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Laboratory Experiment

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Anodizing and Coloring Aluminum Alloys

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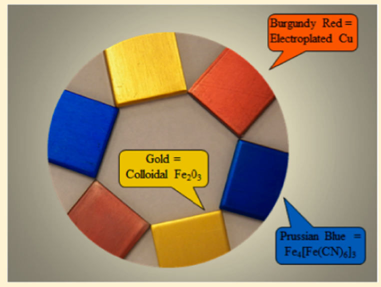
Supporting Information

ABSTRACT: In this experiment, wrought aluminum alloys, fashioned into 3 in. × 1 in. coupons, were anodized and subsequently colored. To achieve the most attractive colors the 1100 Al alloy was selected over other wrought Al alloys. To further ensure attractive colors, an eight-step pretreatment protocol was followed. Sulfuric acid was used as the electrolyte solution in the anodizing bath, and a five-step post-treatment protocol was followed prior to coloring the anodized Al coupon. Inorganic pigments (yielding gold and Prussian blue) and electrolytic coloring (yielding burgundy red) were utilized to achieve the colored Al alloys. Using oxalic acid in place of sulfuric acid as the electrolyte solution in the anodizing bath and anodizing other wrought Al alloys, namely, the 2024, 3003, 5052, 6063, and 7075 wrought Al alloys, was also explored. This experiment was performed during two successive 3 h laboratory periods in a second-semester general chemistry course designed for undergraduate engineering students.



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Anodizing is an important industrial process that has been practiced for nearly a century. The list of anodized aluminum products is long and diverse: e.g., outdoor architectural components, appliances, furniture, sports equipment, consumer goods, and airplane, boat, and automobile components.[1] The primary motivation for anodizing aluminum alloys is to create a more corrosion resistant surface than is provided by aluminum's natural oxide coating. Other benefits derived from anodizing aluminum are an attractive finish, the opportunity to color the anodized piece, a more wear-resistant surface, creation of an insulating surface, and creation of a superior surface for adhesion. Anodizing is an electrochemical process performed in an acidic solution in which the metal piece to be anodized is made the anode. When the electrolyte solution contains a polyprotic acid, the anodizing process converts the Al metal surface to a porous aluminum oxide layer with a unique (unusual) structure that permits organic dyes, inorganic pigments, or metals to be deposited in its pores.[2]

Several anodizing experiments and demonstrations have been previously described in this *Journal*.[3−6] In all these instances sulfuric acid was used as the electrolyte and organic dyes were used to color the anodized Al piece.

Aluminum is the most common metal to be anodized in an industrial setting. However, other early transition metals can also be anodized. Gaul has described the use of anodizing by artists to achieve a rainbow of colors by varying the thickness of the oxide coating on the surface of titanium, niobium, and tantalum.[7] Other approaches are available to apply an aluminum oxide coating. Vohs and co-workers recently described in this *Journal* the chemical vapor deposition of aluminum oxide thin films on a silicon wafer.[8]

This laboratory experiment was designed for engineering students enrolled in a two-semester general chemistry course that employs the theme of *Chemistry and the Automobile*[9−15] and has been performed many times during the second semester when the topic of electrochemistry is covered in lecture. An estimated 480 students have completed this laboratory experiment: approximately 60 students per year over eight years.

■ ALUMINUM IN CONTEXT

In recent years, the philosophy and goal of sustainable development has garnered increasingly greater support in the private sector as well as the education community. As regards the teaching of chemistry this is evident from an examination of Chapter 0 in the latest edition (7th edition) of *Chemistry in Context*.[16] The rapid rise of the discipline of industrial ecology—the study of material and energy flows through industrial systems—and the information provided by material flow analysis (MFA) is also extremely helpful in achieving these goals. Several examples of this type of analysis pertaining to aluminum have recently been published.[17,18] One study presents the global flows of aluminum from liquid metal through several intermediate steps to four major end-use categories—vehicles, industrial equipment, construction, and metal products—each of which are further subdivided and yield a grand total of a dozen subcategories.[18] The largest subcategory of the dozen is cars, accounting for 17.6% of the global consumption of end-use Al. The recycling of industrial scrap created during manufacturing steps (e.g., forming and fabrication steps) and end-of-life scrap is also included in this diagram. This map of material flows is presented in a Sankey

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diagram format and shows that roughly half of global end-use aluminum is made from bauxite and half from scrap.[18]

The laboratory experiment described therein is part of the package of materials used in the aforementioned general chemistry course to deliver to the students a broad overview of aluminum production and use in modern society. This is deemed appropriate given the audience (engineering students) and since aluminum is the second most used metal in the 21st century after iron/steel. Moreover between 1900 and 2000 the rate of consumption of aluminum increased more dramatically than that of any other metal: a 30-fold increase from 1950 to the present.[18] By comparison there has been a 4-fold increase in steel consumption since 1960.[19]

Primary aluminum production is covered in lecture and touches on the following themes (with more detail provided in Appendix 1 of the Supporting Information):

- The history of primary aluminum production
- Bauxite formation, composition, extraction, and shipment to locales with cheap electricity
- The Bayer process, the amphoteric behavior of Al, hydrated alumina
- Red mud: a significant environmental challenge[20]
- Conversion of hydrated alumina to anhydrous alumina
- Construction and operation of the Hall−Héroult cell and the importance of cryolite in lowering the operating temperature of the cell
- Challenges connected with the operation of the Hall−Héroult cell:
    - A very energy intensive process that consumes 3.5% of global electricity[18]
    - Generation of fluorocarbons (CF_4 and C_2F_6) which are potent greenhouse gases (GHG)
    - CO and CO_2 emissions
    - HF emissions
    - Periodic replacement of the anode (because it is consumed during electrolysis)

This laboratory experiment is used to augment the students' understanding of aluminum's properties and uses. It introduces students to the two categories of Al alloys—cast (high in alloying elements) and wrought (lower in alloying elements)—see Appendix 2 of the Supporting Information. It provides hands-on experience anodizing wrought Al alloys, including the pretreatment steps, the anodizing operation itself, post-treatment steps, and coloring the anodized Al alloy using inorganic pigments and electrolytic coloring.

LEARNING OUTCOMES AND ASSESSMENT

After completing this experiment, students were able to explain why wrought aluminum alloys are anodized and how they are anodized. They were also able to describe the unique structure of the anodized surface, how it lends itself to the introduction of colorants, the variety of colorants that can be used, and the techniques available for introducing pigments into the pores of the anodized surface. They demonstrated their comprehension of these tasks by (1) completing a 30-question prelab assignment, (2) preparing four anodized Al coupons, three of which contained pigments, and (3) providing explanations for why practitioners might fail to obtain high-quality anodized aluminum products containing colorants. The students' colored aluminum coupons were graded based on the quality of the final product. In addition, on one of the five course exams ~25% of that exam involved questions about aluminum from both lecture and this laboratory experiment. Finally, students were asked to answer an essay question that explored how aluminum use might be considered advantageous during the in-use phase of a product's life, but nonsustainable over its entire lifetime.

When the students were polled (in the form of a final course evaluation letter) about their laboratory experience for the semester, this laboratory experiment received the highest rating. For one recent semester (N = 58), 43% of the students identified this laboratory experiment as their favorite and did not mention any other experiment. Another 35% of the students identified this laboratory experiment along with one or two others as their favorites. The three other laboratory experiments identified as favorites—two of which have been previously described in this *Journal*—included the identification of the soap thickeners in grease,[13] thermal analysis of plastics,[14] and the preparation of two ceramic materials (an alumina crucible and the 1−2−3 superconductor). All four of these laboratory experiments involve applied chemistry which students characterized as "interesting", "relevant", and "real-world".

OVERVIEW OF THE ANODIZING EXPERIMENT

Although this laboratory experiment could be structured to emphasize the quantitative aspects of electrolysis, it is not. Rather, it is delivered as a qualitative experience. This laboratory experiment is preceded by a quantitative laboratory experiment involving galvanic cells and E_{cell} measurements and followed by a quantitative laboratory experiment involving the determination of Faraday's constant.

In this laboratory experiment students are introduced to the difference between cast and wrought Al alloys. A half dozen different wrought Al alloys have been used in this lab: specifically 1100, 2024, 3003, 5052, 6063, and 7075. Before the Al coupon (~3 in. × 1 in. × 1/8 in.) was anodized, its surface was thoroughly cleaned in an eight-step pretreatment process in order to remove dirt, oils, and the natural oxide coating. The Al coupon was anodized using a dc power source in a very simple setup. Sulfuric acid, as well as oxalic acid, has been used as the electrolyte solution for anodizing. The first Al coupon the students anodized was not colored. This was done to illustrate the change in appearance of the Al surface after anodizing. Subsequent Al coupons were anodized and colored by one of two approaches: the insertion of an inorganic pigment or the electrodeposition of a metal in the pores of the anodized surface.

This laboratory experiment can be tailored to meet the needs of the instructor/students. The students work in groups, perform some of the pretreatment steps in a hood, and anodize their Al coupon in a hood. Students can easily anodize and color two Al coupons in one 3-h lab period. This laboratory experiment has been performed as either a two-week experiment or three-week experiment. When it is run as a two-week experiment, four anodized Al coupons are produced per group: one is just anodized, a second colored gold by means of an inorganic pigment, a third colored Prussian blue by means of an inorganic pigment, and a fourth colored burgundy by means of electrodeposition of elemental copper. When run as a three-week experiment, the students select or design a special project centered on anodizing and coloring these Al alloys.

THE PRETREATMENT, ANODIZING, AND POSTTREATMENT PROTOCOLS

The process of anodizing aluminum was presented to the students as a five-step process: (1) selection of the Al alloy to be used and the definition of the operating conditions, (2) pretreatment of the Al alloy, (3) anodizing the Al coupon, (4) post-treatment of the Al alloy, and (5) seal or color and then seal the anodized Al coupon. The 1100 wrought Al alloy is the closest alloy to pure Al and therefore offers the smoothest path to success, if achieving attractive colors is the goal. However, the other aforementioned Al alloys have also been anodized. Their use can pose greater challenges (than the 1100 alloy) but sometimes yield interesting results. The Aluminum Anodizers Council (AAC) provides a guide for anodizing the different series of Al alloys.[21]

The pretreatment protocol is summarized below. This protocol was suggested by an industry practitioner.[22]

- Step 1. Mechanical Cleaning: Polish the Al bar with wet 1200 grit paper. Follow that with polishing using wet 2000 grit paper. Polish lengthwise only.
- Step 2. Detergent Wash: Wash the Al bar with Alconox detergent and then rinse.
- Step 3. Alkaline Cleaning: Place the Al bar in an aqueous NaOH bath held at 40 °C for 1 min. **This step is performed in the hood.**
- Step 4. Tap Water Rinse: Rinse the Al bar with tap water for 1 min.
- Step 5. Acid Cleaning: Place the Al bar in 96.5% H_3PO_4/3.5% HNO_3 at 85 °C for 1 min. **This step is performed in the hood.**
- Step 6. Tap Water Rinse: Rinse the Al bar with tap water for 1 min.
- Step 7. Distilled Water Rinse: Rinse the Al bar with distilled water for 1 min.
- Step 8. Drying: Place the Al bar on filter paper to air-dry.

The anodizing conditions employed with the 1100 Al alloy coupons were as follows: anodizing time, 30 min; anodizing temperature, 20 ± 2 °C; electrolyte solution. 15 wt % H_2SO_4; current density, 1.5 A/dm^2; and mode of operation, constant current. Satisfactory results are still obtained for shorter anodizing times (e.g., 15−20 min). If oxalic acid was used in place of sulfuric acid as the electrolyte solution, a 5 wt % solution was used. The anodizing setup is very simple and is pictured in Figure 1. The pretreated Al coupon was made the anode and was connected to the dc power supply. The cathode is also made of Al. Although other metals can be used as the cathode (e.g., Pb), contemporary industry practice is to use an Al alloy cathode and to size the cathode to achieve an ∼3:1 anode to cathode surface area ratio.[2,23]

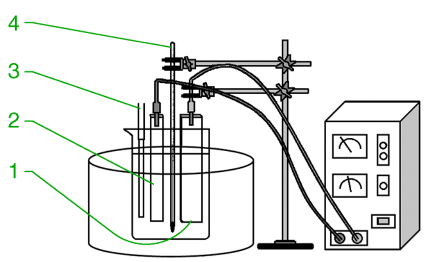

Figure 1. Anodizing setup. 1 = Al coupon to be anodized (anode). 2 = Al cathode. 3 = air from air jet to agitate the solution. 4 = thermometer.

After the Al coupon was anodized, it was subjected to a post-treatment protocol as outlined below.

- Step 1. Tap Water Rinse: Rinse the Al coupon with tap water for 1 min.
- Step 2. Acid Rinse: Swirl the Al coupon in a beaker of 25% nitric acid for 4 min at room temperature.
- Step 3. Tap Water Rinse: Rinse the Al coupon with tap water for 1 min.
- Step 4. Distilled Water Rinse: Rinse the Al bar with distilled water for 1 min.
- Step 5. Drying: Dry the Al coupon with filter paper and then with an air jet.

This protocol was recommended by an industry practitioner.[22] The acid rinse insures that the pores of the anodizing surface are open in order to optimize any coloring procedure that follows.[22]

If the anodized Al coupon was not going to be colored, it was sealed by immersing it in a beaker of boiling distilled water for 30 min. Sealing is considered essential to the long-term integrity of the anodized surface. Aqueous solutions containing nickel acetate and cobalt acetate are also used to seal anodized aluminum.[2,24] Chemically the sealing process is thought to involve the hydration of the anodized coating to yield boehmite or gamma aluminum oxide hydroxide (γ-AlOOH).[24] Physically, the formation of the boehmite results in a volume increase in the surface oxide which closes the pores and prevents impurities from entering the pores and colorants from exiting the pores.

COLORING WITH INORGANIC PIGMENTS

The composition of the baths used to color the anodized Al coupons with inorganic pigments is found in the Supporting Information along with the experimental details. The experimental procedures for these coloring baths are well-established, and many have been used in industrial settings.[25,26] The chemistry associated with the gold and Prussian blue colors is described below.

The gold color is achieved by the decomposition (hydrolysis) of ferric ammonium oxalate reaction:

$$2(NH_4)_3Fe(C_2O_4)_3(aq) + 3H_2O(l) \rightarrow Fe_2O_3(s) + 6NH_4^+(aq) + 6HC_2O_4^-(aq)$$

The formation of colloidal $Fe_2O_3(s)$ is responsible for the gold color. Unlike many inorganic pigment coloring systems, the gold color is achieved using a single bath. Because this process is more straightforward, this coloring procedure was normally performed during the first week of this two-week laboratory experience.

Coloring the anodized Al coupon to achieve Prussian blue requires immersion in two baths and is performed during the second week. The Prussian blue color is achieved according to reaction:

$$3K_4Fe(CN)_6(aq) + 4Fe(NO_3)_3(aq) \rightarrow Fe_4[Fe(CN)_6]_3(s) + 12KNO_3(aq)$$

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■ ELECTROLYTIC COLORING

The setup for electrolytic coloring is pictured in Figure 2. The anodized Al coupon is electrolytically colored using an ac power supply operated at constant voltage. The electroplated metal is deposited in the pores of the anodized surface, and this is accomplished by making the anodized Al coupon the cathode and the other metal (to be electroplated into the pores) the anode. The electrodes sit in an aqueous electrolyte solution which contains a salt (or salts) whose cation is identical to the oxidized form of the anode. To achieve a burgundy red color, copper was electroplated in the pores of the anodized Al surface. This was achieved by using an ac power supply that delivers 15 V for between 5 and 15 min. The anodized 1100 Al alloy was made the anode, and the cathode consisted of a copper strip. These electrodes were immersed in an aqueous solution containing $CuSO_4$ and H_2SO_4. This experimental procedure was obtained from the literature.[27]

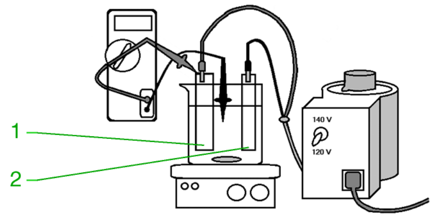

Figure 2. Electrolytic coloring cell, Fluke meter, and power supply. 1 = anodized Al coupon (cathode). 2 = copper strip (anode).

■ HAZARDS

Nitric acid, phosphoric acid, and sulfuric acid are corrosive and cause burns on contact with skin and eyes. Nitric acid and sulfuric acid are also strong oxidizers. Oxalic acid is toxic and may be fatal if swallowed. Sodium hydroxide causes burns to skin and eyes. Ferric ammonium oxalate is a respiratory and skin irritant. Ferric nitrate causes irritation to skin, eyes, and respiratory tract and is a strong oxidizer. Potassium ferrocyanide is harmful if swallowed. Copper sulfate is a respiratory irritant. Extreme caution should be used when operating an ac electrolytic cell designed for the electrodeposition of Cu into the pores of the anodized aluminum surface: see Figure 2. Electrocution is possible.

■ RESULTS AND DISCUSSION

Figure 3 compares the appearance of the 1100 Al alloy before and after anodizing. Figures 4–6 provide student-prepared examples of colored 1100 Al alloys. Each group was typically assigned a different coloring time. Figure 4 shows the coupons colored gold (using ferric ammonium oxalate) for between 3 and 6 min. Figure 5 shows the progression in intensity of Prussian blue colors between 7 and 12 min achieved by a two-bath system that deposits $Fe_4[Fe(CN)_6]_3(s)$ in the pores. Figure 6 shows the range of burgundy red colors achieved between 8 and 12 min by electrolytic deposition of Cu(s) into the pores of the anodized surface.

Figure 3. Untreated 1100 Al coupon (left) and anodized 1100 Al coupon (right).

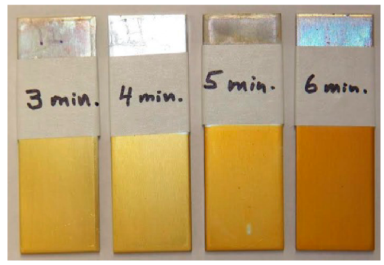

Figure 4. Anodized 1100 Al coupons colored gold after treatment for 3–6 min with $(NH_4)_3Fe(C_2O_4)_3(aq)$.

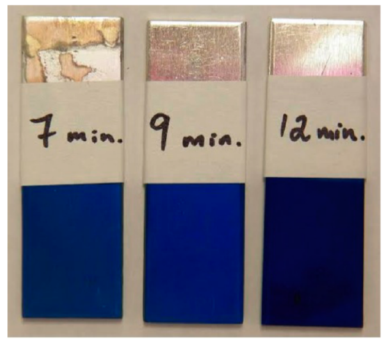

Figure 5. Anodized 1100 Al coupons colored Prussian blue after treatment for 7, 9, and 12 min with $Fe(NO_3)_3(aq)$ followed by $K_4Fe(CN)_6(aq)$.

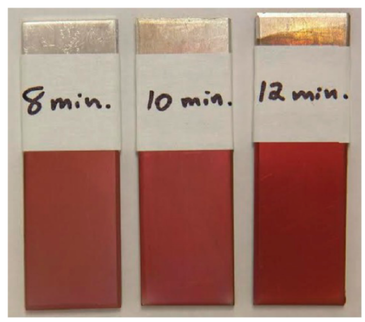

Figure 6. Anodized 1100 Al coupons colored burgundy by electrodeposition of Cu(s) in the anodized pores for 8, 10, and 12 min.

■ SPECIAL PROJECTS

When this laboratory experiment has been taught as a three-week experiment, the students were asked to select their own project for the third week. They were expected to anodize two or three additional Al coupons and to perform an experiment or experiments they had not done during the first 2 weeks. They were supplied with a list of additional experiments to choose from (see Supporting Information) or were invited to come up with their own experiment. A few examples of these are special projects are mentioned below:

- Switch to a different Al alloy other than 1100

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- Switch the anodizing electrolyte solution from 15 wt % sulfuric acid to 5 wt % oxalic acid
- Select a different inorganic pigment to color the 1100 Al alloy
- Select a different system to color the 1100 Al alloy by electrolytic coloring
- Examine the effect of alloying elements in an Al coupon as it goes through the pretreatment protocol

Photos of some of the results students obtained upon completing these special projects are provided in the Instructor's Notes in the Supporting Information.

CONCLUSIONS

This laboratory experiment introduces students to the surface treatment of an important construction material (i.e., wrought aluminum alloys) and to an example of applied electrochemistry (anodizing) that is used, among other reasons, to minimize the corrosion of aluminum products. It simulates, to a high degree, the industrial practice of pretreating and post-treating a material before some process (in this case anodizing) is performed on it: a topic rarely addressed in academic laboratory experiments. The coloring of the anodized Al coupons by use of inorganic pigments and the electrodeposition of metals enriches the laboratory experience further. With the inclusion of electrolytic coloring in the lab experiment, the students are exposed to two different examples of electrolytic cells, where, in one, the object of interest is made the anode and, in the other, the same object of interest is made the cathode. Initially, during the first week of this experiment, students are intimidated by all the steps involved in achieving a colored anodized Al coupon. However, by returning to anodize more Al coupons during the second week, the students' confidence increases dramatically.

ASSOCIATED CONTENT

Supporting Information

Student laboratory procedure; instructor's notes (including photos of the setups); prelab questions (with answers); four appendices: (A1) Production and Uses of Aluminum, (A2) Aluminum Alloys, (A3) Anodizing Aluminum, and (A4) The Structure of the Anodized Coating. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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