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Title: Localized Bathless Metal-Composite Plating via Electrostamping

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **No**

3. **Interview statements:** Considering the COVID-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? One of the 2 options below would work.

   ☒ Interviewees wear masks until videographer steps away (≥6 ft/2 m) and begins filming, then the interviewee removes the mask for line delivery only. When take is captured, the interviewee puts the mask back on. Statements can be filmed outside if weather permits.

   ☒ Interviewees self-record interview statements. JoVE can provide support for this option.

4. **Filming location:** Will the filming need to take place in multiple locations? **No**

Current Protocol Length

Number of Steps: 20
Number of Shots: 47

Introduction

1. Introductory Interview Statements

1.1. **Troy Townsend:** Electrostamping works by trapping composite particles in the electrolyte, forcing them to plate with the metal. With this new technique, high composite particle loadings are possible even on water-sensitive objects.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.2. **Juli Hancock:** Unlike typical electroplating, this technique does not require submerging the object in a liquid bath. Any conductive object regardless of its size or shape can be coated with functional materials.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.3. **Jason Shaw:** Fluorescent metal composite coatings can have far-reaching applications for dim-light environments including aircraft maintenance equipment location, road-sign illumination and tribological wear identification on mechanical machine parts.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. Author NOTE: This interview was conducted remotely, movie file: IMG_7160.mov

Introduction of Demonstrator on Camera

~~1.4. **Troy Townsend:** Demonstrating the procedure will be Juli Hancock and Carter Russell, researchers from my laboratory.~~

~~1.4.1. INTERVIEW: Author saying the above.~~

~~1.4.2. The named demonstrator(s) looks up from workbench or desk or microscope and acknowledges the camera.~~

Protocol

2. Preparing coating salts

2.1. To begin, weigh nickel sulfate, nickel chloride hexahydrate and boric acid as mentioned in the text manuscript and combine them in a vial together **[1]**. Grind this salt mixture thoroughly to a fine powder **[3]**. Make sure to use proper protective equipment, fume hoods, and a hazardous waste disposal system **[4].**

2.1.1. Talent weighing the chemicals. *Video editor show Table 1 as an the inset*

2.1.2. Talent grinding the salt mixture. Vid NOTE: Misslated 2.2.1 take 1

2.1.3. Talent using gloves, goggles, lab coat, fume hood during the experiment.

2.2. Next, weigh 1.8 grams of europium, dysprosium doped strontium aluminate, europium doped yttrium oxide, or europium doped barium magnesium aluminate **[1]** and grind it into a fine powder using a porcelain mortar and pestle for approximately 10 minutes **[2]**.

2.2.1. Talent weighing the composite powder Vid NOTE: 2.2.1 take 3

2.2.2. Talent grinding the composite powder. Vid NOTE: 2.2.2 take 2

2.3. Combine the ground composite powder with the salt mixture in a container for storage **[1]**. Weigh 0.188 grams of this mixture per square centimeter of the coating area **[2]** and add it to an open top container **[3]**.

2.3.1. Talent adding ground composite to salt mixture Vid NOTE: take 2

2.3.2. Talent weighing the mixture.

2.3.3. Talent adding the mixture into an open container.

2.4. Add 40 microliters of water per square centimeter of coating area to this mixture **[1]** and stir to dissolve the salts and form a thick paste **[2].** *Videographer: This step is important!*

2.4.1. Talent adding water in the open container.

2.4.2. Talent stirring the mixture to form a thick paste.

3. Preparing the electrodes

3.1. Using a scissor, cut the anode to a size and shape that matches the object to be plated **[1]**.

3.1.1. Talent cutting nickel anode using scissor. Author NOTE: focus on the anode cutting. The anode is then cleaned with NaOH or KOH followed by water, followed by HNO3. The cathode happens to look identical. The cathode, however, does need to be treated with NaOH or KOH followed by water, followed by HCl.

3.2. In order to remove organic material from the surface of the anode foil and the cathode, clean it with 10 molar potassium hydroxide or sodium hydroxide using a cotton swab or cloth **[1]**. Rinse the surface with water to remove the excess base **[2]**.

3.2.1. Talent cleaning the cathode and anode foil with base. Author NOTE: Both are cleaned the same way

3.2.2. Talent washing the surface with water.

3.3. Then, activate the metal surface to receive the coating by wiping it with a selective concentrated acid using a cotton swab or cloth, following the recommendations for activating specific metal surfaces and alloys. Perform this in the fume hood to avoid exposure to hydrogen chloride vapors **[1-TXT]**. *Videographer: This step is difficult!*

3.3.1. Talent cleaning the cathode foil with acid. **TEXT: 37% v/v HCl for nickel**

Commented [P1]: Only the cathode gets HCl. The anode must have HNO3.

3.4. Quickly deposit the prepared coating paste onto the cathode object, covering the entire area and making sure to avoid gaps **[1].** *Videographer: This step is difficult!*

3.4.1. Talent applying coating paste on the cathode.

3.5. Activate the anode surface by wiping it with concentrated acid using a cotton swab or cloth **[1-TXT]**. If calculation of current efficiency is required, record the mass of the anode and cathode using an analytical balance **[2]**. *Videographer: This step is difficult and important!*

3.5.1. Talent cleaning the anode with concentrated acid. **TEXT: 70% v/v HNO_3**

3.5.2. Talent weighing the anode and cathode.

4. Assembly and coating

4.1. Pre-set a power supply to the desired current or voltage mode **[1-TXT]**. Cut a piece of a hydrophilic membrane, such as a nylon sheet **[2-TXT]** and place it on top of the anode coating paste to avoid direct contact with the cathode **[3]**. Add a small amount of paste or dry salt mixture onto the sheet **[4]**. *Videographer: This step is important!*

4.1.1. Talent setting the current and voltage on the power supply. **TEXT: Constant current of 0.1 A per 4 cm^2 = 0.025 A/cm^2**

4.1.2. Talent cutting nylon sheet. **TEXT: Size larger than the anode**

4.1.3. Talent placing the sheet on the anode.

4.1.4. Talent applying dry salt mixture on the sheet.

4.2. Next, add two drops of water to partially dissolve the salt **[1]**. This process makes the nylon sheet conductive to allow the mass transport of ions through the electrolyte, which is necessary to balance charge in the coating reaction. This can also be accomplished by dipping the nylon membrane in an aqueous nickel salt mixture **[2]**.

4.2.1. Talent adding drops of water on the paste.

4.2.2. Talent making the nylon sheet conductive.

4.3. Place the activated anode on top **[1]** and attach both the negative and positive leads to the cathode object **[2]**. *Videographer: This step is important!*

4.3.1. Talent placing the anode on top.

4.3.2. Talent attaching the leads.

4.4. Cover the entire system with plastic to help retain water and apply moderate pressure **[1]**. Turn on the power supply **[2-TXT]** and continue coating for a desired duration **[3]**. *Videographer: This step is important!*

4.4.1. Talent covering the system with plastic

4.4.2. Talent turning on the power supply. **TEXT: 100 g per cm^2 area.**

4.4.3. Talent coating the anode and cathode.

4.5. Turn off the power supply **[1]** and expose the system **[2].** Disconnect the leads **[3]** and rinse the cathode object with water **[4]**.

4.5.1. Talent turning the power supply machine off.

4.5.2. Talent removing the plastic from the system.

4.5.3. Talent removing the leads.

4.5.4. Talent rinsing the cathode with water.

4.6. Wash the other components of the system by soaking them in water **[1]** and then discard this aqueous solution in the properly labeled hazardous waste container **[2]**.

4.6.1. Talent soaking the other items of the system in water.

4.6.2. Talent discarding the water.

4.7. To remove any uncoated composite particles, gently rub the cathode object by hand, wearing gloves **[1]**. Record the mass of the anode and cathode using an analytical balance **[2]** and calculate the difference with their original mass **[3]**.

4.7.1. Talent rubbing the cathode object with gloves.

4.7.2. Talent weighing the cathode and anode.

4.7.3. Talent doing the calculations of the mass.

Added Step:

4.8 Observe fluorescent coatings with an ultraviolet lamp to verify the brightness and consistency of the metal composite **[1-TXT]**.

4.8.1. Added shot: Observation showing the bright colors. **TEXT: Lamp Wavelength: 254 nm** Author NOTE: This is cool!

5. Characterization with electrochemistry

5.1. Use chronopotentiometry to monitor changes in voltage under constant current and chronoamperometry to monitor changes in current under constant voltage **[1].** Turn on the potentiostat and designate the duration and the applied current or voltage **[2]**.

5.1.1. Talent using the chronopotentiometer and chronoamperometer for reading. NOTE: Audio slated

5.1.2. Talent taking reading on the potentiometer. NOTE: no reading was taken, device was just turned on

5.2. Prepare the coating as previously described **[1]**. Normalize the voltage to a reference standard using a calibrated 3-electrode system **[2]**.

5.3. Talent preparing the coating. NOTE: This step was not visualizable. Authors put the Pt wire into the paste layer here.

5.4. Place a platinum wire as a pseudo reference electrode between the anode and the nylon sheet **[1]** and cover it with a separate nylon sheet to avoid contact **[2]**. Deposit a few drops of water and a small amount of coating paste on the anode **[3]**.

5.4.1. Talent placing platinum wire between the anode and nylon sheet. NOTE: Reorder the shots 5.3.2, 5.3.3, 5.3.1

5.4.2. Talent covering the electrode with another nylon sheet.

5.4.3. Talent putting water and dry powder on the anode.

5.5. Finally, connect the leads to the electrodes **[1]**, then seal, press, and begin coating as described in the text manuscript **[2]**. Monitor the changes in voltage or current **[3]**.

5.5.1. Talent connecting the electrode leads.

5.5.2. Talent assembling the apparatus.

5.5.3. Talent monitoring the voltage change and current.

Results

6. Morphological and electrochemical analysis of the metal-composite deposition

6.1. Fluorescent or colored particle incorporation can be observed due to a change in appearance compared to the uncoated surface **[1]**. Optical microscopy was used to investigate the surface coverage and to observe the surface morphology of the coating **[2]**. Samples were observed top-down **[3]** or cut to reveal the cross-section **[4]**.

6.1.1. LAB MEDIA: Figure 1. *Video editor emphasize A1-A3*

6.1.2. LAB MEDIA: Figure 1.

6.1.3. LAB MEDIA: Figure 1. *Video editor emphasize B1-B3 and C1-C3*

6.1.4. LAB MEIDA: Figure 1. *Video editor emphasize D1-D3*

6.2. The percent composite particle surface coverage as a function of time for chronoamperometry **[1]** and as a function of current density for chronopotentiometry increases during coating **[2]**. Surface coverage is also correlated with thickness **[3]**.

6.2.1. LAB MEDIA: Figure 2A. *Video editor emphasize on blue data*

6.2.2. LAB MEDIA: Figure 2A. *Video editor emphasize on red data*

6.2.3. LAB MEDIA: Figure 2B.

6.3. Coating parameters can be monitored under constant voltage using chronoamperometry **[1]** and under constant current using chronopotentiometry **[2]**.

6.3.1. LAB MEDIA: Figure 3A. *Video editor emphasize on blue graph*

6.3.2. LAB MEDIA: Figure 3A. *Video editor emphasize on red graph*

6.4. The brightness of the metal-composite coatings was quantified with fluorescence spectroscopy **[1]** and the calculation for luminescence quantum yield was done using the ratios of the peak areas **[2]**.

6.4.1. LAB MEDIA: Figure 3B.

6.4.2. LAB MEDIA: Figure 3B. *Video editor emphasize the equation.*

Conclusion

7. Conclusion Interview Statements

7.1. **Carter Russell:** When attempting this protocol, it is important to properly clean and activate the electrodes before coating. In addition, thoroughly grinding the precursor electrolyte mixture will help lead to a smooth, even coating.

7.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.1, 3.2 – 3.5*

7.2. **Juli Hancock:** The pre-cursor composite electrolyte paste can also be deposited by spray coating or powder coating to save time and materials. This way, objects can be coated at a larger scale.

7.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

7.3. **Troy Townsend:** This new technique may stimulate scientific exploration to incorporate other large or hygroscopic composite particles, which were previously incompatible with bath, jet or brush plating.

7.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.