacement
Age	24 hours minimum	standard

Table 2. Impedance data summary for talc coated and chromate coated alloys.

Alloy	Chromate		Talc		Heat Treated Talc†		Bare	
	Log(R_{tot}) $\Omega\cdot cm^2$	C_{tot} $\mu F/cm^2$	Log(R_{tot}) $\Omega\cdot cm^2$	C_{tot} $\mu F/cm^2$	Log(R_{tot}) $\Omega\cdot cm^2$	C_{tot} $\mu F/cm^2$	Log(R_{tot}) $\Omega.cm^2$	C_{tot} $\mu F/cm^2$
1100	4.35	2.19	4.34	5.56	5.37	5.24	3.82	11.17
2024-T3	4.11	8.81	3.92	61.0	3.72	38.0	4.00	41.4
6061-T6	7.02	1.26	4.46	2.12	5.23	1.86	4.25	9.71
7075-T6	4.51	2.03	3.99	23.1	4.66	4.87	3.26	1.12

† Heat treated in air for 3 hours at 70° C.

Table 3. Salt spray test results for chromate conversion coated and talc coated 6061-T6 after 3 hours' exposure to elevated temperatures.

Temperature (°C)	Chromate Conversion	Talc Conversion
25	pass†	pass
70	pass	pass
115	**fail**	pass
160	**fail**	pass
205	pass	pass
250	pass	pass
300	**fail**	pass

† pass indicates that more than 75% of tested panels pass 168h of salt spray exposure.

Table 4. Summary of salt spray test results.

Alloy	Cu	Mg	Zn	Si	Coating Time (minutes)	Salt Spray Result	Maximum Time Tested (hours)
5052	--	2.5	...	...	<5	pass	168
1100	**0.12**	...	...	...	5	pass	336
6061-T6	--	1.0	...	0.6	10	pass	336
7075-T6	**1.6**	2.5	5.6	1.6	300	pass	168
2024-T3	**4.4**	1.5	...	...	900	fail	168

Table 5. DC Resistivity Measurements Summary for 6061-T6.

Test Method	Bare	Talc Conversion	Chromate Conversion	MIL-C-5541E requirement
As-coated				
Cu-platen (mΩ/in^2)	0.49±0.3	3.81±1.9	0.228±0.07	< 5
4 pt. probe (mΩ)	0.360±0.04	0.370±0.04	0.360±0.04	
Hg Probe (Ω)	4.7±0.4	(8.41±6.2) x 10^9	17.2±13	
After 168 h salt spray				
Cu-platen (mΩ/in^2)	...	0.53±0.3	2.07±1.5	< 10
4 pt. probe (mΩ)	...	0.120±0.03	0.110±0.01	
Hg Probe (Ω)	...	3.0 x 10^{10}	3.3	

Table 6. Paint adhesion results summary for Zn-chromate epoxy, chromate conversion and talc conversion coatings used as primers for a high solid white urethane topcoat.

Test Method	Zn-chromate (MIL-P-23377)	Talc Conversion	Chromate Conversion
Method 6301	pass	fail	fail
D3359 (dry)	5B (0%)	5B (0%)	5B (0%)
D3359 (500 h salt spray)	5B (0%)	5B (0%)	1B (35-65%)
paint creepback (500 h salt spray)	none	< 0.3 mm	not tested

Table 7. Paint adhesion results for Zn-chromate epoxy primer and talc conversion coating used as a primer for a polyurethane enamel topcoat.

Test Method	Zn-chromate (MIL-P-23377)	Talc Conversion
Method 6301	pass	pass
D3359 (dry)	...	...
D3359 (500 h salt spray)	5B (0%)	5B (0%)
paint creepback (500 h salt spray)	none	none

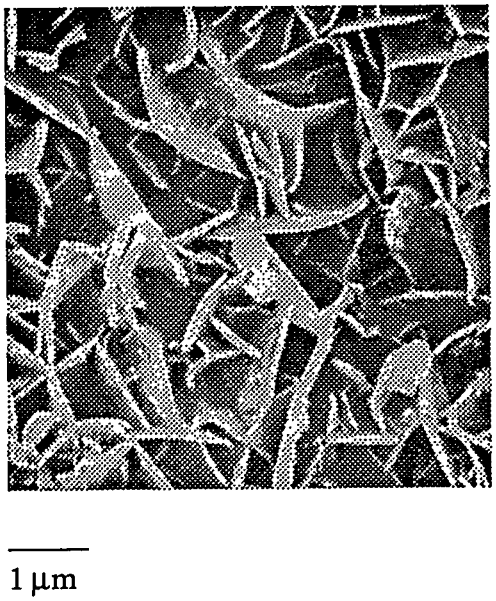

Figure 1. Plan view SEM of talc conversion coated surface.

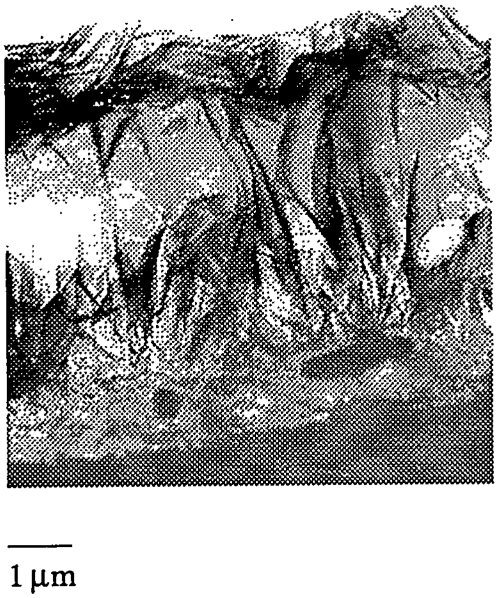

Figure 2. Section TEM of the talc conversion coating.

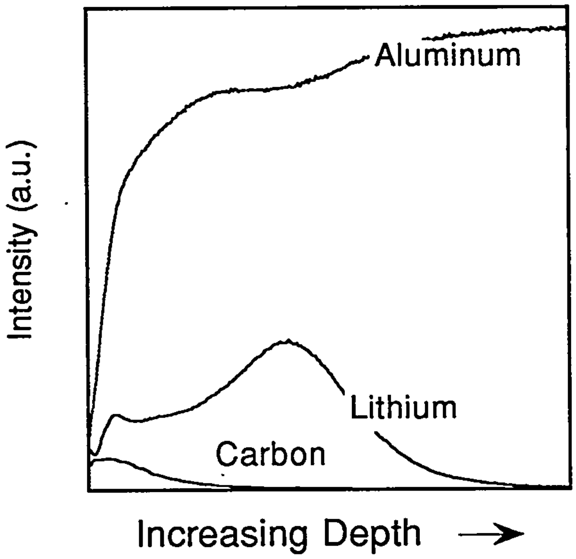

Figure 3. Sputter depth profile of talc coated 1100 Al.

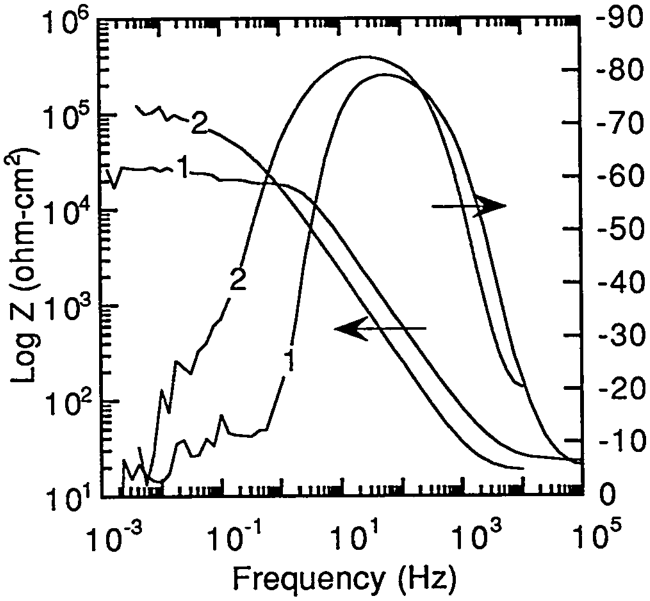

Figure 4. Representative Bode plots for talc conversion coated 1100 Al.

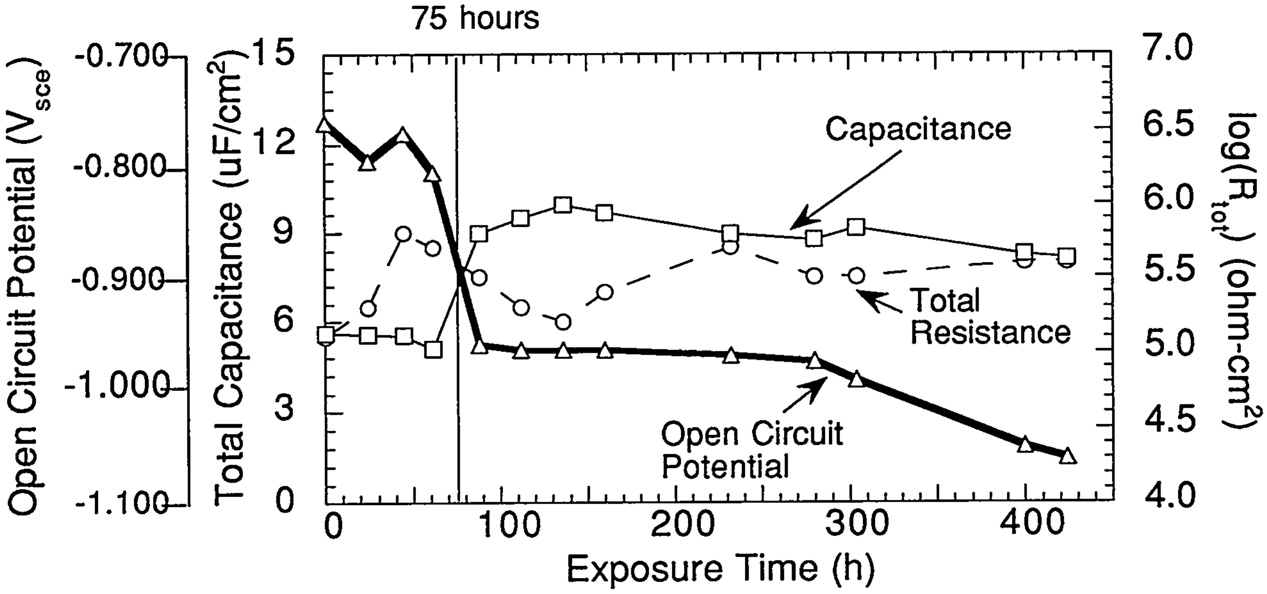

Figure 5. E_{oc}, R_t and C_t as a function of time for talc conversion coated 1100 exposed to air-sparged 0.5M NaCl solution.

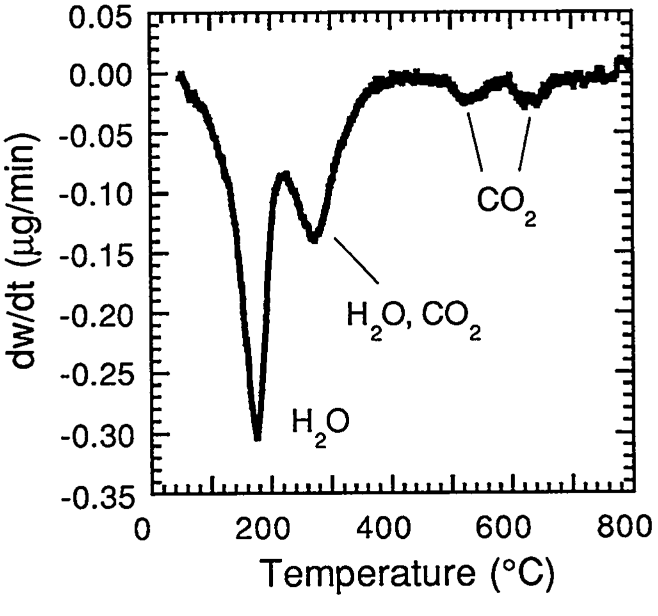

Figure 6. DSC thermogram for pure isolated hydrotalcite showing gaseous species evolved as a function of temperature.

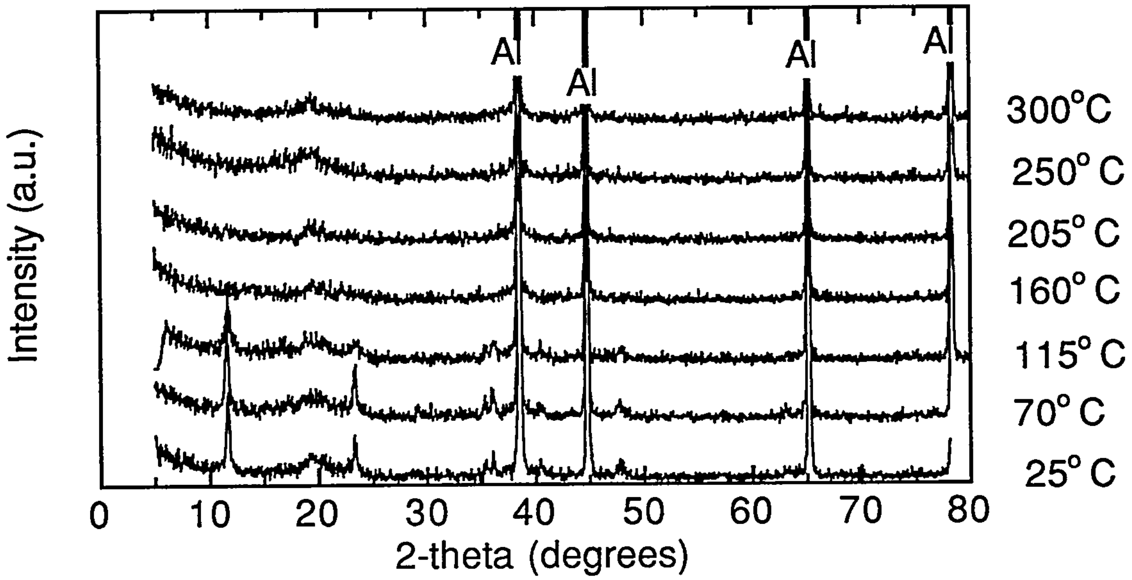

Figure 7. XRD data sets collected from talc coated surfaces subjected to 3 hour isothermal heat treatments at the indicated temperatures.

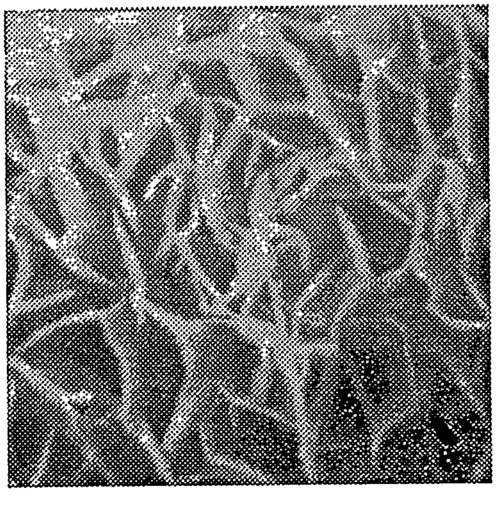

Figure 8. SEM of the talc coating after exposure at 25° C.

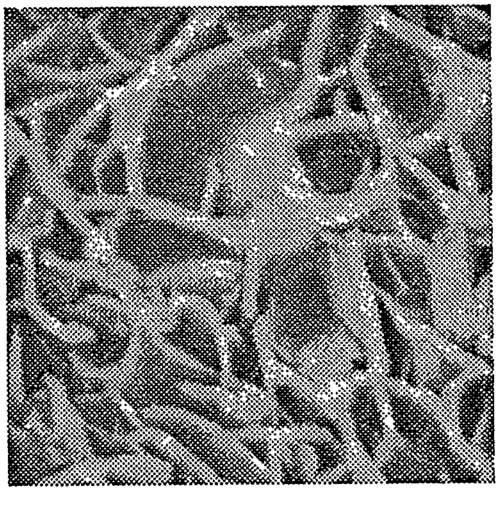

Figure 9.SEM of the talc coating after exposure at 300° C.

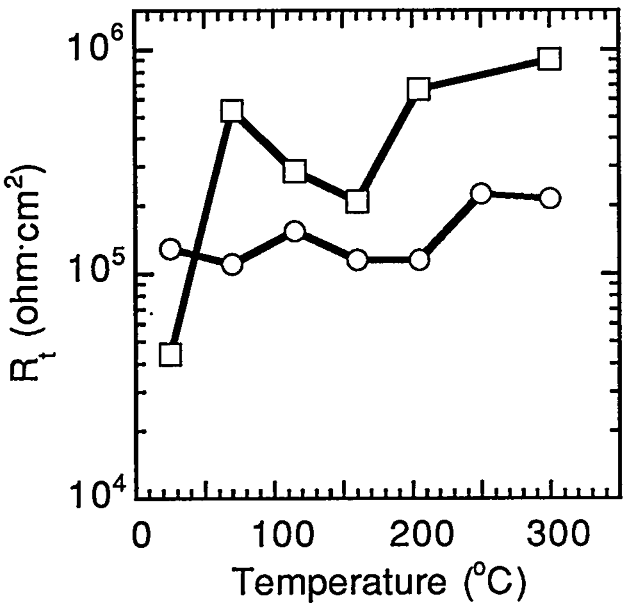

Figure 10. Total resistance R_t versus heat treatment temperature for talc coated (squares) and chromate conversion coated (circles) 6061-T6 determined in air-sparged 0.5 M NaCl solution.

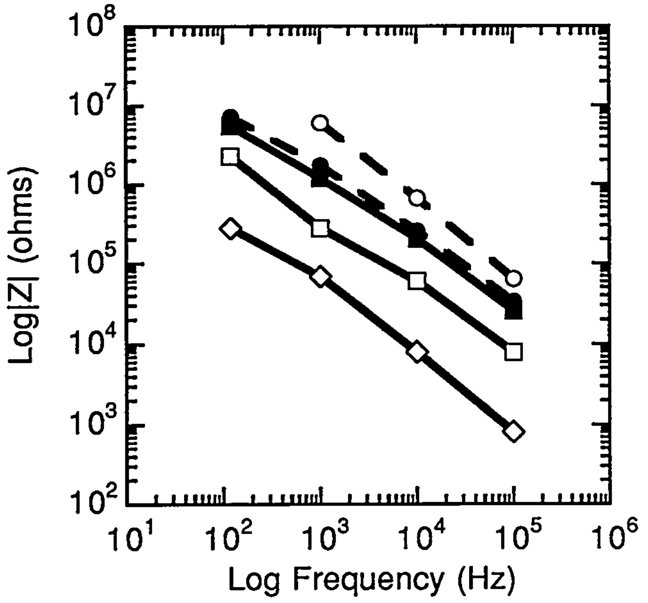

Figure 11. Total impedances determined using the Hg probe for talc coated (dashed line) and chromate conversion coated (solid lines) in the as-coated condition (open data) and after salt spray (closed data).

DISCLAIMER