

# WORK PERMIT

Department of Chemical and Biological Engineering

化學工程及生物分子工程學系

Project Title: Controllable Synthesis of Highly Effective  
Catalyst for Photoelectrocatalytic Hydrogen  
Evolution Reaction and its Combination  
with CO<sub>2</sub> Reduction.

Researcher(s): Cong LI

Supervisor(s): Prof. Xijun HU



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Signature of Approval: 

Prof. John Burford  
Department of Safety

## 2. ADDENDUM TO WORK PLAN

Addendum to Work Plan 16106

### Changes to Work Plan

- ✓ Additional Chemicals
- ☐ Additional Instrumentation Work in Common Labs
- ☐ Additional Instrumentation Work in MCPF
- ☐ Additional Instrumentation Work in other Labs
- ☐ Additional Experimental Procedures
- ☐ Change in Existing Experimental Procedures
- ☐ Additional Name/s to existing Work Plan
- ☐ Removal of Name/s to existing Work Plan

### NOTE

Addition or removal of names to work plan are discouraged as every individual should have their own work plan number. Group members in work plan are usually applied to FYP projects or special circumstance whereby group training is needed on common equipment.

**The Hong Kong University of Science & Technology**

**Department of Chemical Engineering**

**Work Plan No.: 16106** (Amend(I))

**Title: Controllable synthesis of highly effective catalyst for  
photoelectrocatalytic hydrogen evolution reaction and its  
combination with CO<sub>2</sub> reduction**

<b>Researcher:</b>	Cong LI
<b>Supervisor:</b>	Prof. Xijun Hu & Prof. Frank Leung-Yuk Lam
<b>Research area:</b>	Chemical and Biomolecular Engineering
<b>Date:</b>	3 July 2017

## Section 1 General Information

**Name of Researcher** : Cong LI  
**Name of Project Supervisors:** Prof. Xijun Hu & Prof. Frank Leung-Yuk Lam

**Project Title** : Controllable synthesis of highly effective catalyst  
for photoelectrocatalytic hydrogen evolution reaction  
and its combination with CO<sub>2</sub> reduction

**Research Area** : Chemical and Biomolecular Engineering

**Proposed Location** : HKUST Academic Building Room 7103

**Start Date** : 1 November 2016

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## Section 2: Project Description

### 2.1 Project Aims & Objectives

The aims and the objectives of this project are:

- a.) to develop photoelectrocatalyst system for high-performance hydrogen evolution reaction (HER).
- b.) to optimize the growth parameters for preparation of photoelectrocatalyst with large surface area and high conductivity.
- c.) to characterize the physical and chemical properties of the photoelectrocatalyst, like thickness and crystallinity.
- d.) To develop photoelectrochemical cell using optimized photoelectrocatalyst to test the HER efficiency.
- e.) to study the combination of both catalysts for HER and CO<sub>2</sub> reduction, and its photoelectrocatalytic activity, such as Faraday efficiency, product yield and selectivity.

### 2.2 Method

Layered transition metal dichalcogenides (TMDCs) are my priority as HER catalyst. In this study we plan to utilize chemical vapor deposition (CVD) method to accomplish the catalyst preparation. There are two kinds of TMDCs catalyst, including MoS<sub>2</sub> and VS<sub>2</sub>. All CVD synthesis can be performed on a multi-temperature-zone tube furnace equipped with a 1-in. -diameter quartz tube. The samples will be characterized by optical microscopy, Raman spectroscopy, AFM, SEM, TEM and so on. And HER efficiency measurements will be achieved on an electrochemical workstation using a conventional three-electrode system with the as-prepared electrode as the working electrode, a saturated calomel electrode (SCE) as the reference electrode and a platinum foil<sup>1</sup> as the counter electrode in 0.5M H<sub>2</sub>SO<sub>4</sub><sup>2</sup> aqueous solution. Commercial gold<sup>3</sup>, mica<sup>4</sup>, sapphire<sup>5</sup>, molybdenum trioxide<sup>6</sup> (MoO<sub>3</sub>), sulfur powder<sup>7</sup>, vanadium chloride<sup>8</sup> (VCl<sub>3</sub>), sodium bicarbonate<sup>9</sup> (NaHCO<sub>3</sub>), etc., will be used if applicable. All the above materials will be examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). To ensure the functions of the developed materials, some measurement will be performed. All the work will be carried out at HKUST.

### Section 3: Equipment List

Equipment	Location
Electronic balance to +/- 0.0001g accuracy	7103
Multi-temperature-zone tube furnace	7103
Mechanical pump	7103
Heating belts & temperature controller	7103
Optical microscopy (OM)	7101
Orion pH meter	7103
Scanning electron microscope (SEM)	MCPF
Raman spectroscopy	7106
Beakers (of various volumes), test-tubes, sample holder bottles	7101
X-ray diffraction (XRD) equipment – PW 1840 diffractometer (Philips)	2150
X-ray fluorescence (XRF) – PW 1400 spectrometer, Philips	2150
Nitrogen adsorption measurements – Omnisorp 100CX (Coulter)	7101
X-ray photoelectron spectroscopy (XPS) – Perkin-Elmer model 560 spectrometer with a double-pass cylindrical mirror analyzer and a vacuum system giving a base pressure of $\sim 10^{-9}$ Torr	2149
Transmission electron microscope (TEM) – JEOL 2010F microscope with EDEX analysis	2218
Induced Coupled Plasma (ICP) - Perkin Elmer Optima 3000 XL	7101
Fourier Transform Infrared (FTIR) Spectroscopy – Perkin Elmer 2000 FTIR Spectrometer with MCT detector	7106
Elemental Analyser – CHONS (Vario ELII Eliminator)	2149
Thermogravimetric Analysis (TGA) – Shimadzu TGA-50	2152
Electrochemical workstation	7103
Gas chromatograph (GC)	7103
High performance liquid chromatography (HPLC)	7101
UV-vis spectroscopy	7101
Heating Stage	7101
Spin coater	7103

## Section 4: Experimental Procedures

### 4.1 Preparation of layered nanoelectrocatalyst via CVD/LPCVD on various substrates

#### 4.1.1 Preparation of 2D layered $\text{MoS}_2$ with high nucleation density and/or dendritic property by low-pressure CVD.

Specific substrates should be selected carefully. Take the sulfur-rich atmosphere in the furnace into consideration, gold<sup>3</sup> foil and some kinds of insulator like mica<sup>4</sup> and sapphire<sup>5</sup> are suitable for this purpose. In this regard, insulator foils should be polished to a mirror finish by either/both sides before use. The growth of  $\text{MoS}_2$  is performed inside a multi-temperature-zone tube furnace equipped with a 1-in. -diameter quartz tube. Sulfur<sup>7</sup> powder is mildly sublimated at  $\sim 102^\circ\text{C}$  with heating belts outside the hot zone. Then Ar gas (50 standard cubic centimeters per minute (sccm)) is introduced to carry the sulfur vapor to the downstream growth zone.  $\text{MoO}_3$ <sup>6</sup> powders and substrate lie sequentially inside the hot zone, where the reaction occurs. A temperature of  $526^\circ\text{C}$  –  $530^\circ\text{C}$  is adopted for  $\text{MoO}_3$ <sup>6</sup> precursors.  $\text{MoO}_3$ <sup>6</sup> in the far left temperature zone is first heated into vapor state, which is then partially reduced by upstream vaporized S carried by Ar gas to form  $\text{MoO}_{3-x}$ . The volatile suboxide species  $\text{MoO}_{3-x}$  undergo further sulfurization to eventually form  $\text{MoS}_2$  on substrates, which is located on the middle zone of the furnace. A cooling device containing acetone<sup>10</sup> and drikold<sup>11</sup> is placed between the furnace and the pump in order to cool and deposit the waste gas mainly containing sulfur vapor.

#### 4.1.2 Preparation of 2D layered metallic $\text{VS}_2$ by atmospheric-pressure CVD.

Similar to the above description, a solid-phase reaction in CVD is performed to synthesize  $\text{VS}_2$  nanoflakes. But this time we choose  $\text{VCl}_3$ <sup>8</sup> rather than metallic oxide ( $\text{VO}_x$ ) as the V-contained precursor to react with sulfur<sup>7</sup>. To note, sulfur<sup>7</sup> powder and  $\text{VCl}_3$ <sup>8</sup> powder are placed in the same holder in the suitable place of the furnace, as well as the substrate. When the temperature arrives the set value, both the precursors holder and the substrate are push into set points of the furnace to trigger the reaction. Then Ar gas (50 sccm) and  $\text{H}_2$  gas (10 sccm) are introduced to carry the sulfur vapor to the downstream growth zone. When the temperature of furnace drops to room temperature after growth, substrate can be taken out to perform further characterizations.



## 4.2 Sample transfer to arbitrary substrates

**This part is for the further characterization and constructing co-catalyst for CO<sub>2</sub> reduction.**

### 4.2.1 MoS<sub>2</sub> transfer to arbitrary substrates

To transfer the as-grown MoS<sub>2</sub> flakes, the MoS<sub>2</sub>/substrate sample is first spin coated with 0.1 ml poly (methyl methacrylate) (PMMA<sup>12</sup>), and after 5-minute heating on the heating plate with 170 °C. The above part of experiments will be done on the fumehood in Room 7103. Then the sample is soaked in hydrofluoric acid (HF<sup>13</sup>) or other proper etching reagents<sup>14-16</sup> for the separation of sample and substrate. Finally, the PMMA<sup>12</sup>-supported MoS<sub>2</sub> was rinsed with deionized (DI) water for at least three times to completely remove the etching solution. A fresh substrate like SiO<sub>2</sub>/Si<sup>17</sup> and PET<sup>18</sup> is then used to ‘fish out’ the PMMA<sup>12</sup>-capped MoS<sub>2</sub> film, followed by rinsing with acetone<sup>10</sup> and annealing for removing the PMMA<sup>12</sup>. This part of experiments will be done at Room 3234 in NFF with the supervision of NFF engineer (Mr Henry Chun Fai Yeung). To attain intact transfer of MoS<sub>2</sub> from original substrate to current substrate (c-substrate), the MoS<sub>2</sub>/c-substrate is put under infra-red lamp or on the heating plate for ten minutes to get rid of residue DI water. This will be done in Room 7103. As for the HF-related experiments, the whole set of protective equipment will be provided by NFF. All the operations will be done under the supervision of the NFF engineer after I pass the safety training assessment of NFF.

### 4.2.2 VS<sub>2</sub> transfer to arbitrary substrates

For VS<sub>2</sub>, it is much easier than the MoS<sub>2</sub> transfer. The above procedure still applies to transfer of VS<sub>2</sub> with single layer or bilayer. In regard to multilayered VS<sub>2</sub> transfer, mechanical exfoliation is dominant. We can directly press c-substrate onto the surface of sample and keep for a few seconds, most of VS<sub>2</sub> flakes will attach to the c-substrate. This process can be achieved due to the loose packing effect and weak Van de Waals interaction between the neighboring VS<sub>2</sub> layer.

## 4.3 HER measurements

All the electrochemical measurements are performed in a three-electrode system on an electrochemical workstation, using transferred MoS<sub>2</sub>/VS<sub>2</sub> or as-grown MoS<sub>2</sub>/VS<sub>2</sub> on

conductive substrate as the working electrode, a carbon rod<sup>19</sup> or a platinum<sup>1</sup> foil as a counter electrode, and a saturated calomel (SCE) reference electrode. All the potentials are calibrated to a reversible hydrogen electrode (RHE). Linear sweep voltammetry with a scan rate of  $5 \text{ mV s}^{-1}$ , from +0.30 V to -0.32 V vs. RHE is conducted in  $0.5 \text{ M H}_2\text{SO}_4$  and some other neutral solutions like  $\text{NaHCO}_3$ <sup>9</sup> (sparged with pure  $\text{N}_2$  for 40 minutes, purity 99.999%). AC impedance measurements were carried out at  $\eta = 0.15 \text{ V}$  from 100 kHz–0.01 Hz. Durability test with a scan rate of  $50 \text{ mV s}^{-1}$ , from +0.30 V to -0.32 V vs. RHE is conducted for 1000 cycles. After that a linear sweep voltammetry will be conducted to detect the stability of catalyst during test. In all electrochemical tests, the reference electrode will be connected with electrolyte by a home-made salt bridge. 30g of agar powder<sup>21</sup> is first mixed with KCl powder in hot water ( $100^\circ\text{C}$ ) and then directly filled into a U-shape glass tube before it solidifies. This is a regular method for salt bridge.

For the photoelectrochemical measurements, the whole system will be in an irradiation of  $10 \text{ mW/cm}^2$ . Then we will follow the above procedure to perform further experiments.

#### 4.4 Sample Characterization

The reactant/products will be characterized using the techniques below:

##### *X-ray Diffraction (XRD)*

X-ray diffraction (XRD) patterns are recorded on a PW 1840 diffractometer (Philips), Co-K $\alpha$  radiation (40 kV, 25 mA), Fe filter, 0.2 mm slit. The samples are crushed into a powder or directly placed in the sample holder where the analysis is automated. The  $2\theta$  ranges from 10 to  $90^\circ$ , the scan rate used will be  $0.025^\circ/\text{min}$ . This characterization will be conducted in MCPF.

##### *Nitrogen Adsorption*

The BET surface area and porosity of the catalyst is obtained from the nitrogen adsorption isotherms at liquid nitrogen temperature, performed on an automated adsorption instrument (Quantachrome NOVA 1200, USA). The surface area of the catalyst is calculated using the BET equation, and the total pore volume measured at  $P/P_0 = 0.98$ . Physisorption is used to characterize pore size distribution, pore shape and surface area of the catalyst. Firstly, the sample is weighed and placed in the sample holder where it is connected to the vacuum pump for outgassing. The sample holder can then be heated to the desired outgassing temperature. Once degassing is completed it can be removed and the bath with the reference tube can be filled with liquid nitrogen.

Finally, the sample is transferred to the measurement port. This characterization will be conducted in 7101.

#### *Fourier Transform Infrared (FTIR) Spectroscopy*

FTIR spectra are measured on a Perkin Elmer 2000 FTIR spectrometer equipped with MCT detector. This characterization will be conducted in MCPF.

#### *Transmission Electron Microscope (TEM)*

The TEM images of a typical sample are obtained on a JEOL 2010F microscope with energy dispersive X-ray analysis. This characterization will be conducted in MCPF.

#### *Thermogravimetric Analysis (TGA)*

The weight loss, dehydration and dihydroxylation for the sample is evaluated by a TGA (Shimadzu TGA-50) technique. About 10 mg of the sample is placed in a platinum pan and heated from room temperature to 1000 °C at a heating rate of 2K/minute in air with a flow rate of 50ml/min. This characterization will be conducted in 7101.

#### *Scanning Electron Microscopy (SEM)*

The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. The electron beam is generally scanned in raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. This characterization will be conducted in MCPF.

#### *X-ray photoelectron spectroscopy (XPS)*

X-ray photoelectron spectroscopy (XPS) is a surface-sensitive quantitative spectroscopic technique that measures the elemental composition at the parts per thousand range, empirical formula, chemical state and electronic of the elements that exist within a material. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 0 to 10 nm of the material being analyzed. This characterization will be conducted in MCPF.

### *Gas chromatograph (GC)*

The gaseous compounds being analyzed interact with the walls of the column, which is coated with a stationary phase. This causes each compound to elute at a different time, known as the retention time of the compound. The comparison of retention times is what gives GC its analytical usefulness. This characterization will be conducted in 7101 or 7103.

### *High performance liquid chromatography (HPLC)*

Each component in the sample interacts slightly differently with the adsorbent material, causing different flow rates for the different components and leading to the separation of the components as they flow out the column. This characterization will be conducted in 7101.

### *UV-vis spectroscopy*

Molecules containing  $\pi$ -electrons or non-bonding electrons (n-electrons) can absorb the energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals. This characterization will be conducted in 7101.

### *X-ray fluorescence (XRF)*

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays. This characterization will be conducted in MCPF.

### *Micro Raman*

Raman spectroscopy is a spectroscopic technique used to observe vibrational, rotational, and other low-frequency modes in a system. It relies on inelastic scattering, or Raman scattering, of monochromatic light, usually from a laser in the visible, near infrared, or near ultraviolet range. The laser light interacts with molecular vibrations, phonons or other excitations in the system, resulting in the energy of the laser photons being shifted up or down. The shift in energy gives information about the vibrational modes in the system. This characterization will be conducted in 7106.

### *Optical microscope (OM)*

The optical microscope is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. The image from an optical microscope can be captured by normal light-sensitive cameras to generate a micrograph. This characterization will be conducted in 7101.

#### *X-ray fluorescence (XRF)*

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays. This characterization will be conducted in MCPF.

### **Section 5: Operating Conditions**

#### **Preparation of 2D layered MoS<sub>2</sub>.**

- Pressure : low pressure (~1 Pa)
- Temperature : 850 - 880 °C
- Flow rates: batch operations therefore not applicable

#### **Preparation of 2D layered metallic VS<sub>2</sub>.**

- Pressure : atmospheric
- Temperature : 750 - 900 °C
- Flow rates: batch operations therefore not applicable

#### **MoS<sub>2</sub> transfer to arbitrary substrates.**

- Pressure : atmospheric
- Temperature : 25 °C
- Flow rates: not applicable

#### **VS<sub>2</sub> transfer to arbitrary substrates**

- Pressure : atmospheric

- Temperature : 25 °C
- Flow rates: not applicable

#### **Electrochemical HER measurements.**

- Pressure : atmospheric
- Temperature : 25 °C
- Flow rates: gas flow rate of reactants at 20 ml/min

#### **Photoelectrochemical HER measurements.**

- Pressure : atmospheric
- Temperature : 25 °C
- Flow rates: gas flow rate of reactants at 20 ml/min

### **Section 6: Services List**

- Electricity (AC 220V, 50Hz)
- Double de-ionized water
- Tap water
- Argon gas
- Hydrogen gas
- Light source
- Carbon dioxide
- Fume hood

### **Section 7: Procedure Template**

<b>Experimental Procedure No.</b>	<b>Experimental Procedure Description</b>	<b>Scale(Mass/Volume)</b>	<b>Location</b>	<b>Method</b>
4.1.1	Sulfur <sup>7</sup> powder is mildly sublimated at ~102 °C with heating belts outside the hot zone. Then Ar gas (50 standard cubic centimeters per minute (sccm)) is introduced to carry the sulfur vapor to the downstream growth zone. MoO <sub>3</sub> <sup>6</sup> powders	~7.2 L gas flow per experiment; Sulfur <sup>7</sup> powder: 5 g; MoO <sub>3</sub> <sup>6</sup> powders: 1 g	Chemical vapor deposition system in 7103	Existing

	and substrate lie sequentially inside the hot zone, where the reaction occurs. A temperature of 526 °C – 530°C is adopted for MoO <sub>3</sub> <sup>6</sup> precursors.			
4.1.2	Sulfur <sup>7</sup> powder and VCl <sub>3</sub> <sup>8</sup> powder are placed in the same holder in the suitable place of the furnace, as well as the substrate. When the temperature arrives the set value, both the precursors holder and the substrate are push into set points of the furnace to trigger the reaction. Then Ar gas (50 sccm) and H <sub>2</sub> gas (10 sccm) are introduced to carry the sulfur vapor to the downstream growth zone. When the temperature of furnace drops to room temperature after growth, substrate can be taken out to perform further characterizations.	~7.2 L gas flow per experiment; Sulfur <sup>7</sup> powder: 5 g; VCl <sub>3</sub> <sup>8</sup> powder: 2 g	Chemical vapor deposition system in 7103	Existing
4.2.1	The MoS <sub>2</sub> /substrate sample is first spin coated with 0.1 ml poly (methyl methacrylate) (PMMA <sup>12</sup> ), and after 5-minute heating on the heating plate with 170 °C.	PMMA <sup>12</sup> : 0.2 ml;	Fumehood In 7103	Existing
	Then the sample is soaked in hydrofluoric acid (HF <sup>13</sup> ) or other proper etching reagents for the separation of sample and substrate. Finally, the PMMA-supported MoS <sub>2</sub> was rinsed with deionized (DI) water for at least three times to completely remove the etching solution. A fresh substrate like SiO <sub>2</sub> /Si <sup>17</sup> and PET <sup>18</sup> is then used to 'fish out' the PMMA <sup>12</sup> -capped MoS <sub>2</sub> film, followed by rinsing with acetone <sup>10</sup> and annealing for removing the PMMA <sup>12</sup> .	30 mL per experiment; HF <sup>13</sup> : 10 ml; DI water: 100 ml	NFF lab (Room 3234)	Existing
	To attain intact transfer of MoS <sub>2</sub> from original substrate to current substrate (c-substrate), the MoS <sub>2</sub> /c-substrate is put under infra-red lamp or on the heating plate for ten minutes to get rid of residue DI water.	---	Fumehood In 7103	Existing
4.2.2	We can directly press c-substrate onto the surface of sample and keep for a few seconds, most of VS <sub>2</sub> flakes will attach to the c-substrate	2 g per experiment	Fumehood in 7103	Existing
4.3	All the electrochemical measurements are performed in a three-electrode system on an	2 L gas flow and 100 mL mixed solution per experiment	Fumehood in 7103	Existing

	electrochemical workstation, using transferred MoS <sub>2</sub> /VS <sub>2</sub> or as-grown MoS <sub>2</sub> /VS <sub>2</sub> on conductive substrate as the working electrode, a carbon rod <sup>19</sup> or a platinum <sup>1</sup> foil as a counter electrode, and a saturated calomel (SCE) reference electrode. The electrolyte solution is 0.5M H <sub>2</sub> SO <sub>4</sub> <sup>2</sup> acid solution.			
	In all electrochemical tests, the reference electrode will be connected with electrolyte by a home-made salt bridge. 30g of agar powder <sup>21</sup> is first mixed with KCl powder in hot water (100oC) and then directly filled into a U-shape glass tube before it solidifies.	30 g agar powder and 27 g KCl per experiment	Fumehood in 7103	Existing

### Section 8: Chemicals List

No.	Chemical	Purity	Quantity per Experiment
1	Platinum	99.99%	10 g
2	Sulfuric acid	98%	2.5 ml
3	Gold foil	99.95%	1 cm <sup>2</sup>
4	Mica	100%	0.5 cm <sup>2</sup>
5	Sapphire	100%	0.5 cm <sup>2</sup>
6	Molybdenum trioxide	>99.5%	0.5 g
7	Sulfur	>99%	5 g
8	Vanadium chloride	97%	1 g
9	Sodium bicarbonate	>99%	80 ml
10	Acetone	99.9%	200 ml
11	Drikold	100%	100 g
12	PMMA	10%	0.1 ml
13	Hydrofluoric acid	45-55%	5 g
14	Sodium hydroxide	100%	24 g
15	Ferric chloride	100%	5g
16	Iodine	100%	2g
17	SiO <sub>2</sub> /Si	100%	1 cm <sup>2</sup>
18	PET foil	100%	>1 cm <sup>2</sup>
19	Carbon rod	99.95%	10 g
20	Ethanol	99.5%	100 ml
21	Agar	Cell-cul	30g



## Section 9: Summary of Relevant Hazards and Incompatibilities

HAZOP Template							
Hazard and Operability Analysis							
Activity: 4.1.1 Preparation of 2D layered MoS <sub>2</sub> with high nucleation density and/or dendritic property by low-pressure CVD.							
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	MINIMISE RISK BY	RESIDUAL RISK
1	Skin contact and inhalation of acetone <sup>10</sup>	Causes damage to the following organs: upper respiratory tract, skin, central nervous system (CNS), eye, lens or cornea. Very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.	H	L	H	Splash goggles. Lab coat. Dust respirator. Use an approved respirator or equivalent. Gloves.	L
2	Skin contact and inhalation of ethanol <sup>20</sup> , Volatilization of ethanol <sup>20</sup>	It is flammable, its vapor and air can form explosive mixture. Open flame, high heat cause burning explosion. And oxidant contacts occur chemical reaction or cause combustion.	L	L	L	Do not work close to heat/fire sources, wear eyeglasses or chemical safety goggles and wear gloves and clothing to prevent skin exposure	L
3	Leak of hydrogen gas	Combustible gas. May cause combustion and explosion.	H	L	H	Place sealing gaskets at the joint. Set up gas	L

4	Skin contact of drikold <sup>11</sup>	Dry ice sublimates at $-78.5^{\circ}\text{C}$ . Dangerous due to burns caused by freezing.	L	L	L	Wear gloves and lab coat	warning system. Monitor the gas flow time to time	L
5	Powder inhalation	Toxic by inhalation, in contact with skin and if swallowed. Causing respiratory tract	H	M	H	Take the measurement within the fume hood		L
6	Wrongly programmed furnace setting	Uncontrolled temperature increase	H	M	H	Set up the furnace carefully and check the temperature within 2h		L
7	Skin contact of hot surface of the furnace	Skin burn if touched	H	M	H	Set a warning sign and operate with protective gloves. Take out the samples until the temperature decrease below $100^{\circ}\text{C}$ .		L
8	Leak of waste gas ( $\text{H}_2\text{S}$ )	Have corrosive effect on human tissue, with the potential to damage respiratory organs, eyes, skin, and intestines irreversibly.	M	L	M	Wear gloves, protective eye goggles. Set up waste gas treatment system.		L
FINAL ASSESSMENT: Have some Risks that can be controlled by careful operation by wearing PPE and careful operation								OVERALL RISK:
								L

HAZOP Template						
Hazard and Operability Analysis						
Activity: 4.1.2 Preparation of 2D layered metallic VS <sub>2</sub> by atmospheric-pressure CVD.						
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	RESIDUAL RISK
1	Skin contact and inhalation of ethanol <sup>20</sup> ; Volatilization of ethanol <sup>20</sup>	It is flammable, its vapor and air can form explosive mixture. Open flame, high heat cause burning explosion. And oxidant contacts occur chemical reaction or cause combustion.	L	L	L	L
2	Leak of hydrogen gas	Combustible gas. May cause combustion and explosion.	H	L	L	L
3	Powder inhalation	Toxic by inhalation, in contact with skin and if swallowed. Causing respiratory tract	H	L	L	L
4	Wrongly programmed furnace setting	Uncontrolled temperature increase	H	M	L	L
5	Skin contact of hot surface of the furnace	Skin burn if touched	H	M	H	L

6	Leak of waste gas (HCl)	Have corrosive effect on human tissue, with the potential to damage respiratory organs, eyes, skin, and intestines irreversibly.	M	L	H	gloves. Take out the samples until the temperature decrease below 100 °C.  Wear gloves, protective eye goggles. Set up waste gas treatment system.	L
FINAL ASSESSMENT: Have some Risks that can be controlled by careful operation by wearing PPE and careful operation							OVERALL RISK: L

HAZOP Template							
Hazard and Operability Analysis							
Activity: 4.2.1 MoS <sub>2</sub> transfer to arbitrary substrates. & 4.2.2 VS <sub>2</sub> transfer to arbitrary substrates.							
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	RESIDUAL RISK	
1	Skin contact of hydrofluoric acid <sup>13</sup>	A highly corrosive liquid, hydrofluoric acid is also a powerful contact poison.	H	L	H	Chemical-resistant clothing is recommended, including gloves, apron, and goggles.	L
2	Skin contact and inhalation of sodium hydroxide <sup>14</sup>	May be fatal if swallowed. Harmful is inhaled. Causes burns to any area of contact.	H	L	H	Wear eyeglasses or chemical safety goggles and wear gloves and clothing to prevent skin exposure	L

3	Contact of heating plate	Burned if contacted	M	L	M	Wear appropriate protective gloves	L
4	Skin contact and inhalation of acetone <sup>10</sup>	Causes damage to the following organs: upper respiratory tract, central nervous system (CNS), eye, lens or cornea. Very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.	H	L	H	Splash goggles. Lab coat. Dust respirator. Use an approved respirator or equivalent. Gloves.	L
FINAL ASSESSMENT: Have some Risks that can be controlled							OVERALL RISK: L

HAZOP Template Hazard and Operability Analysis						
Activity: 4.3 HER measurements						
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	RESIDUAL RISK
1	Skin contact and inhalation of sulfuric acid <sup>2</sup>	It is fatal if inhaled, absorbed through skin or swallowed. It can cause severe eye and skin burns, respiratory tract burns, suspect cancer hazard. Contact with other material	H	L	H	Wear self-contained breathing apparatus, rubber boots and heavy rubber gloves. Full suit.
						L

		may cause fire. It is water reactive.						
FINAL ASSESSMENT: Have some risks that can be controlled by wearing PPE and careful operation								OVERALL RISK: L

HAZOP Template								
Hazard and Operability Analysis								
Activity: 4.4 Sample Characterization (Operated by MCPF)								
N O	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	MINIMISE RISK BY	RESIDUAL RISK	
	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
FINAL ASSESSMENT: Have some risks that can be controlled by wearing PPE and careful operation								OVERALL RISK: N/A

Remark: Severity - L=Low (Minor injuries, first aid); M=Medium (Hospitalization, medical leave); H=High (Serious injuries, fatality)

Probability - L=Low (Unlikely); M=Medium (Possible); H=High (Very Likely)

Note: Severity x Probability = Risk (eg. LxL=L; LxM=M; LxH=H; HxM=H; the product follows the higher severity or probability]

Higher Risk requires extensive risk minimization procedures

## **Section 10: Safety issues about hydrofluoric acid (HF<sup>13</sup>)**

### **10.1 Standard Operation Procedure about hydrofluoric acid (HF<sup>13</sup>)**

To transfer the as-grown MoS<sub>2</sub> flakes, the MoS<sub>2</sub>/substrate sample is first spin coated with 0.1 ml poly (methyl methacrylate) (PMMA<sup>12</sup>) for 2 minutes, and after 5-minute heating on the heating plate with 170 °C. Then the sample is soaked in 10 ml hydrofluoric acid (HF<sup>13</sup>) or other proper etching reagents<sup>14-16</sup> in plastic containers for the separation of sample and substrate. Finally, the PMMA<sup>12</sup>-supported MoS<sub>2</sub> was rinsed with 30 ml deionized (DI) water for at least three times to completely remove the etching solution. All the operations above are acted in the fumehood (the HF-related experiments will be done at NFF lab (Room 3234, Technician is Henry Chun Fai Yeung)) in Room 7103. The whole set of protective equipment will be provided by NFF. All the operations will be done under the supervision of the NFF engineer after I pass the safety training assessment of NFF.

### **10.2 Emergency actions in case of HF<sup>13</sup> acid spillage**

#### *10.2.1 Emergency Medical Management for Cutaneous Exposure*

- Immediate removal of contaminated clothing
- Thorough water lavage (15-20 minutes of flushing with a stream of cool water)
- Expeditionary application of a topical treatment
- Oral administration of 6 tables of effervescent calcium gluconate dissolved in water is recommended in case of large burns

#### *10.2.2 Emergency Medical Management for Ocular Exposure*

- Immediate lavage with water for >15 minutes followed by the application of a cold pack
- Irrigation with 1% solution of calcium gluconate when under medical attention
- Several drops of 10% sterile calcium gluconate after irrigation
- Corticosteriod eye drops may be useful
- Consult ophthalmologist

#### *10.2.3 Emergency Medical Management for Inhalation Exposure*

- Removal from contaminated environment
- Closely monitored for difficulty in breathing
- Chest X-ray, arterial blood gases

- Humidified 100% oxygen
- 2.5%-3% calcium gluconate solution (6 ml) by nebulization in attempt to neutralize the fluoride ion
- Monitor electrolytes ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^{+}$ ) and ECG

#### 10.2.4 Emergency Medical Management for Systemic Effects

- Closely monitor fluid and electrolyte levels
- Monitor ECG, renal and hepatic function
- Add calcium gluconate to IV fluid (slowly)
  - a.) Dermal exposure to >50% HF to 1% surface
  - b.) Any dermal exposure of >5% surface
  - c.) An inhalation of vapors from >60% HF
  - d.) Ingestion of HF solutions

### Section 11: Summary of Relevant Hazards and Incompatibilities

No.	Chemical	Relevant Hazards	Incompatibilities
1	Platinum	Massive inhalation of platinum dust may cause irritation	Strong acids and bases
2	Sulfuric acid	Causes severe skin burns and eye damage May cause respiratory irritation	Strong oxidizing agents, combustible material, bases, organic materials, reducing agents, powdered metals, peroxides
3	Gold foil	Harmful if inhaled. May affect the brain or nervous system causing dizziness, headache or nausea. Vapors may cause flash fire or explosion. Harmful if swallowed. Extremely flammable liquid and vapor.	Strong oxidizing agents, strong acids and strong alkalies
4	Mica	Hazardous in case of ingestion, of inhalation. Slightly hazardous in case of skin contact (irritant), of eye contact (irritant)	Strong acids and alkalies
5	Sapphire	May cause mild eye or skin irritation.	Strong oxidizer and acids.
6	Molybdenum trioxide	Toxic if swallowed. Irritating to eyes and	Alkali metals. Strong oxidizing agents.



		respiratory system. Suspected of causing cancer.	Magnesium. Reacts with interhalogens (e.g. bromine pentafluoride). Reacts with hot sodium, potassium or lithium.
7	Sulfur	May form combustible dust concentrations in air; Causes skin irritation; Causes serious eye irritation; May cause respiratory irritation	Strong oxidizing agents
8	Vanadium chloride	Harmful if swallowed. Causes burns by all exposure routes. Reacts violently with water. Target Organs: Respiratory system, gastrointestinal system, eyes, skin.	Water, metals, strong oxidizing agents.
9	Sodium bicarbonate	May be harmful if swallowed	Strong oxidizing agents. Strong acids.
10	Acetone	Vapour and/or liquid may cause moderate eye irritation. May cause moderate skin irritation. May cause respiratory tract irritation. Low toxicity. May irritate upper digestive tract if swallowed.	Strong oxidizing agents (e.g. perchloric acid), strong reducing agents (e.g. hydrides), oxidizing agents (e.g. peroxides), strong acids (e.g. hydrochloric acid)
11	Drikold	Can cause severe frostbite. Vapor can cause rapid suffocation. Vapor can increase respiration and heart rate. Vapor may cause nervous system damage. Vapor may cause dizziness and drowsiness.	Alkali metals, alkaline earth metals, metal acetylides, chromium, titanium above 550 C, uranium above 750 C
12	PMMA	Non-Hazardous	Strong Oxidizers
13	Hydrofluoric acid	May be corrosive to metals. Fatal if swallowed. Fatal in contact with skin. Fatal if inhaled. Causes severe skin burns and	Strong oxidizing agents. Acids. Strong bases. Ammonia. Organic compounds. Glass. Cyanides. Fluorine. Metals. May attack

		eye damage. Causes damage to organs. Causes damage to organs through prolonged or repeated exposure	some plastics, rubber and coatings
14	Sodium hydroxide	May be corrosive to metals Causes severe skin burns and eye damage May cause respiratory irritation	Strong oxidizing agents
15	Ferric chloride	Very hazardous in case of ingestion. Hazardous in case of skin contact (irritant), of eye contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator). Corrosive to eyes and skin	Alkali metals, allyl chloride, ethylene oxide, nylon, styrene, strong oxidizing agents, strong bases
16	Iodine	Harmful in contact with skin or if inhaled	Organic materials, reducing agents, alkali metals, metal powders
17	SiO <sub>2</sub> /Si	May cause cancer by inhalation. Causes damage to lungs through prolonged or repeated exposure by inhalation.	Powerful oxidizers such as fluorine, chlorine trifluoride, and oxygen difluoride and hydrofluoric acid
18	PET foil	Non-toxic and non-hazardous in small quantities	Strong oxidizers
19	Carbon rod	Not toxicological or eco-toxicological	Strong oxidizers such as chlorates, bromates, and nitrates
20	Ethanol	Highly flammable liquid and vapor. Causes serious eye irritation. May cause genetic defects. May cause cancer. Suspected of damaging the unborn child. May be fatal if swallowed and enters airways. May cause damage to organs through prolonged or repeated exposure	Oxidizing materials, acids and alkalis

## Section 12: Waste List

The following is the information of the possible waste generated:

- (1) Spent mixed solution of sulfuric acid<sup>2</sup> and sodium bicarbonate<sup>9</sup>, 400 ml per week, disposed in the waste container of inorganic solution. The pH of the mixed solution is approximately 5 and will be disposed to inorganic acid waste container after proper pH adjustment. The location is on 7/F.
- (2) Spent mixed solution of hydrofluoric acid<sup>13</sup> and sodium hydroxide<sup>14</sup>, 100ml per week, disposed in the waste container of inorganic acid solution after diluting the solution with the concentration lower than 0.5%. The location is on NFF (Room 3234).
- (3) Waste gas of HCl, sulfur vapor and H<sub>2</sub>S, 4L/week, treated with the set-up waste gas treatment system. The location is on the 7101.

## Section 13: Assessment of Significant Risks

- The main hazard associated with the experiments is the use and transport of all the chemicals, some of which are solvents (e.g. HF, NaOH etc). Corrosive solution may cause harm if contacted to the operator and others working in the vicinity. Therefore, care should be exercised when performing the experiments and when transporting and storing chemicals. Suitable protective clothing should be worn by the operator and others working in the vicinity (more on precautions in section 14).

When using it, I will wear a full set of protective equipment to prevent myself from contacting, inhaling and swallowing it. I will two pairs of chemical resistant gloves, safety glasses with side shields (or goggles) and a face shield if necessary when handling it. The whole set of protective equipment will be provided by NFF. All the operations will be done under the supervision of the NFF engineer after I pass the safety training assessment of NFF.

I know the first-aid measures for this chemical well. Immediate medical attention is required. If breathing is difficult, give oxygen. Keep victim warm. Ensure that emergency personnel are aware of the material involved, and take precautions to protect themselves. Some measures can be acted when contacting the chemical with skin: Immediately remove contaminated clothing under a shower. Flush

exposed areas with large quantities of water for five minutes. Wash carefully behind ears, under nails and in skin folds. Get medical attention immediately. For those providing assistance, avoid further skin contact to yourself and others. Wear HF impervious clothing with face shield or goggles and HF impervious gloves. If available, apply calcium gluconate gel (2.5%) into burn area continuously for 15 minutes or until pain relief. For a larger area, use iced Benzalkonium Chloride 0.13% soaks until pain has resolved at least 30-40 minutes. If calcium gluconate gel or Benzalkonium Chloride is not available, continue to wash exposed areas with water until patient is seen by a physician and is taken to a hospital. Insure that contaminated clothing and shoes are properly bagged and discarded. Insure that jewelry is removed and soaked in calcium gluconate solution to decontaminate.

- Fire risks can be minimized by keeping flammable chemicals inside suitable containers at a safe distance from open flames or heat sources.
- Powders and small particles may irritate the respiratory system therefore masks should be worn.
- Open flames or hot/heating apparatus may cause fire or burns related injuries to the operator and others in the vicinity. Therefore, care should be exercised and signs should be placed to warn others.
- Risk of high temperature as the reaction temperature can reach temperatures of 800°C
- Control the flow of carrier gases and gaseous reactants (H<sub>2</sub>, Ar) carefully and thoroughly check for the gas delivering system connection. Ensure all unreacted waste gases are exhausted through fume hood.

## **Section 14: Safety Precautions**

### **Personal protective equipment**

- Laboratory coat, safety goggles, and nitrile/polyvinyl alcohol gloves because of the presence of alkali such as potassium hydroxide (from Instant Glove + CPC Database) as the outer layer with rubber gloves as the inner layer must be worn at all times when performing the experiments, transporting, handling and cleaning chemicals and equipment and clearing chemical spills.
- Dust mask should be worn when collecting and weighing solid particles.
- Heat resistant gloves will be worn when handling hot items.

### **Fume cupboard**

- Experiments that release waste gases and those that use solvents will be conducted inside a fume cupboard with the extraction system on.
- Leak testing will be conducted before performing experiments where gases are involved.

### **Training**

- The researcher must attend regular courses (and in some courses obtain a passing result) offered by the Safety and Environmental Office of the University (HKUST) and understand thoroughly the safety concepts of these courses and be able to apply them before being allowed to conduct any experiments or use any of the equipment associated.
- The researcher must understand and be aware of all the procedures for handling, and in some cases dealing, with all possible emergency situations and scenarios.
- The researcher must undergo training in handling equipment and conducting experiments competently before performing it on his/her own.
- Equipment training offered by technical staff for nitrogen adsorption (Omnisorp 100CX), ICP (Perkin Elmer Optima), Elemental Analyser (Vario ELII Eliminator), TGA (Shimadzu TGA-50) are required before conducting the tests.
- XRD, XRF, XPS, TEM, and FTIR will be conducted by the technical staff of the MCPF.

### **Other Precautions**

- All chemicals, samples, and reagents should be stored in appropriate containers with clear labels specifying the name, type, date and any warning signs. These should be checked and monitored on a regular basis. Instructions on labels of chemicals should be followed.
- Good house-keeping should be observed.
- Place everything where they should be when finished using them.
- Passage ways and exits should be kept clear and unobstructed.
- Everyone is responsible for the safety of their neighbors and others working in the vicinity.
- All laboratory rules should be obeyed.
- Warn others of hot/heating equipment and put warning signs if necessary.
- Check that the keys to the main valve of gas cylinders are connected to the cylinder valve at all times so that the cylinder can be shut off instantly in case of an emergency.
- Pipes and fittings should be checked at regular intervals

**Emergency shut down procedure for reactor (catalytic activity measurements)**

1. Switch off power supply to the reactor/furnace
2. Close all valves in the direction that feeds the reactor i.e. allow all the gases to be vented out
3. Turn off all supply of gases to the reactor

**Emergency shut down procedure for the volumetric adsorption rig (adsorption measurements)**

1. Switch off power supply to the vacuum pump, MKS transducer and thermocouple.
2. Close all valves

**Section 15: Action in Case of Abnormal or Emergency Situations**

The following emergency procedures are based on references to the HKUST emergency procedures issued by the safety and environmental protection office ([www.ab.ust.hk/sepo/emergency.htm](http://www.ab.ust.hk/sepo/emergency.htm)).

**Service failure**

- Perform emergency shut down procedures described in the above section.
- Inform the safety officer/appropriate personnel of the failure.
- If necessary, evacuate everyone in the vicinity.

**Hazardous chemical spill in research laboratory**

- Alert co-workers
- If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.
- Ask for assistance if necessary.
- Press the emergency ventilation button (do not activate this button in case of fire).
- Inform the security office at x8999 or 23588999 with mobile phone when it is safe to do so.
- Evacuate everyone in the affected area. Leave contaminated clothing and close the door.
- Activate local warning system to prevent others from entering the room.

- If possible, maintain a safe distance from the scene, keep the entrance or access routes in sight and help to prevent entry to the affected room.
- If conditions allow, remain to assist the emergency response team.

#### **Contact with chemicals**

- Flush eyes, skin and/or area of contact with chemical with tap water for at least 15 minutes and remove any contaminated clothing. If necessary, use the nearest emergency shower for further cleaning. Contact and consult the safety officer or dial 8999.

#### **Gas leakage**

- Follow emergency shut down procedures outlined in the previous sections. Ventilate and evacuate the vicinity (if necessary). Contact and inform the safety officer.

#### **When the fire alarm is heard**

- Check if there is any sign of fire in the vicinity.
- If there is fire or smoke, or there is an announcement to evacuate, then evacuate to the assembly point as far as practicable and report to the fire & safety officer.
- If there is no sign of a fire, stay alert and pay attention to announcement until the fire alarm is silenced.
- Evacuate if the alarm has sounded for more than two minutes.
- If the buzzer sound which indicates fire alarm is activated in an adjacent fire zone is heard, stay alert and pay attention to announcement.
- If both the buzzer and the fire alarm are heard, treat as if the fire alarm is heard.

#### **If a fire is discovered**

- Perform emergency shut down procedures if possible.
- Activate the fire alarm by pressing the breakglass fire alarm button.
- Report to Security Control Centre by dialing 8999.
- Alert other people. If safe to do so, try to put out the fire by firefighting equipment.
- Do not take any personal risk. If the fire gets beyond control, evacuate immediately as listed above.
- Close the door of the room on fire.

### **Firefighting equipment**

- Water from the hose reels is good for wood and paper fire, structural fire, but not for oil, electrical or metal fire.
- The most common fire extinguishers on campus is the carbon dioxide type (black container) which are good for general purposes, including oil and electrical fire.
- Some laboratories have dry powder fire extinguishers (blue container), which are good for chemical fire, including metal fire.
- Sand can be used to contain flammable liquid as well as put out a fire, including metal fire.
- Fire blanket can be used when someone's clothing catches fire.

### **Evacuation procedures**

- Remain calm. Walk; do not run, especially when travelling on staircases.
- Immediately leave the building and go to the assembly point using the nearest exit.
- Try to help those who may have difficulties traveling such as disabled and pregnant persons.
- Do not use the lifts.
- Report to your Fire & Safety Officer at the assembly point as far as practicable.
- Do not return to the building until permission is given by the Fire Services Department Officer in charge of the scene.

### **When clothing is on fire**

- Do not run
- Drop to the floor and roll to extinguish the fire.
- If fire blanket is available, wrap around body to help smother the fire.

### **When someone is injured or ill**

- Call Security Control Centre (SCC) by dialing 8999.
- Call for Community Emergency Service directly by dialing (9)999 if the situation is urgent or serious and inform SCC subsequently.
- Do not conduct rescue operation unless it is safe and the proper procedures are used. Careless rescue operation may endanger the rescuers when, for example, the



victim is inside a room filled with toxic gas, or is still in contact with live electricity.

- Do not move an injured person, especially when there are signs of spinal injury or bone fracture, unless it is absolutely necessary to do so for safety reason.
- Keep the injured or ill person comfortable, warm, and lying down.
- Give First aid treatment if necessary.
  - Acid and alkali burns--flush with running water; use emergency shower if necessary. Do not attempt to neutralize.
  - Heat or cold burns--flush with cold water.
  - Chemical in eyes--flush eyes with emergency eyewash.
  - Major bleeding--apply direct pressure to the wound using a clean cloth.
  - Toxic gas inhalation--expose to fresh air.
  - Hydrofluoric acid exposure--use antidote immediately
  - Cyanide exposure--use antidote immediately

#### **If trapped in lift**

- Remain calm
- Press the alarm button in the lift
- Communicate through the intercom unit which connects directly to Security Control Centre.
- Never try to force open the lift door or get out through the manhole at the ceiling of the lift car. Such attempts may result in fatal accidents.
- Be patient and wait for help.

#### **Damaged glassware or equipment**

- Safety officers/appropriate personnel should be informed for further instructions, warning signs must be posted to inform others, and safety precautions must be taken if glassware is present. If chemicals are released, then the above procedures should be followed.

**In all the above mentioned actions in case of abnormal or emergency situations, everyone should be evacuated and the safety officer should be informed or dial 8999 as soon as it is safe to do so if the accident or situation is deemed uncontrollable or severe. When phoning 8999 or any other emergency number, the person should inform the authorities of the name of the person, the location and the nature of the emergency.**

## **Appendix**

The Material Safety Data Sheets for the chemicals listed in section 8 are attached at the end of this work plan; these are obtained from the MSDS databases provided by the HKUST (Sigma-Aldrich and CCOHS).