**Handling/Mixing of**

**Micron- and Nano-Sized Powders**

Standard Operating Procedure

Lab: 206J - Lambros

Department: Aerospace Engineering

PI/Manager of Space: Prof. John Lambros

Written By: Joseph Gonzalez

**Section 1: Overview**

Type of SOP: Process Hazardous Material Hazardous Class of Materials Equipment

Synopsis:

*This SOP describes the proper equipment use, handling, and mixing of micron and nano-sized powders. This method should be reviewed prior to handling any powders to avoid risks of exposure via inhalation or skin. This SOP covers all powders existent within 206J for battery manufacturing and also polymer composite samples.*

**Section 2: Risk Assessment Summary (Hazards and control measures)**

*Information obtained from performing a risk assessment should be entered into this section.*

Materials:

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| **Material (name, CAS #, other ID)** | **Hazards** |
| Silicon Oxide Nanopowder 15-20nm (CAS 7631-86-9, US Research NanoMaterials-US3440) | Harmful by inhalation or skin contact. Irritation to respiratory system and eyes. |
| Silicon Dioxide Nanopowder 20-30 nm (CAS 7631-86-9, US Research NanoMaterials-US3438) | Harmful by inhalation or skin contact. Irritation to respiratory system and eyes. |
| Tin Nano Powder 60-80 nm (CAS 7440-31-5, US Research Nanomaterials-US1136) | Harmful by inhalation or skin contact. Irritation to respiratory system and eyes. |
| Tin Micron Powder (10 μm, <45 μm, and 150 μm, CAS 7440-31-5, Sigma Aldrich) | Harmful by inhalation or skin contact. Irritation to respiratory system and eyes. |
| Silicon Micron Powder (<45 μm, CAS 7440-21-3, Sigma Aldrich) | Harmful by inhalation or skin contact. Irritation to respiratory system and eyes. Flammable solid. |
| Graphite Micron Powder (< 25 μm, CAS 7782-42-5) | Not classified as hazardous substance or mixture. Avoid inhaling and skin contact. |
| Carbon Black (CAS 1333-86-4, TimCal) | Health hazards: Harmful by inhalation or skin contact and listed under as carcinogen (potential cause of cancer). |
| Polyvinylidene Flouride (PVDF) (CAS 24937-79-9, Sigma Aldrich) | Not classified as hazardous substance or mixture. Avoid inhaling and skin contact. |
| Sodium Carboxymethyl Cellulose (CMC) (CAS 9004-32-4, Sigma Aldrich) | Avoid inhaling and skin contact. Acute aquatic toxicity - harmful to aquatic life (avoid release to the environment and dispose correctly). |
| Comco Glass Beads (CAS 65997-17-3, Comco Inc. - 35, 50, 75 μm) | Not harzardous but avoid inhalation or skin contact. Irritation to respiratory system and eyes. Spilled material is slippery. |

Relevant References for Material Hazards:

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| Find MSDS for all materials above either in MSDS Binder in 206J or at the following web pages (direct link to MSDS)  **Silicon Oxide Nanopowder 15-20nm & Silicon Dioxide Nanopowder 20-30 nm:** http://s.b5z.net/i/u/10091461/f/MSDS-NANOPOWDERS/US3438.pdf  **Tin Nano Powder 60-80 nm:** http://s.b5z.net/i/u/10091461/f/MSDS-NANOPOWDERS/US1136.pdf  **Tin Micron Powder:** http://www.sigmaaldrich.com/catalog/product/aldrich/520373?lang=en&region=US  **Silicon Micron Powder:** http://www.sigmaaldrich.com/catalog/product/aldrich/215619?lang=en&region=US  **Graphite**: http://www.sigmaaldrich.com/catalog/product/aldrich/496596?lang=en&region=US  **Carbon black**: http://www.mtixtl.com/msds/timical%20msds.pdf  **PVDF**: http://www.sigmaaldrich.com/catalog/product/aldrich/182702?lang=en&region=US  **CMC**: http://www.sigmaaldrich.com/catalog/product/aldrich/419303?lang=en&region=US  **Glass Beads**: http://www.comcoinc.com/wp-content/uploads/2012/06/SD1002.pdf |

Equipment Hazards:

Equipment is considered low hazard. Take caution when cutting samples and removing from oven (wear oven mitts to prevent severe burns). Replace filter on the filtration hood every 3-6 months depending on usage. Avoid heat, flames, and sparks for Silicon powder (non of which are achievable with current equipment in 206J).

Hazardous Conditions:

At the conditions of manufacturing and handling taken in 206J, conditions are considered low hazard.

Technique Hazards:

* Weighing mass for the different powders (can go airborne if handled poorly)
* Mixing powders (too fast of mixing can cause powders to go airborne)
* Placing molds in oven and removing
* Cutting samples for experimental implementation (first – aid kit located on shelf above sink).

Personal Protective Equipment

The following are in addition to proper dress for work in a laboratory:

- Safety glasses

- Nitrile disposable gloves

- Lab coat

- Respiration mask (not using large quantities of powders so basic mask can be purchased from Roger Adams Lab)

Engineering Controls

- Store powders and mixtures in a proper plastic container.

- Store acetone and isopropyl alcohol in cabinet under the oven in a plastic container (refer to acetone and isopropyl alcohol MSDS sheets for proper handling).

- Mix powders within the ductless fume hood while the hood is off. When the circulation is on, it causes powders to go airborne while measuring mass. Following completion of mixing, can turn the hood ventilation on.

**Section 3: Procedures (generic mixing of powders)**

1. Wear PPE equipment (glasses, gloves, mask, lab coat) prior to starting
2. Gather mixing powders that you will be using for the particular sample that you are making and place inside the ductless fume hood (powders should be located next to ductless fume hood inside plastic containers (do not touch without PPE gloves).
3. Get all proper equipment needed for mixing: three spatulas, mortar and pestle (mixing powders), isopropyl alcohol, towels, weighing dishes or paper, and a glass container for storing the final mixed powder.
4. Using one spatula, weigh the correct amount of powder #1 by pouring onto the weighing paper or dish. Then pour the correct amount into the mortar.
5. Using a new spatula, weigh the correct amount of powder #2 on a new weighing paper or dish and pour into the mortar with powder #1.
6. Begin to slowly mix powders #1 and #2 for a few minutes.
7. Using a third spatula, weight the correct amount of powder #3 on a new weight paper or dish, and pour into the mortar powder mixture.
8. Slowly mix all three powders for an additional 1-2 minutes.
9. Repeat the process until all desired powders are acquired and mix until you observe a uniform color.
10. Once final powder is obtained, weight the amount of total powder to implement into the mold for curing in the oven. Place into mold carefully and break down any clumps that exist within the mold (molds are either stainless steel or aluminum).
11. Depending on binder used, different curing times will be used. Refer to the procedures for curing that come from the manufacturer or from other research papers online. In the case of PVDF, the melting temperature is 168 C so cure at 180C for 1.5 hrs to ensure binder melts.
12. Turn off oven and open the oven door and let mold cool down to room temperature (typically 2 hrs).
13. Unscrew mold sections and remove the final sample. Using a **NEW** razorblade each time, cut the desired sample sizes and store unused pieces in a container. Dispose razborblade into the red sharps container.
14. Clean up area per Section 4 below.
15. Remove PPE equipment. Dispose gloves in contaminated trash, hang up lab coat on hanger, put away glasses (clean with towel and isopropyl alcohol), and set aside the respiratory mask (after 3 uses for each mask, dispose and use a new one).
16. Wash your hands in sink!

**Section 4: Waste Disposal/Cleanup**

After curing samples and mixing powders, cleaning up the workstation is vital to prevent any contamination of subsequent samples from prior ones. Follow below procedure:

1. Turn ductless fume hood on and spray all surfaces with isopropyl alcohol
2. Wipe surfaces with towels or kim-wipes and repeat to ensure all powder mixtures are gathered
3. Dispose of the towels and kim-wipes in special trash container which includes towels and gloves with trace of powders listed above. Do not throw away contaminated gloves or towels in regular trash can.
4. Initially clean the spatulas used in mixing with isopropyl alcohol under the ductless fume hood and wipe surfaces under and dispose of contaminated towels. After getting powders off the spatulas, use the powder cleaner next to sink to clean the spatulas (biodegradable) and let dry. **Do not dispose Acetone or isopropyl alcohol in sink (they are flammable).**
5. Any trays of used for the mixing process should also be tossed in the contaminated trash with the towels and PPE gloves (if contaminated).
6. Cleaning of equipment can also be done using the sonicator after wiping with towels (dispose to contaminated trash). If using, place all parts within glass jar for cleaning and fill with isopropyl alcohol. Run sonicator for 60 minutes and dispose of isopropyl alcohol in the waste bottle under the sink (labeled – use a funnel to help with pouring). Rinse the inside with distilled water and pour a second amount of distilled water for another cleaning of 60 minutes in the sonicator. Following, dump water into the sink and dry components either manually or in oven at 110 C for a few hours.
7. Contact DRS for pick up of contaminated trash and isopropyl alcohol waste.

**Section 5: Emergency Response**

A dry powder fire extinguisher should be nearby.

Review the location of nearest safety shower before starting the procedure.

In case of a fire, identify two routes to exit the room.

**Section 6: Additional Information**

Advice:

1. Have computations done prior to mixing to prevent long exposure to powders and potentially using your laptop to make such calculations.
2. Write amounts for each component for mixing on paper and then the actual mass implemented in mixing on the paper. Input these parameters **after** you wash your hands and finished up clean up onto your computer. Do not use your computer during mixing these powders to avoid any transfer of powders.
3. Oven takes a little while to heat up; turn on to specified temperature when you begin mixing procedures to avoid waiting.
4. If using Carbon Black powder, cautious with sudden movements and when placing powder to weigh. Often times, fast abrupt movements cause the CB to “jump” and become messy. Handle with care to prevent to much contamination on weighing equipment or other areas.

Checklist:

Read (Material) Safety Data Sheets.

Completed Lab Safety Training (General, Chemical Handling)

Proper fire extinguisher is nearby.

Another researcher is nearby and knows the hazards present.

All calculations are done prior to beginning the procedure.

Additional gloves and masks are available in case those being used become contaminated.

References:

*Mixing procedures and setup can be found in the thesis of Joseph Gonzalez (UIUC – Ideals). Also, refer to the DRS wesbsite for any questions you may have on disposal and training.*

* Antartis D, Dillon S, Chasiotis I (2015) “Effect of porosity on electrochemical and mechanical properties of composite Li ion anodes”, J. Composite Mat., Vol 49(15).
* J. Gonzalez, K. Sun, M. Huang, J. Lambros, S. Dillon, I. Chasiotis (2014), Three dimensional studies of particle failure in Silicon based composite electrodes for Lithium ion batteries, Journal of Power Sources, 269: 334-343. DOI: 10.1016/j.jpowsour.2014.07.001
* Gates, M., J. Gonzalez, J. Lambros, and M. T. Heath (2014), "Subset Refinement for Digital Volume Correlation: Numerical and Experimental Applications.”, Experimental Mechanics: 1-15, DOI: 10.1007/s11340-014-9881-3

**Training Documentation**

Signing this document means that you have read and understand all aspects of this Standard Operating Procedure.

The supervisor is the person that acknowledges you took the training and understand the procedure. They can be a lab manager or researcher assigned by the PI to oversee this particular SOP.

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