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HAZOP & Operation Procedures for Template Growth Process

Team: Ray Chen, Zhengwei Li, and Ian Yan

Experiment Procedures:

1. The metal salts (typically nitrates or chlorides) solutions, complexing agent solutions and major doping agents solutions are prepared with certain concentrations (typically lower than 0.1mol/L) and stored in advance of the template growth process;
2. For each batch of template growth, metal salts, complexing agent, doping solution, additives, are mixed together in a beaker with a certain ratio, the total solution volume is about 80ML;
3. The solution is then transferred into a cap-locked glass container with the prepared templates (thin oxide films on glass substrates) in it;
4. The cap-locked container is then heated in an oven in which the temperature is kept at 95°C;
5. The process time is controlled according to different experimental purpose;
6. After the experiment is finished, the container is removed from furnace, and the templates are rinsed by deionized water and air dried before stored;
7. The container is then cleaned with water, acetic acid, deionized water in order.

List of main chemical involved in this procedure:

- $Zn(NO_3)_2$
- HMT (hexamethylenetetramine)
- $AgNO_3$
- $NH_3 \cdot H_2O$
- NaOH
- PEI
- HNO_3
- $Mg(NO_3)_2$
- $Al(NO_3)_3$
- HCl

Potential Hazards, Consequence and Safeguards:

Potential Hazards	CONSEQUENCE	SAFEGUARDS	ACTION
Contact or inhalation of some chemicals	May cause sensitisation, burns to skin, damage to eyes, or other health problems	Always wear lab coat, gloves safety glasses while handling chemicals; Wear mask while handling powders NH_3 , HNO_3 and HMT.	Strictly follow the procedure described on MSDS
High temperature of oven and container when removing samples	May cause burns if not using appropriate equipment	Wearing insulating gloves to remove the contained from the oven	Run cold water over the burnt area for 10 minutes for first aid

Operation and Shutdown Procedures for Salt Spray Cabinet

The salt spray cabinet is used for accelerated evaluation of corrosion resistance of metals, alloys and/or coatings on various substrates. The normal operation of the cabinet could be followed the procedures described below:

- 1) Switch on the main power supplied to the cabinet and adjust the set point to the desired temperature;
- 2) Open the filling valve on top of the humidifier, fill the vessel with clean drinking quality water (the level should be ~100mm from the top), close the filling valve;
- 3) Connect the humidifier to the power supply and adjust the set point to the desired temperature;
- 4) Prepare the brine solution. Fill the reservoir with brine solution until the float valve just becomes buoyant. Fill the header tank with brine solution and replace the cap. Open the cock on the header tank;
- 5) Put samples into the cabinet with suitable supporting rack;
- 6) Secure the spray baffle in an upright position;
- 7) Replace the lid and make sure that the bottom edge of the lid engages in the perimeter trough. Pour about 50mL of water carefully over the lid;
- 8) Set the air selector switch on top of the humidifier to the right position. Pull out the knob on the face of the humidifier to unlock the air pressure regulator. Slowly turn the knob clockwise to admit air into the vessel. Adjust the air pressure to 1.6bar and press the knob in to lock the regulator;
- 9) Spray nozzle should now be working;
- 10) Check the solution level in the salt header tanks and the water level in the humidifier regularly;
- 11) To shutdown the system for sample check: shut off the compressed air supply, switch off the brine supply from the header tank, and wait for the cabinet is clear with significantly reduced salt fog.

For emergency shutdown:

Turn off the compressed air supply;

Switch off the power supply to cabinet and humidifier

Potential hazards:

In general, sodium chloride (NaCl, NON-HAZARDOUS SUBSTANCE, NON-DANGEROUS GOODS) was used to prepare dilute salt solutions (<5wt.%) for this equipment and measurements.

The potential hazard involved might be related to the release of salt fog. To avoid this, the trough must be filled with enough amount of water to make sure that the cover lid is water sealed during test and the vent is clear and placed at the right location.

Switch on the fan over the windows during operation.

Always wear personal protective equipment, such as safety glasses, mask, and gloves during operation.

List of operators:

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Operation and Shutdown Procedures for High Temperature Furnaces

The furnaces are mainly used for heat treatment and oxidation experiments. The maximum temperature can be 1200°C, normal operation will be 400 to 800°C.

The operation can be described as the following:

- 1) Check the furnace tube is clean;
- 2) Switch on the main power supply;
- 3) Adjust the temperature set point on the temperature controller;
- 4) Allow the furnace to reach the desired temperature;
- 5) Push the sample into the middle of the furnace tube using a long metallic rod (this can be done before heating started depending on requirement);
- 6) Pull the sample out for characterization;
- 7) After use, switch off the power

For emergency shutdown:

Turn the main power supply off

Potential hazards:

No chemicals will be used with these furnaces. A potential hazard might be the relatively high temperature of the furnace shell after long term running, this might result in burning when touching. To avoid this, notice should be given. When operation, wear personal protective equipments, such as safety glasses, heat-resistant gloves. When burn happens, run cold water over the burnt area for at least 10 minutes for first aid.

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Operation and Shutdown Procedures for Wet Oxidation Equipment

The wet oxidation equipment is used to carry out oxidation of metals or alloys at temperatures lower than 600°C with the incorporation of water vapor. The addition of water into the oxidizing atmosphere is to control the growth of low-dimensional oxide nanostructures.

The equipment has 5 parts: furnace, reaction chamber produced by quartz tube, water bath, compressed gas bottle, gas flow regulator. The operation of this equipment is described as the following:

- 1) Turn the power to the water bath on;
- 2) Attach the sample to the left-hand side of the pushing rod;
- 3) Seal the quartz tube with the cap;
- 4) Open the compressed gas bottle and introduce the carrier gas into the reaction chamber by passing the bottle sitting in the water bath; The flow rate should be controlled with the regulator carefully;
- 5) Switch on the temperature controller of the furnace and adjust the set-point;
- 6) Allow the furnace to reach the set-point and the reaction chamber is flushed by the carrier gas with a long enough time period;
- 7) Re-adjust the gas flow if necessary;
- 8) Push the sample into the reaction chamber using the magnet attached and start oxidation;
- 9) Pull the sample out; close the compressed gas bottle; open the sealing cap and take the sample out;
- 10) Switch off the power supplied to the furnace and water bath

For emergency shutdown:

Turn the main power supply off

Potential hazards:

No chemicals will be used with this equipment.

Potential hazards might be:

- 1) The temperature of the furnace steel shell after long term running might be high; this might result in burning when touching. To avoid this, notice has been given. When operation, wear personal protective equipments, such as safety glasses and heat-resistant gloves; If burned, please run cold water over the burnt area for at least 10 minutes for first aid;
- 2) The temperature of the heating band to the tube connected between the water bath and the reaction chamber should be carefully controlled. If the temperature is too low, water vapour may be condensed and block the gas flow. A temperature indicator is inserted into the band to monitor the real temperature during operation.
- 3) Water level in the water bath should be high enough to ensure that the bottle inside is heated uniformly. If the water level is low, the heater will switch off automatically; this will not result in any hazards to the equipment or operator. However, during operation, check the water level regularly and make sure the reservoir water tank is filling water into the water bath at a suitable rate continuously.

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HAZOP and operation/shutdown procedures

Project: Sol-Gel Processing of ZnO thin films

By: Chun (Melanie) Li and Noviana Tjitra Salim

Project Objectives:

1. To achieve the desired properties of the ZnO thin films by sol-gel technique and to develop p-type ZnO thin films
2. To achieve the desired properties of the ZnO thin films by sol-gel technique and to develop insulating ZnO thin films
3. To establish the relationship between processing conditions and microstructure of the ZnO thin films by so-gel technique

Experimental Procedure:

1. Zinc precursors and oxygen precursors were dissolved in de-ionized water, and mixed together
2. The solution is stirred at 90°C for 2 hours
3. ZnO thin films were prepared on glass substrate by the use of dip-coater adjusted at previously fixed pulling rate of 6 cm/minute
4. After depositing each layer, the sample was dried in an oven for 10 minutes at 90-100°C and this process of dipping and drying is repeated
5. The film is annealed in air at 500°C for 1 hours in a tube furnace

List of Chemicals Used:

1. Zinc nitrate
2. Zinc carbonate
3. Zinc chloride
4. Zinc acetate
5. Citric acid
6. Starch
7. Polyethylene glycol
8. Absolute ethanol
9. Potassium chloride
10. Urea

HAZOP Analysis:

Potential hazards involved	Consequence	Suggestions
Solution overheated on hotplate	Solution evaporation	Carefully monitor heating temperature and time

Hot samples taken from the tube furnace	Skin burn	Wear PPE always
Skin touches chemicals	Skin irritation	Wear PPE always
Flammable ethanol close to the hot source	Fire	Ethanol away from furnace and stored in a safe and cool place

Personal Protective Equipments (PPE) Needed:

1. Lab coats
2. Safety glasses
3. Gloves