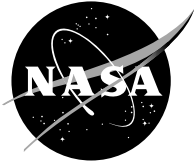


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# Conversion Coatings for Aluminum Alloys by Chemical Vapor Deposition Mechanisms

John T. Reye, Lisa S. McFadden, and Jorge E. Gatica  
Cleveland State University, Cleveland, Ohio

Wilfredo Morales  
Glenn Research Center, Cleveland, Ohio

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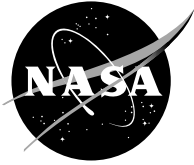
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Glenn Research Center

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This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

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# **Conversion Coatings for Aluminum Alloys by Chemical Vapor Deposition Mechanisms**

John T. Reye, Lisa S. McFadden, and Jorge E. Gatica  
Cleveland State University  
Cleveland, Ohio 44115-2425

Wilfredo Morales  
National Aeronautics and Space Administration  
Glenn Research Center  
Cleveland, Ohio 44135-3191

## **ABSTRACT**

With the rise of environmental awareness and the renewed importance of environmentally friendly processes, the United States Environmental Protection Agency has targeted surface pre-treatment processes based on chromates. Indeed, this process has been subject to regulations under the Clean Water Act as well as other environmental initiatives, and there is today a marked movement to phase the process out in the near future. Therefore, there is a clear need for new advances in coating technology that could provide practical options for replacing present industrial practices. Depending on the final application, such coatings might be required to be resistant to corrosion, act as chemically resistant coatings, or both. This research examined a chemical vapor deposition (CVD) mechanism to deposit uniform conversion coatings onto aluminum alloy substrates. Robust protocols based on solutions of aryl phosphate ester and multi-oxide conversion coating (submicron) films were successfully grown onto the aluminum alloy samples. These films were characterized by X-ray Photoelectron Spectroscopy (XPS). Preliminary results indicate the potential of this technology to replace aqueous-based chromate processes.

## **INTRODUCTION**

Chromate-based coating processes have been used by industry for many years. The aerospace, automotive and the construction industry, among others, have benefited from this process as a means to generate protective coatings on metal surfaces to ensure the success of subsequent applications. These processes, however, are subject to regulation under the Clean Water Act and other environmental initiatives because these processes not only generate large amounts of hazardous waste water but also pose a danger to worker health and safety. New approaches in coating technology, therefore, need to be investigated to provide alternative practical options to chromate-based coatings in order to meet EPA mandates [1]. In order to be a feasible replacement alternative any non-chromate-based coating used must possess properties, such as corrosion resistance or adhesion enhancement, equivalent to those of coatings yielded by chromate-based technologies.

Chemical Vapor Deposition (CVD) is a common technique used for depositing uniform layers on solid substrates. This technique has been found useful in a wide spectrum of applications, such as insulation, synthesis of advanced materials, protective coatings, etc. Despite being a mature industrial process, CVD still involves a complex interrelation between chemical thermodynamics, kinetics and transport phenomena; thus, the formulation of CVD approaches for alternative applications require a multi-task approach to resolve problems associated with these three areas.

A CVD method employing non-chromate-based reagents is investigated in this study. The process was applied to the deposition of conversion coatings on aluminum alloy 3004 substrates. This method utilizes solutions of aryl phosphate esters and transition metal organic additives which, upon

vaporization, interact with aluminum samples to generate protective conversion coatings. The chemical reaction between this solution and the aluminum alloy surface is “catalyzed” by the presence of transition metal organic additives in the reaction environment and generates a phosphate/multioxide conversion coating. This method was an outgrowth of the high temperature, vapor phase lubrication work performed by Morales and Handschuh [2] who conducted a deposition study using an aryl phosphate ester. These authors basically dipped a clean aluminum alloy coupon into an aryl phosphate ester/ethanol solution, withdrew the coupon and allowed the ethanol to evaporate leaving a thin uniform phosphate film. They next inserted this coated coupon into a 300 °C heated oven for two minutes. They then repeated this procedure but this time dipping a new aluminum alloy coupon into an aryl phosphate ester/ethanol solution containing ferric (III) acetylacetonate. Surface analysis of the first aluminum coupon revealed only the presence of aluminum oxide, with some carbonaceous material, and no phosphorous. Surface analysis of the second coupon revealed the presence of oxygen, carbon, iron and phosphorous but very little aluminum, thus indicating the presence of a film thick enough to cover the strongly absorbing aluminum substrate. Their study showed the inability of a phosphate type film to form on an aluminum surface without the use of the iron-based additive.

The main objective of the present study is to validate the hypothesis that iron-based organic additives are essential in the production of quality conversion coatings on aluminum alloys. This study focuses on the completely different deposition technique to those used before: a chemical vapor deposition (CVD) process. The three major focus of this research are (i) understanding the chemical interaction between additives, solvents and phosphates, (ii) characterization of the deposited films, and (iii) the development of the deposition environment which would ensure reproducibility and control of the CVD process.

## EXPERIMENTAL

The CVD method utilized solutions of an aryl phosphate ester (commercially known as Durad 620B) and transition metal organic (organometallic) additives. In this study the effect of iron acetate is reported. The iron acetate is typically supplied as a highly hygroscopic powder and has limited solubility in the aryl phosphate ester. In the absence of published solubility data, one of the goals of this research was to assess the solubility of iron acetate in the phosphate ester by conducting a parametric study. Another goal was to test the chemical independence of the iron acetate dissolved in the aryl phosphate ester and thus ensure no chemical reaction between the two prior to deposition.

Different molar solutions (see table 1) were prepared by first dissolving a measured amount of iron acetate in histological grade methanol. This iron acetate/methanol solution was then mixed thoroughly with the required amount of aryl phosphate ester. The final stage in the preparation consisted of the elimination of the methanol by evaporation. To accelerate this process, and avoid heating, the evaporation was facilitated by continuous stirring under a light vacuum generated by an aspirator connected to a faucet for five to seven days. A significant color and viscosity change of the solutions was used as an indicator for determining when methanol evaporation was complete. All final solutions appeared homogenous and, depending on amount of iron acetate, their color varied from a light tan (solutions prepared with anhydrous iron acetate) to a deep reddish brown (solutions prepared using hydrated iron acetate). As the anhydrous iron additive solutions age they became reddish brown.

Infrared spectroscopy was used to analyze these solutions. A ThermoNicolet™ Magna 560 FTIR unit was used. Solutions were setup for analysis by being sandwiched between KBr windows.

CVD experiments were performed in a Thermolyne™ 21100 Tube Furnace using a Mullite tube 0.9-inch ID. A cast iron block ( $3.82 \times 0.86 \times 0.25$ -inch<sup>3</sup>) with a machined 0.8-ml cylindrical well was used as a solution and sample holder. The well was filled with a solution and an aluminum coupon was placed on the flat stage of the holder. Once ready, the sample holder was inserted half way into the furnace. Aluminum alloy 3004, 0.5-mm thick, was used in this study. The furnace was

then programmed for controlled heating from room temperature to 400 °C at 5 °C/min. In addition to the temperature controller used with the furnace, the sample holder was fitted with a copper-constantan thermocouple. All experiments were performed under a fume hood. Depositions inside the Mullite tube occurred in the presence of stagnant air. Exploratory studies revealed two things: (1) that the CVD process occurred within the range of 300 to 400 °C; therefore, an experiment was considered complete when the holder reached 400 °C, (2) successful conversion coatings were visibly yellow.

Early attempts to characterize the film thickness with a Scanning Electron Microscope (SEM) were unsuccessful. The films were too thin to be seen by SEM. The films were then analyzed using X-ray Photoelectron Spectroscopy (XPS) [3]. XPS spectra were acquired on a commercial spectrometer operated at 100-eV pass energy. Non-monochromatized, Al K-alpha X-rays were used. The areas of the peaks in the XPS spectra were calculated by subtracting a Shirley background, and the composition of the coupon surface was calculated from the areas by applying sensitivity factors provided by the instrument manufacturer.

## RESULTS AND DISCUSSION

The parametric solubility study revealed that only the 1:1 molar ratio solution failed to yield a homogeneous final solution and significant precipitates were observed. Those solutions with molar ratios from 1:2 to 1:1000 separated to some small degree over time and this was confirmed from observed precipitate evolving at the bottom of the solution vials.

One major concern, when formulating these solutions, was whether the iron acetate would chemically react with the aryl phosphate ester while in solution. Analysis of the FTIR spectra of the solutions, compared to the FTIR spectrum of the pure aryl phosphate ester, revealed no peak shifts, differences in peak locations, new peaks, or missing peaks in the spectra. Figure 1 is the FTIR composite spectra comparing a sample solution (1:10 ratio) to the pure phosphate. Differences in height between the two spectra may simply be due to slight differences in solution thickness sandwiched between KBr windows. It was concluded that no reaction occurred during the formulation of the solutions in table 1. As the malt colored solutions aged, they became reddish brown. It is believed the solution may be picking up water over time. Anhydrous iron acetate left open to the atmosphere also turns from malt to reddish brown. Both types of solution were used successfully in growing the thin phosphate films so the addition of water does not seem to be a hindrance.

Figure 2 is the XPS depth profile of a 3004 aluminum alloy coupon subjected to CVD using pure aryl phosphate ester. The atomic concentration of several elements, in percent, is plotted versus depth of the surface of the film into the substrate. A quantity of aluminum and oxygen, about 10% and 50% respectively, is revealed near the surface. In addition, quantities of carbon (25%), phosphorous (10%) and magnesium (5%) are detected. The aluminum concentration increases, in a parabolic manner, to nearly 80% beyond 60 nm whereas the oxygen concentration decreases to about 25%. The phosphorous concentration decreases rapidly to zero at 20 nm. Both the carbon and magnesium concentrations slowly decrease to less than 2% beyond 60 nm. Visual inspection of the aluminum coupon surface revealed no yellow coloring.

Figure 3a is the XPS depth profile of a 3004 aluminum alloy coupon subjected to CVD using the iron acetate/aryl phosphate ester solution (1:1000). It is dramatically different compared to the XPS profile of figure 2. The oxygen concentration varies linearly from 60% at the surface to just above 50% at 70 nm, whereas the aluminum concentration varies linearly from 0% at the surface to nearly 11% at 70 nm. The magnesium concentration varies in a slight parabolic manner from 9% at the surface to about 26% at 70 nm, whereas the phosphorous concentration varies from 18% at the surface to about 7% at 70 nm. The carbon concentration, although nearly 14% at the surface, drops sharply and then stays nearly constant at 4% throughout the 70 nm film. No iron was detected. Visual inspection of the aluminum coupon revealed a uniform yellow coating. Figure 3b is the XPS depth

profile for the aluminum metal and aluminum oxide species. Near the surface only aluminum oxide is detected. As one proceeds into the film, a small concentration of aluminum metal is detected. Both the aluminum metal and aluminum oxide concentrations increase as the film depth approaches 70 nm.

Figure 4a is the XPS depth profile of the 3004 aluminum alloy coupon subjected to CVD using the more concentrated iron acetate/aryl phosphate ester solution (1:100). It is very similar to figure 3a with the observation that the aluminum concentration is zero at the surface and only increases to about 5% at 70 nm. Figure 4b, the XPS depth profile for aluminum metal and aluminum oxide, reveals that only near 70 nm were the signals strong enough for detection. Again, no iron was detected and visual inspection of the coupon revealed a uniform yellow coating.

The XPS results show that without the iron additive, a phosphorous-containing conversion coating cannot be produced on the 3004 aluminum alloy surface. Except for the presence of some phosphate ester decomposition products (fig. 2), the film is mainly aluminum oxide. With the iron additive, however, a coating is formed (figs. 3a and 4a) thick enough to reduce the aluminum concentration to 0% at the surface. Two curious issues deserve attention. (1) The puzzling absence of iron in the 70 nm film suggests that either the iron simply acts as catalyst at the surface and is not incorporated into the film or that the iron lies beyond the 70 nm film. (2) The substantial amount of magnesium in the film (3004 aluminum alloy typically contains no more than 1% Mg) and its slight parabolic profile indicates possible diffusion of magnesium from the bulk aluminum alloy. Future work will focus on these issues.

The effectiveness of the CVD deposition technique used in this study can be demonstrated by a simple comparison with preliminary deposition experiments. For instance, figure 5a presents the results obtained by first wetting a clean untreated aluminum coupon with the phosphate/iron additive solution, and then inserting the wetted coupon into a furnace. Visual inspection revealed that the resulting coating was a highly non-uniform pyrolytic deposition. Replicate experiments confirmed that a uniform coating could not be induced by this method. On the other hand, coatings deposited using the CVD method were consistently uniform. Figures 5b and 5c show two coupons that were coated using the CVD method. Spectroscopic (XPS) analyses of the film structure indicate that the film characteristics are reproducible for the precursor-substrate system used.

## CONCLUSIONS

A CVD method was successfully developed to produce conversion coatings on aluminum alloys surfaces with reproducible results. A well defined protocol to prepare the precursor solutions and deposition process was formulated. It was demonstrated that solutions prepared following such a protocol could be used to systematically generate protective coatings onto aluminum surfaces. Experiments with a variety of formulations revealed that a refined deposition protocol could yield not only reproducible but also conversion coatings of controlled composition. The significance of this work was the practical demonstration that chemical vapor deposition (CVD) techniques can be used to systematically generate protective/conversion coating on aluminum alloy surfaces. Preliminary surface analyses clearly demonstrated that the aryl phosphate ester, without the iron acetate additive, could not induce the deposition onto aluminum alloy 3004 with a significant degree of adhesion. In order to become an effective approach to replace chromate-based pre-treatment processes, namely in the aerospace or automobile industry, the process parameters must be defined more precisely. Moreover, the feasibility of scale-up designs necessitates a more comprehensive characterization of the fluid flow, transport phenomena, and chemical kinetics interacting in the process. Future work will concentrate on refining the process through computer simulations and further experimental studies on the effect of other transition metals to induce deposition of conversion/protective films on not only aluminum alloys but other metallic substrates.



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TABLE 1.—FORMULATIONS OF SOLUTIONS IRON ADDITIVES-ESTER PHOSPHATES TESTED

Solution	Mole% aryl phosphate, percent	Mole% iron additive, anhydrous, percent	Iron additive, anhydrous, grams	Solvent, grams	Aryl phosphate, grams
1:10000	99.99	0.01	0.00115	32.7	47.4
1:1000	99.9	.1	.0115	32.7	47.4
1:100	99.0	1.0	.115	32.7	47.4
1:20	95.2	4.8	.575	32.7	47.4
1:10	90.9	9.1	1.150	32.7	47.4
1:5	83.3	16.7	2.300	32.7	47.4
1:4	80.0	20.0	2.875	32.7	47.4
1:3	75.0	25.0	3.833	32.7	47.4
1:2	66.7	33.3	5.750	32.7	47.4
1:1	50.0	50.0	11.500	32.7	47.4

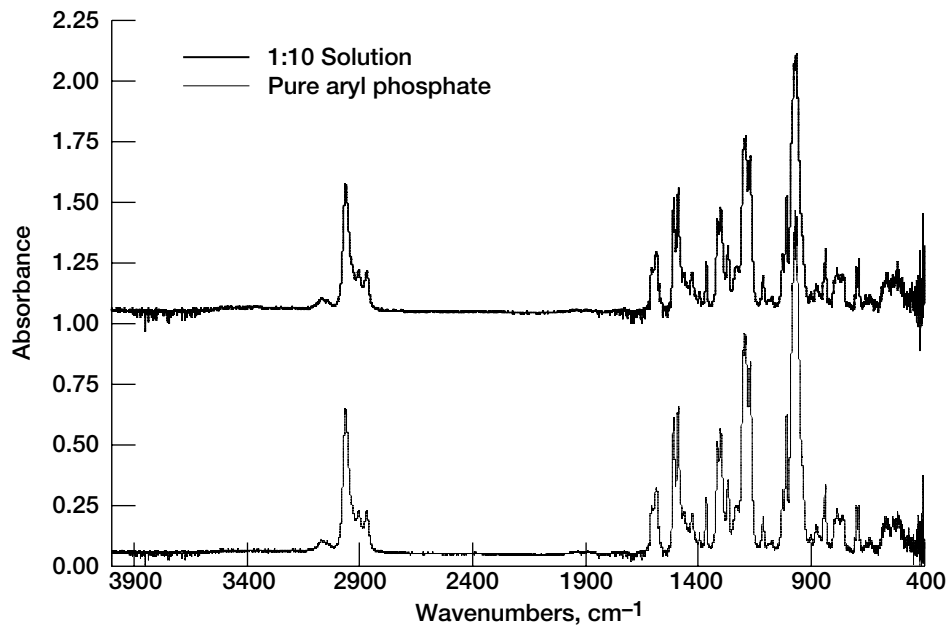


Figure 1.—FTIR of pure aryl phosphate and solution of aryl phosphate with additive (the bottom line corresponds to pure aryl phosphate).

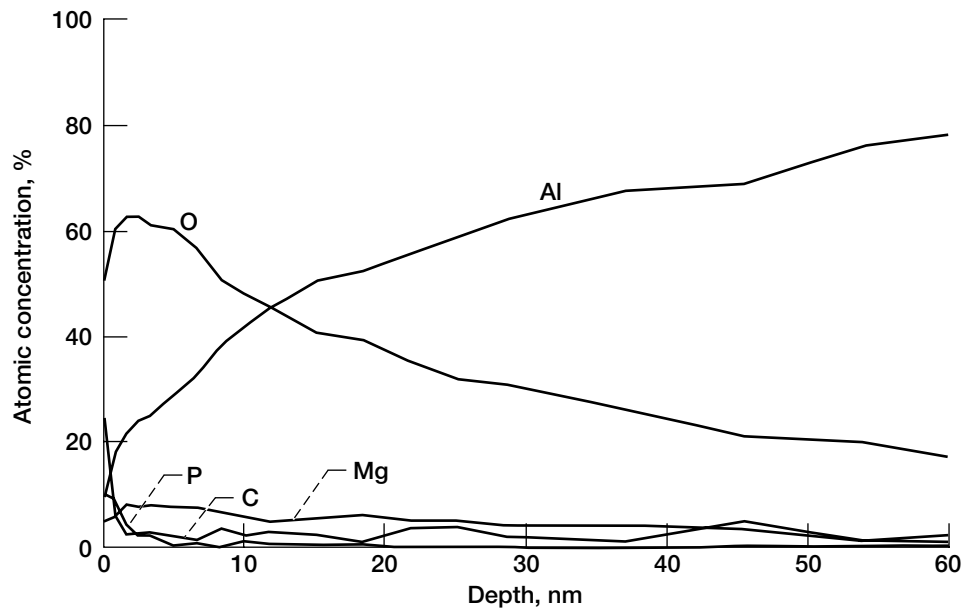


Figure 2.—XPS depth profile, pure phosphate ester on aluminum alloy 3004.

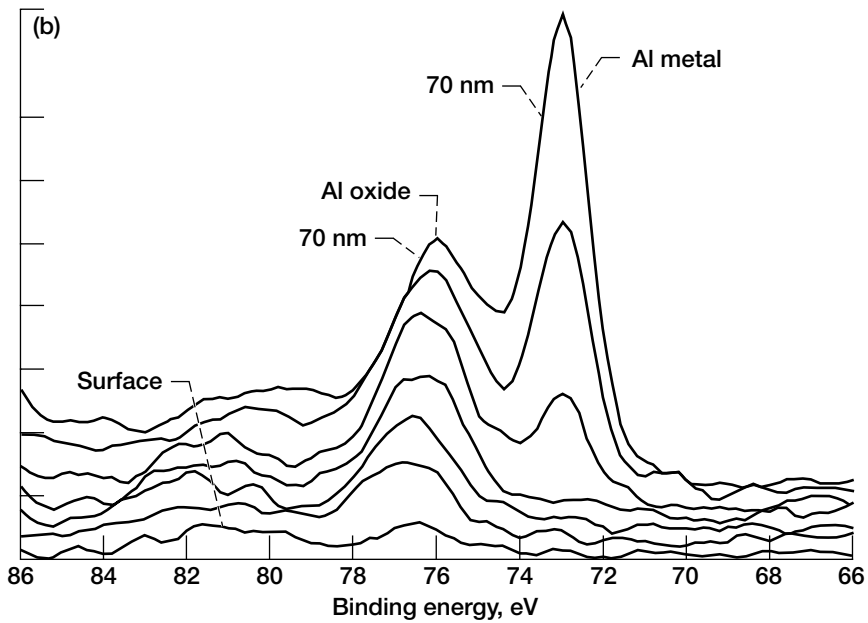
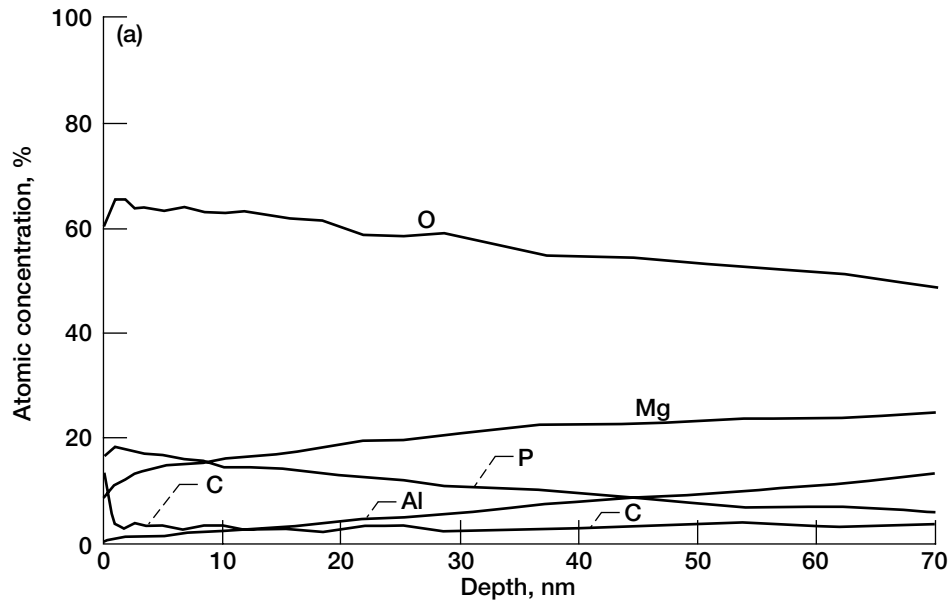


Figure 3.—XPS depth profile. (a) 1:1000 iron acetate/phosphate ester solution on aluminum alloy 3004. (b) Aluminum oxide and aluminum metal.

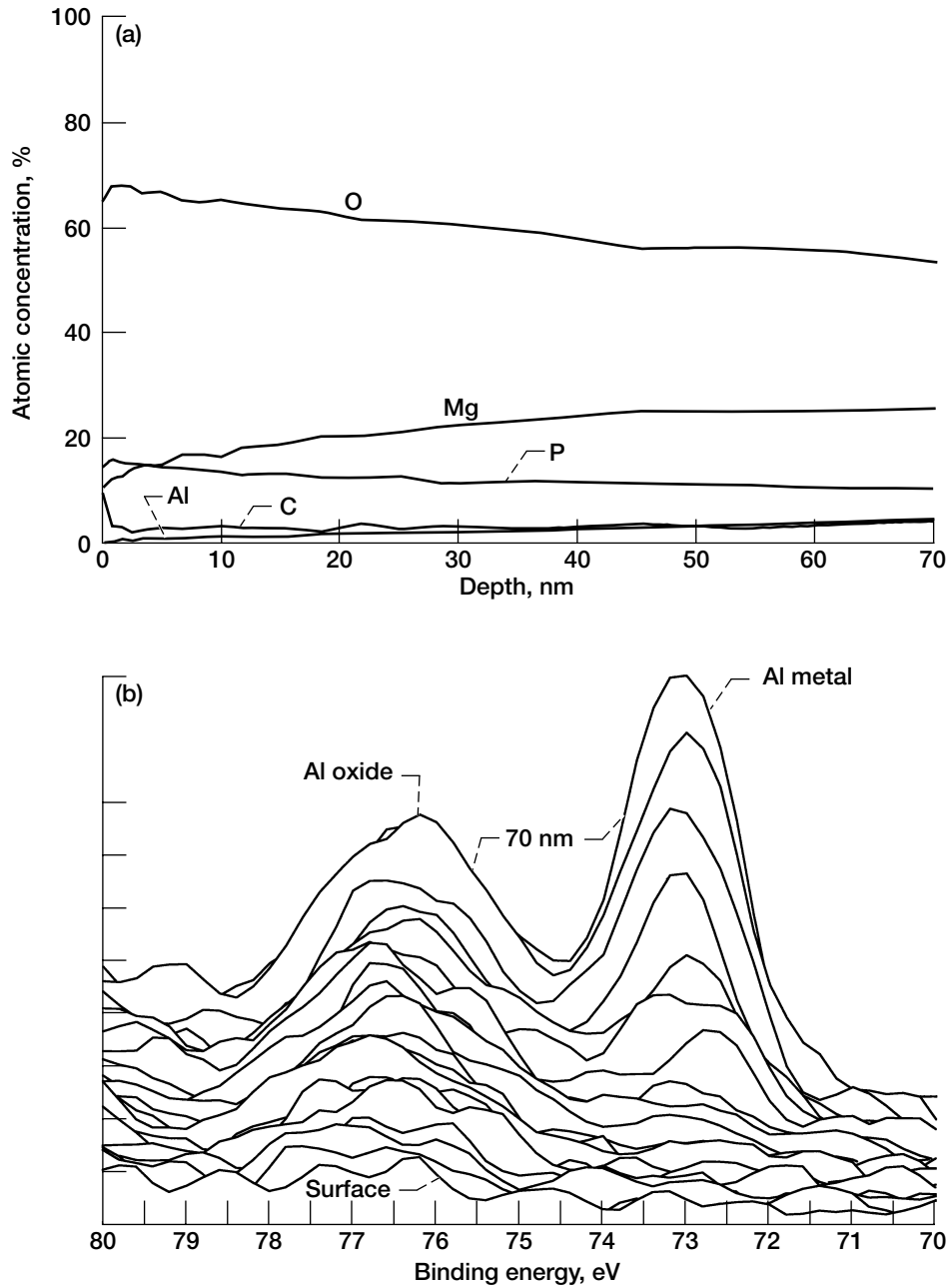


Figure 4.—XPS depth profile. (a) 1:100 iron acetate/phosphate ester solution on aluminum alloy 3004. (b) Aluminum oxide and aluminum metal.

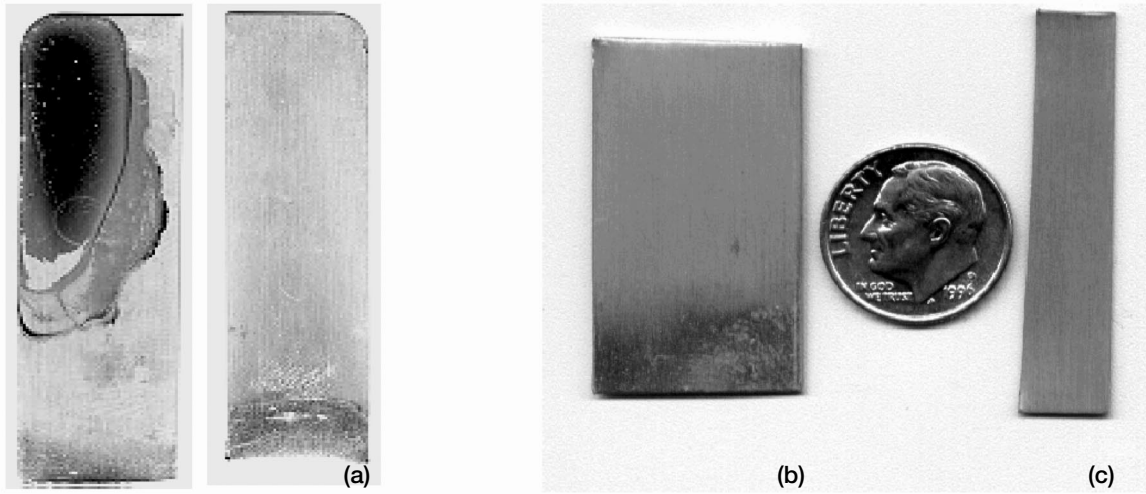


Figure 5.—Film deposits. (a) Solution film deposited. (b) Film deposited in open furnace. (c) Film deposited in capped furnace.

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