Good Practice Report

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Leaded or unleaded? Homemade microscale tin electroplating

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Abstract: Social distancing measures due to the SARS-CoV-2 virus have profoundly challenged the educational experimental work. We have sought to remediate this issue by designing a series of low cost, low risk, quick, and qualitative electrochemistry and corrosion experiments to be performed in the student's homes at the microscale with a kit provided by the teacher. One such experience is the electroplating of Sn from an aqueous chloride solution using readily available soldering wires (e.g., Sn–Pb alloy, or Sn–Ag–Cu alloy). This process catches students' attention due to its simplicity and variety of possible applications that include corrosion protection, fabrication of electronic components, plating of cooking utensils, lithium batteries, etc.

Keywords: electrochemistry; home experimentation; plating; tin.

Introduction

The SARS-CoV-2 pandemic prompted us to design a series of homemade chemistry experiments. We performed 20–25 experiments in each of the Fall 2020 and Spring 2021 semesters in our Electrochemistry and Corrosion elective course for Chemical Engineering junior and senior students. Similar experiences were then replicated in the Summer 2021 period with three groups of an average of 20 high school and undergraduate teachers. Descriptions of the overall experience and of some experiments have been published (Aguilar-Charfen, Castro-Sayago, Turnbull-Agraz, & Ibanez, 2021; Cesin-AbouAtme, Lopez-Almeida, Molina-Labastida, & Ibanez, 2021).

One of such student-designed experiments was Sn electroplating using soldering wire as the Sn source that can be performed in less than 30 min (Herrera, Cervantes-Herrera, Gutiérrez-Vallejo, & Ibanez, 2021). The experimental procedure is rather simple and with adequate supervision it could be used with secondary-level students as well.

Electroplating is a technique for metal deposition in which typically a thin layer of a desired metal adheres to another metal or substrate that is intrinsically or has been made conductive (Kanani, 2004). Modern and versatile plating approaches involve the fabrication of nanostructured deposits and multilayer coatings (Kanthiah, Manimaran, & Pactulingam, 2020), the use of environmentally-sound baths and additives (Abd El Rehim, Abd El Meguid, & Abass, 2011), the application of pulses during plating for tailoring different properties (Leimbach, Tschaar, Schmidt, & Bund, 2020), the preparation of composites (Walsh, Wang, & Zhou, 2020), and the like. Examples of electrodeposition basics and applications discussed in the educational arena include

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metal deposition on screen-printed electrodes (Chyan & Chyan, 2008), nanowire fabrication (Bentley et al., 2005), hexacyanoferrate deposition for detection purposes (Garcia-Jareno, Benito, Navarro-Laboulais, & Vicente, 1998), Zn electrodeposition (U. C. Davis, 2021), Cu electrodeposition (Thompson, 2016), electrodeposition objectives (Chemistry Libre, 2020), and the like.

Sn plating is used for multiple purposes including the fabrication of various alloys (Surface Treatment, 2017), Li battery cycling (Li et al., 2018; Zhang, Fan, & Chunsheng, 2017), corrosion protection (Bao, Zhou, & Li, 2020), fabrication of electronic components (2021), plating of cooking utensils (Walsh & Low, 2016), etcetera. A comprehensive review covers many of such applications (Walsh & Low, 2016).

Due to its standard potential, Sn deposition is a non-spontaneous process and therefore requires a source of direct current that flows through the metal by means of the valence electrons in Sn's outer shell (Poyner, 2005).

A readily available source of Sn involves soldering wire alloys. The most typical composition used is 60% Sn – 40% Pb. When a positive potential is applied in an electrochemical cell to one of these wires, two possible oxidation reactions can occur: that of Pb or that of Sn. Fortunately for the present purpose, Sn^0 is more easily oxidized in an acidic medium than Pb^0 due to their standard potentials given below (Lide, 2004–2005).

In addition, Pb probably forms insoluble species upon oxidation at potentials above 1.5 V versus the Normal Hydrogen Electrode (NHE) that either remain at the anode or precipitate (Pourbaix, 1974).

If preferred, a lead-free alternative consists of a commercially available Sn–Cu–Ag alloy (e.g., 99% Sn – 0.7% Cu – 0.3% Ag) (2021) which avoids the handling of Pb.

As a result of being in an acid medium there will be H^+ ions in the electrolyte and, consequently, these will also competitively reduce at the cathode to produce hydrogen gas, H_2 :

The Sn-plated object is then mechanically polished as described below.

Experimental procedure

To prepare the surface of the metal to be coated (e.g., a copper coin), wet a paper napkin with a few drops of ethanol and vigorously rub with it the surface to be coated to remove any dirt and grease that might interfere with the deposition process. To prepare the anode, cut 3–5 cm of the Sn–Pb or Sn–Cu–Ag solder wire and clean it with the same procedure. Tin electrodeposition is set up by clipping the object to be coated with an alligator clip and doing the same with the Sn source to partially dip both in a reservoir that contains a suitable electrolyte, as shown in Figure 1. A useful electrolyte consists of adding 10 drops of 3 M HCl (prepared from commercially available muriatic acid, a 28% HCl solution) to 5 mL of water in a 10 mL beaker. HCl is used because of its high conductivity. A milder electrolyte (i.e., NaHSO_4) has been recently proposed (Suzuki & Inoue, 2021).

Once every cell component is in its place, the solder wire (anode) is connected through an alligator clip to the positive terminal of the electrical source (i.e., a 9 V battery or a current converter), and the conductive object to be coated is made the cathode of the circuit by connecting it with another alligator clip to the negative terminal. The metal from the alligator clips must not touch the electrolyte because, otherwise, the metal in the clips will interfere with the deposition process. The Sn^{n+} species that form at the anode diffuse towards the cathode where their reduction occurs, thereby depositing Sn^0 atoms onto the desired substrate. The electrodeposition of Sn on the metallic piece starts immediately. Allow the deposition to occur during 3–5 min to ensure a good coverage.

Once this time has elapsed, disconnect the battery and remove the coated metallic piece from the solution. Eliminate the excess acid by rinsing the plated object with water. Dry the coated piece with a napkin. Then, rub it vigorously with powdered NaHCO_3 as abrasive with the aid of another napkin to obtain a visually more attractive surface after polishing. A simple test can be performed if desired for low-adhesion electrodeposits by attempting to peel them off with a pressure-sensitive adhesive tape (Ajuria-Garza, 1972).

Figure 1: Sn electrodeposition on a Cu coin.

The easiness for bronze formation from such deposits is fascinating and has been presented in the educational arena (Kuntzleman, 2021; Suzuki & Inoue, 2021). For example, a copper coin becomes silvery when placed with tin metal in an aqueous NaOH solution and heated to boiling. If the resulting surface is heated with a Bunsen burner, its color turns to gold – signaling the formation of a Sn–Cu bronze alloy (Suzuki & Inoue, 2021).

Results and discussion

In the present experiment, a low pH and the application of a positive potential to an Sn alloy wire with a 9 V battery produces a transition involving corrosion of the Sn(s). A second transition occurs at the cathode and starts from the Sn^{n+} generated at the anode into the immunity domain where tin deposits on the cathode as $Sn^0(s)$. A Pourbaix diagram of Sn (also called potential-pH diagram) (Pourbaix, 1974) in a chloride medium is shown in Figure 2 (House & Kelsall, 1984). Soluble and ionic stable species in the acidic domain include $SnCl_2$, $SnOHCl$, $SnCl_6^{2-}$, and $Sn(OH)_2^{2+}$. From that diagram, it is clear that to deposit Sn^0 at the cathode from this Sn-chloride system the pH needs to be below 4, which is attained by HCl addition. By adding 10 drops of 3 M

Figure 2: Pourbaix diagram for Sn (adapted from House & Kelsall, 1984, with permission).

Figure 3: Copper coins after and before Sn plating.

HCl to 5 mL of H_2O , the pH obtained is in the vicinity of 0.6. Here, SnCl_2 and SnCl_6^{2-} are the stable Sn species produced near the anode that diffuse towards the cathode and get reduced there to Sn^0 . Nascent hydrogen gas may contribute to this reduction. These are depicted in a generalized form as Sn^{n+} in Figure 1.

If desired, the magnitude of the electrical current can be measured with a multimeter, and the amount of deposited Sn can be estimated from Faraday's law (Gamburg & Zangari, 2011).

Here, the theoretical mass of metal, m (in g) electroplated by a current flowing through the electrodes, i (in A) is proportional to the amount of electric charge passed, Q (in Coulombs):

$$m = [(\text{g/mol}_{\text{metal}}) \cdot \text{Coul}] / [(\text{mol e}^- / \text{mol}_{\text{metal}}) (\text{Coul/mol e}^-)] = M \cdot Q / (nF) \quad (5)$$

where M is the molar mass of the plated metal, n is the number of electrons transferred in the reaction per mol of metal deposited, and F is Faraday's constant (96,485 C/mol). If a multimeter were available, one could measure the current and multiply it by the duration of the experiment to obtain the total charge passed. From this and the other constant amounts, the mass of metal can be estimated (assuming a 100% Faradaic efficiency).

For example, for a 5 min experiment at $i = 100$ mA, one would expect for the $\text{Sn}(\text{II}) \rightarrow \text{Sn}(0)$ a deposition of:

$$m = [118.71 \text{ g/mol}_{\text{Sn}} (0.100 \text{ A} \cdot 300 \text{ s})] / [(2 \text{ mol e}^- / \text{mol}_{\text{Sn}}) (96,485 \text{ Coul/mol e}^-)]$$

$$m = 0.0185 \text{ g}$$

To ensure that Sn (and not Pb) was deposited in the case of the experiment with the Sn–Pb alloy, we run a series of scanning electron microscopy (SEM) analyses in different zones of the plated coin that showed the absence of Pb in the deposit (see Supplementary Information). Lastly, a Sn-plated coin is compared to a bare coin in Figure 3.

Hazards and disposal

Hydrochloric acid is highly corrosive and dangerous (Hydrochloric, 2007). It is poisonous in case of ingestion and provokes severe burns in the skin as well as ocular lesions. Wear eye protection and safety equipment at all times. Lead is a neurotoxin. Do not touch the Sn–Pb alloy with bare hands. Due to the thermodynamics discussed earlier, the amount of Pb in the resulting solution is expected to be rather small. Neutralize the acid residues for appropriate disposal according to local regulations.

Conclusions

Electrodeposition of Sn on a metallic surface can be performed at home at the microscale with readily accessible, low-cost materials. With the corresponding Pourbaix diagram, the redox transitions and equilibria involved in the experiment can be better understood.

Supplementary Information

A separate file containing the results from scanning electron microscopy (SEM) analyses of different zones of a plated coin is provided.

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