Industrial Applications at NSRRC
-Taiwan’s Experiences

Bryan, Shew*, CM Chou, PJ Wu, SW Chen, CW Hu, Shin-An Chen, YH Lin, KY Pan, and MT Tang,
Industry Application Division (IAD), NSRRC
1. In NSRRC, the Industry Application Division (IAD) is the acting window to perform industry application research.

2. In order to maximize the impact to the society, industrial applications are highly cooperated with domestic major industries and key companies in the first stage.

3. IAD focus using advanced SR techniques to solve bottle-neck problems in various industries, and then boost added-up values for genuine industrial concerns.

• **Motivation and Strategy**

• **Industry Cluster @ Taiwan**

Source: Taiwan External Trade Development Council
[1] SEMICONDUCTOR

• Background

Semiconductor is the key industry of Taiwan. By 2022, the output value has exceeded USD 162 billion (Top 3 in the world, about 13% GDP)

TSMC is the No.1 semiconductor foundry in the world, and is just next to NSRRC.

How we work with semiconductor companies?

• Use advanced SR tech. to analyze the buried, ultra-thin films and interfaces in the transistor with high accuracy.

• Based on the geographic advantage, a “tsmc virtual lab” project was performed since 2014 to facilitate the interactions between two sides.

• Major applied techniques: in-plane XRD, XAS, HAXPES.
X-ray Photoelectron Spectroscopy (XPS)

- XPS spectra are obtained by irradiating a material with X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 10 nm of the material being analyzed.
- XPS is a **surface-sensitive** quantitative spectroscopic technique that measures the elemental composition, chemical state and electronic state of the elements that exist within a material.

  - **Website:** [http://www.rowbo.info/XPS.html](http://www.rowbo.info/XPS.html)

- To analyze the underlayer material, **chemical etching** and **Ar sputtering** are always used to remove the cover layer.
- The use of Ar sputtering introduces artifacts such as ion mixing and differential sputtering, then might get a wrong results.
Outcome

• In the first-year project, we successfully use SR techniques to solve a critical current leakage problems of high-k material in the nano chip, and then help to reduce the power consumption of the microprocessor.

• The applied beamtime of TSMC raise up from 140 hours (1st year) to about 1000 hours (2nd year~). TSMC is now the biggest industrial user!

• We are conducting 10th year project.

Other Applications…

- SR EUV for Researches of EUV Lithography
  - from TSMC

- CD-SAXS for in-line, non-destructive metrology of nano devices
  - JDP of ITRI +Hermes +TSMC

- NC-FET for ultra-low power consumption micro processor.
  - JDP of ITRI +TSMC
Three major Li-ion battery companies in Taiwan have used SR techniques to develop advanced Li-ion batteries for power battery, solid-state battery and energy storage applications.

XRD, XAS, X-ray imaging techniques are most-used techniques.
Analytical Challenges in Battery Science

**Elements**

- **Surface/Interface**
  - SEI, CEI, Surface Phase
  - Impedance
  - Cycling behavior
  - Reversibility
  - Rate capability
  - Safety

- **Bulk Materials**
  - Electronic and Crystal Structure
  - Material design
  - Cycling parameters
  - Conductivity
  - Quality control

- **Electrodes**
  - Phase Distributions
  - Reaction mechanisms
  - Cycling behaviors
  - C-rate performance
  - Failure analysis
  - Safety
  - Electrode and cell design

- **Devices**
  - Reactions
  - Cost reduction
  - “Green” chemistry

**Needed**

- Surface sensitive techniques, Depth profiling
- Bulk techniques, *ex-situ, in-situ operando* experiments
- Imaging, *ex-situ, in-situ operando* experiments
- Bulk techniques

**Various length scales**

- Several Å, Several nm
- Tens of nm~100 μm
- μm~mm
- >mm

**Materials Synthesis/Recycling**
• **MoliCEL batteries** (能元科技) is now the biggest Li-ion battery company in Taiwan. They used advanced SR quick XAS technique @TPS to develop advanced power Li-ion battery.

• The Goodwood Festival of Speed is the most important annual international racing festival in UK. At 2022, the pure electric racing car **Spéirling** using MoliCEL batteries surpassed hundreds of F1 cars, won the championship and broke the historical record.
A biggest petrochemical enterprise in Taiwan attempt to produce high-strength carbon fiber because of its potential applications in electrical car, off-shore wind power, aircraft and space industries.

High brilliance, micro-beam SR SAXS was applied to characterize nano/micro structure of a single carbon fiber.

SAXS/WAXS analysis yield across-scale structure information of a single carbon fiber.
Micro-Beam SAXS Experimental Setup

Figure 1. AI-assisted Image Recognition for Synchrotron $\mu$-Beam SAXS on Single Polymer Fiber. Left: $\mu$-beam SAXS experimental setup in the NSRRC Taiwan Photon Source (TPS) beamline; Right: SAXS patterns of the 2D grid scan of a single polymer fiber.
• Using SR SAXS/WAXS techniques, a key structure code which is highly related to its mechanical properties was found, and the tensile strength of their carbon fiber was successively improved to about 6.5 GPa.

• At 2022, the company invested to establish a new production line to produce high-strength carbon fiber.
• Net-zero carbon emissions by 2050 has become the consensus of major countries, and it is also a requirement of international trade.

• Traditionally, steelmaking uses **coke** to reduce iron ore to produce molten iron, which produces a large amount of **carbon dioxide**.

• In order to significantly reduce carbon emissions, the key Taiwan’s steel company invested to develop new **hydrogen metallurgy** technology.

• In this project, high-temperature, in-situ SR technologies including XRD, XAS, X-rat imaging, will be used to understand the kinetic mechanism of hydrogen reduction iron ore, and then develop clean steelmaking technology.

**[4] STEEL INDUSTRY – Hydrogen Metallurgy**

Hydrogen replaces coke to reduces CO₂ emission!
High-Temperature (max. 1650°C), Environment-Controlled Furnace
- for in-situ SR X-ray analysis
Research Map for Hydrogen Metallurgy

(1) Initial Slag
- Lump,
- Sinter Pellet,
- HBI

(2) Medium Slag
- Structure (phase fraction)
- Valence (reduction)
- Morphology (melting, interfacial interaction)

(3) Final Slag
- Reduction Mechanism/Kinetics
- Thermodynamics

Characteristics
- 25°C
- 900°C
- 1200°C
- 1600°C

Fe₂O₃ → Fe₃O₄ → FeO → Fe

Softening
Melting
Dropping

\[ \ln \left( \frac{dx}{dt} \right) = - \frac{E_a}{R \times T} + \ln(A) \]
(A). About Spectrometer ..... 

- Spectrometry is a universal analytical platform since the absorption spectrum of a species is unique, just like the fingerprint.
- Spectrometer can be used in analysis of color, pollution, food safety, bio medicals, and optical communication.

(B). Why “miniaturized” Spectrochip?

- Low cost, portable, integratable!
- The spectrometer chip can couple with mobile phone and internet (AIOT), to do in-situ analysis of food-safety and biomedical analysis. (healthcare!)
Why Concave Grating?

Planar Grating Scheme

Concave Grating Scheme

- Deep X-ray Lithography

**Nano-Micro resolution**

**Perpendicular sidewall**

**Excellent surface quality (R\(_a\)~nm)**

**High aspect ratio (>50)**

**Fabrication tech.: DXL, DRIE**

- schematic diagram of the concave-grating spectrometer
From Optical Chips to Devices

- **X-ray Micro Fabrication/Integration/Assembly**

- **Optical Performance**

  - **Before Optimization**
  
  - **After Optimization**

- **Measured spectrum of a green laser source from the spectrochip**

Source: https://www.spectrochips.com/
**Spectrochip for COVID-19 Antibody Detection**


- All COVID-19 rapid test strips used colloidal gold-conjugated antibodies to detect human IgG and IgM antibodies.
- The IgG and IgM lines of the test strip captured these complexes, upon which colored bands would be presented.
- IgM is helpful for detecting recent infection. In contrast, the presence of IgG antibodies often indicates a past infection as it generally does not appear until 7 to 10 days.

![Image of Spectrochip](image.png)

Fig 3. The workflow of quantitative spectral LFIA platform. The optical-based platform applies 10–20 uL of blood from a fingertip or a vein to the test strip, integrating the immunoglobulins and producing results in 3–10 minutes.
Summaries and Future Works

- After a decade’s promotion, a wide spectrum of industrial applications based on SR analysis techniques has been flourishing in Taiwan. These advanced SR techniques not only help solve critical industrial problems, raise up their global compatibility, but also help industries earn considerable revenue.

- To this extension, a new beamline dedicated for industrial application at TPS is under construction. This automated, multi-purpose beamline is designed for XRD (powder/in-plane), XAS, and WAXS. In particular, the artificial intelligence (AI) technology will be embedded into the facility to enable efficient beam alignment, data retrieval and analysis. This dedicated industry beamline is scheduled to be completed in 2027, at which enriching research capacity for industrial innovations are created.
Synchrotron for Industry!
Light up Your New Vision!

Industry Application Division (IAD), NSRRC
Division Head
Dr. MT Tam

Project Manager
• Semiconductor (Dr. PJ Wu)
• Advanced polymer & carbon fiber (Dr. CM Chou)
• Li-ion Battery (Dr. CW Hu)
• Steel (Dr. SW Chen)
• Pharmaceutics (Dr. YH Lin)
• Micro devices (Dr. BY Shew)