

Equipped with state-of-the-art facilities, the Battery Research Platform was established with funding from the FY2012 supplementary budget, with the aim of accelerating Japanese R&D focused on next-generation batteries. As the core host institution of the Battery Research Platform, NIMS (National Institute for Materials Science) is working with AIST Kansai (National Institute of Advanced Industrial Science and Technology) and WASEDA University on ALCA-SPRING (Advanced Low Carbon Technology Research and Development Program, Specially Promoted Research for Innovative Next Generation Batteries), providing priority support for this project's R&D focused on next-generation batteries.

It also supports other next-generation battery R&D initiatives being carried out by universities, national institutes and companies throughout Japan that are also affiliated to this project.

Managing Director Kohei UOSAKI (Director-General of NIMS GREEN)

# To contribute to the research and development of next-generation batteries



### NIMS Battery Research Platform

The NIMS Battery Research Platform is a world-class facility for next-generation battery R&D covering everything from battery cell assembly to materials analysis. All the equipment is installed under one roof in the NanoGREEN Building at the Namiki site.

To provide the ultra-low humidity environment necessary for battery research, a super-dry room with an area of 80 m<sup>2</sup> was constructed. In this room, you can freely assemble and disassemble battery cells, and analyze them using FTIR, Raman, compact SEM, laser microscope, etc.

The state-of-the-art equipment includes TEM, SEM, FIB-SEM, TOF-SIMS, XPS, HAXPES, AES, SPM, XRD, LC/MS, GC/MS, and ICP/MS. Airtight transfer vessels are available to prevent samples from being exposed to air.

NIMS Battery Research Platform Director Shoji YAMAGUCHI

### 501 Super-Dry Room

- 1. Super-Dry Room
- 2-1. Cell Assembly Equipment
- FTIR 3.
- 4. Laser Raman Microscope
- 5. Ellipsometer
- 6. Gas Transmission-rate Measuring Equipment
- 7. TG/MS
- 8. Compact SEM
- 9. Laser Microscope
- 10. Viscometer
- 11. Battery Charge/Discharge System
- 12. Precision Electrochemical Measurement System
- 13. Single-Particle Measurement System

### 503 Clean Room

- 2-2. Cell Assembly Equipment
- 14. ICP/MS
- 15. LC/MS
- 16. Ion Chromatography

# 505 Laboratory

- 21. XRF
- 22. XPS
- 23. HAXPES
- 24. XRD
- 25. BET
- 26. Particle Size Analyzer

### 206-1 Laboratory

27. Environment-controlled SPM

### Laboratory

28. TOF-SIMS

### 5 Laboratory

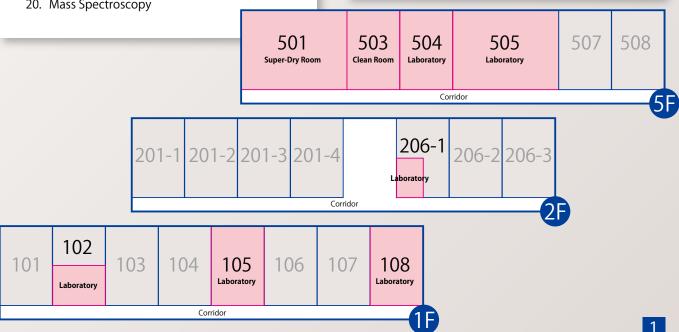
- 29. TEM/STEM
- 30. FIB

### 504 Laboratory

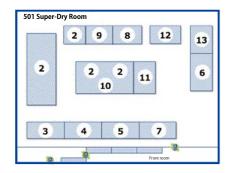
- 2-3. Cell Assembly Equipment
- 17. O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub> analyzer
- 18. GD-OES
- 19. GC/MS
- 20. Mass Spectroscopy

### 108 Laboratory

- 31. FIB-SEM
- 32. SEM
- 33. Scanning Auger Electron Spectroscopy
- 34. Cross-section polisher



501 Super-Dry Room	503 Clean Room	504 Laboratory	505 Laboratory	507	508	
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### 501 Laboratory

### 1. Super-Dry Room

[Maker] Shinryo corporation [Model] Specially ordered [Use for] Battery cell assembly, evaluation of battery properties, analysis of battery materials. [Outline] The world's highest level of dry air is supplied with a dew point of < -90°C

(Water Content < 0.1 ppm). The wide floor space of 80 m<sup>2</sup> can accommodate 5 people working.



#### 2-1. Cell Assembly Equipment



### 4. Laser Raman Microscope

[Maker] Nanophoton [Model] RamanTouch-VIS-NIR [Use for] Identification of chemical bondings, crystallinity and/or

deformation of crystal lattice. [Outline] Information about molecular structure and crystalline

structure of materials can be measured by analyzing the scattered light, which has a different wavelength from the

incident light wavelength (Raman scattering). Spatial resolution (X axis) is 350 nm Laser wavelengths are 488 nm, 532 nm (VIS) and 785 nm (NIR)



### 5. Ellipsometer

[Maker] J.A. WooLLAM [Model] M-2000X [Use for] The primary application of ellipsometry is to characterize film thickness, optical constants, band gap, alloy composition and phase structure.

[Outline] Ellipsometry measures a change in polarization as light reflects or transmits from a material structure. Ellipsometry is primarily used to determine film thickness and optical constants. However, it is also applied to characterize composition, crystallinity, roughness, doping concentration, and other material properties associated with a change in optical response. RCE (rotating compensator ellipsometer) technology to achieve high accuracy and precision, has an amplitude ratio ( $\Psi$ ) of 0 – 90°, and a phase difference ( $\Delta$ ) of 0 – 360° with advanced CCD detection to measure all wavelengths simultaneously.

### 3. FTIR

[Maker] Thermo Scientific [Model] Nicolet iS50 [Use for] Material characterization for qualitative and quantitative analysis [Outline] The molecular structure is defined by measuring the absorption of the infrared radiations. Wavenumber resolution : 0.09 cm<sup>-1</sup>. Scan speed : High scan rate(130 spectra/sec)



### 6. Transmission-rate Measuring Equipment

(Maker) GL Sciences

[Model] GTME 2520

[Use for] Gas transmission-rate measurement for films and materials.

[Outline] Gas transmission-rate measuring equipment with gas chromatograph based on JIS K 7126-2 (Determination of gas-transmission rate -- Part 2: Equal-pressure method)

Three types of detectors are installed, TCD, FID, and PDD.



### 7.TG/MS

[Maker] TA Instruments [Model] Discovery [Use for] Thermogravimetry (TG) of samples and mass spectrometry (MS) of outgases. [Outline] The combination of TG and MS enables simultaneous analysis of the sample weight and the outgases.



### 8. Compact SEM

[Maker] Agilent Technologies [Model] 8500

[Use for] Observation of the surface profile and structure. [Outline] Compact FE-SEM settled in the dry room (resolution of 10 nm).

Accelerating voltage is tunable (0.5 – 2 kV). Measurable without conductive pre-treatment.

BEI (backscattered electron image) is possible.



### 9. Laser Microscope

[Maker] KEYENCE

[Model] VK-X200

[Use for] Observation of the 3D profile and quantitative feature analysis.

[Outline] 3D laser scanning confocal microscope system performs nanometer level profile, roughness and thickness measurements on nearly any material.



### 10. Viscometer

[Maker] AntonPaar

[Model] Lovis2000ME [Use for] Combined measurement of de

[Use for] Combined measurement of density, dynamic viscosity and kinematic viscosity of liquids.

[Outline] Lovis 2000 M/ME is a rolling-ball viscometer which measures the rolling time of a ball through transparent and opaque liquids according to Höppler's principle. Sample volume is 0.1 mL to 0.8 mL. Measurable dynamic viscosity is up to 10,000

 $mPa \cdot s$  with heating from 5 to 100°C. Test duration is a minimum of 30 seconds for 1 measurement.



### 11. Battery Charge/Discharge System

[Maker] sai, Espec

[Model] 580 (16ch) + thermostatic chamber

[Use for] Evaluation of battery characteristics [Outline] 5 terminal measurement is possible. Voltage/current measurements of the cathode and anode are possible with 16 channels. The impedance measurement is possible at 1 kHz. The temperature is also controlled by PC.



#### **12.** Precision Electrochemical Measurement System

[Maker] Solartron, BioLogic

[Model] 12608W (1287 Electrochemical Interface + 1260 Electrochemical Interface), SP-200, VSP-300 (BioLogic Science Instruments)

[Use for] AC impedance and DC polarization measurements. [Outline] 12608W manufactured by Solartron Analytical allows automatic measurement of AC impedance and DC Polarization. SP-200 is a portable potentiostat/galvanostat. VSP-300 is a modular-unit potentiostat/galvanostat with 6 channels.



### 13. Single-Particle Measurement System

[Maker] KEYENCE, BioLogic Science Instruments, Micro Support Co.,Ltd

[Model] Combinations

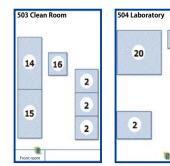
[Use for] Characterization of a single particle of active material. [Outline] A battery electrode is usually a composite of active materials, binders, and conducting agents. This system is used for characterization of individual active material particles in a battery electrode. The system consists of an optical microscope

with high-resolution view, a micromanipulator, and a potentiostat / galvanostat / FRA for electrochemical measurements.



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505 Laboratory 507 508



### 503 Laboratory

### 14. ICP/MS

[Maker] Agilent Technologies [Model] Agilent 7700 ICP-MS [Use for] Qualitative and quantitative analysis of trace elements. [Outline] Using Inductively Coupled Plasma (ICP) as an excitation source,



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this mass analyzer (MS) with a high-frequency quadrupole provides superior ion transmission, resolution and abundance sensitivity and can measure a wide range of elements from Li to U, in a wide range of concentrations from ppt to ppm. The isotope ratio is also identifiable. A laser abrasion unit is also available and applicable for solid samples.

### 15. LC/MS

[Maker] Waters Corp. [Model] Xevo G2-S QTof [Use for] Qualitative and quantitative analysis of solutions. [Outline] Xevo G2-S QTof uses high



performance liquid chromatography combined with an ion detection system of quadrupole mass spectrometry and time-of-flight mass spectrometry (UPLC/MS/MS). It is mainly used for component and quantitative analysis of electrolytes.

## 504 Laboratory

### 17. O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub> analyzer

#### [Maker] HORIBA

[Model] EMGA [Use for] Quantitative analysis of Oxygen, Nitrogen and Hydrogen. [Outline] EMGA-930 is a simultaneous oxygen/nitrogen/hydrogen elemental analyzer with high accuracy and repeatability. The sample is rapidly



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heated up above the melting point in a He atmosphere, and the outgases are analyzed.

### 18. GD-OES

[Maker] HORIBA [Model] GD-Profiler 2 [Use for] Surface and depth analysis of the sample. [Outline] GD-OES allows the analysis



of elemental depth profiles by optical detection of emissions from the excited atoms after Ar plasma sputtering. Low damage and high resolution measurements can be realized by pulsed sputtering. Surface analysis of electrically non-conductive samples is possible by high frequency glow discharge. Bulk quantification of solid materials is also possible.

### 16. Ion Chromatograp

[Maker] Dionex [Model] ICS-2100 [Use for] Quantitative analysis of anions and cations. [Outline] The ICS-2100 system is the



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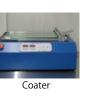
first Reagent-Free<sup>™</sup> ion chromatography system with electrolytic sample preparation (RFIC-ESP<sup>™</sup> system) and eluent generation (RFIC-EG<sup>™</sup> system) capabilities designed to perform all types of electrolytically generated isocratic and gradient IC separations using conductivity detection. Microbore 2 mm columns and standard bore 4 mm columns are fully supported.

#### 2-2. Cell Assembly Equipment









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#### 2-3. Cell Assembly Equipment

Glove Box



### 19. GC/MS

[Maker] Agilent Technologies [Model] Agilent 5977A GC/MS [Use for] Qualitative and quantitative analysis of gases and liquids such as electrolytes.



[Outline] Qualitative analysis

and quasi quantitative analysis is possible simultaneously by splitting the eluted spices from GC-column into MSD and FID. An El source and CI source are available. Simultaneous SIM/Scan operation is possible.

### 20. Mass Spectroscopy

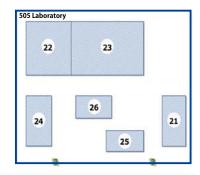
[Maker] JEOL [Model] JMS-700 [Use for] Identification of chemical species such as gases from a battery cell.

[Outline] The use of a 10 kV ion source in combination with a post acceleration conversion dynode-based ion detector allows

for effective detection of either positive or negative ions. A resolution exceeding 60,000 can easily be obtained.



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### 21. XRF

[Maker] SHIMADZU

[Model] LAB CENTER XRF-1800

[Use for] Quantitative analysis of elemental composition [Outline] This system is capable of elemental analysis (except for H, He, Li, Be) with a 250  $\mu$  m scale by detecting the characteristic fluorescent X-rays emitted from each element within samples by X-ray irradiation. It enables to determine the elemental composition and obtain the elemental mapping of the sample surfaces.

### 22. XPS

[Maker] ULVAC-PHI

[Model] PHI 5000 VersaProbe II

[Use for] Surface-sensitive qualitative and quantitative analysis of elemental composition and oxidation states of surface species. [Outline] This system is capable of determination of the elemental composition and oxidation state of surface species by analyzing photoelectrons emitted by X-ray excitation. A monomer Ar ion gun and gas cluster ion gun (GCIB) are

available for depth-resolved analysis. Samples can be introduced from an inert environment into the vacuum chamber by transfer vessel.



### 23. HAXPES

[Maker] Omicron NanoTechnology & ULVAC-PHI [Model] Custom Engineered Product [Use for] Photoelectron spectroscopy excited by hard X-rays. [Outline] This system is capable of elemental analysis of surface species by analyzing photoelectrons emitted by hard X-ray excitation. This system is equipped with a monochromatic micro-focused Cr K $\alpha$ source. It enables chemical analysis of elements in a deeper region (> 10 nm) than conventional XPS. The system is connected to an Ar-filled glove box to avoid moisture / air exposure of samples during transfer.



### 24. XRD

(Maker) Rigaku

[Model] SmartLab<sup>®</sup> [Use for] Analysis of powder, thin film and SAXS

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[Outline] SmartLab is equipped with a high luminance rotating anode generator, an optional in-plane diffraction arm for

in-plane measurements without reconfiguration and focusing / parallel beam geometries. A high-temperature heating stage to 1100°C in inert gas is available.



### 25. BET

[Maker] Micromeritics [Model] 3FLEX

[Use for] Measurement of surface area and pore volume for powder samples.

[Outline] A fully automated, three-station instrument with dedicated pressure transducer provides superior mesopore/ micropore, physisorption,

or chemisorption analysis.



### 26. Particle Size Analyzer

[Maker] HORIBA [Model] LA-950V2 MF

[Use for] Particle size distribution analysis

[Outline] The system is equipped with a dual-wavelength optical system, two light sources of 650 nm (Laser) and 405 nm (LED). The latest theories for data analysis and newly developed calculation algorithms provide stable high-quality analysis performance for all particle diameters.



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### 206-1 Laboratory

### 27. Environment-controlled SPM

[Maker] Bruker AXS

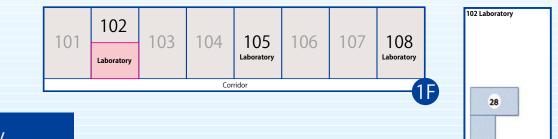
[Model] MultiMode 8 + Glove Box

[Use for] Topography (surface profile), nanomechanical property, quantitative conductivity mapping

[Outline] The MultiMode is the world's most field-proven atomic force microscope (AFM), with a turnkey glove box which can control an environment in which both water and oxygen can be maintained at < 1 ppm. Using this AFM, the surface conditions of the electrode can be evaluated without exposure damage.







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### 28. TOF-SIMS

[Maker] ION-TOF GmbH

[Model] TOF.SIMS5-AD-GCIB [Use for] Detection and identification of trace elements/compounds on the surface, depth profiling, and imaging with the sub-micron lateral resolution [Outline] High sensitivity can be realized by mass spectrometry of secondary ions emitted by ion sputtering. Information on the molecular structure of organic compounds can be obtained under the static SIMS condition. Molecular depth profiling is possible with lower damage by using an Ar gas cluster ion beam (GCIB).



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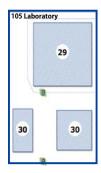
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#### [Maker] JEOL

[Model] JEM-ARM200F (HR) [Use for] TEM/STEM observation, EELS analysis, EDX analysis [Outline] The JEM-ARM200F, with a STEM Cs corrector incorporated as standard, and the mechanical and electrical stability enhanced to the utmost limit, achieves an unprecedented STEM-HAADF resolution of 78 pm at 200 kV.

The cold FEG enables high energy resolution EELS analysis.



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JEOL JIB-4501

### 30. FIB

[Maker] JEOL [Model] JIB-4501

[Use for] Fine milling and TEM thin-film sample preparation. [Outline] The JIB-4601F is a multibeam processing system with a thermionic SEM and a high-performance ion column. The high-power FIB column enables fast milling without any deterioration of the sample using the cryo-stage. The prepared TEM thin-film sample can be mounted to a mesh grid in the glove-box without exposure to ambient air. 1

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## **108** Laboratory

#### **31. FIB-SEM**

[Maker] Hitachi High-Tech Science

[Model] SMF2000

[Use for] FIB processing, SEM/STEM observation, 3D image processing, EDS analysis, sample preparation for SEM/TEM.

[Outline] The ion source of FIB is set perpendicular to the electron source of SEM.

The specimen after FIB processing can be observed by SEM without moving the stage and 3D image acquired automatically. Low damage FIB processing is possible with a cryo-stage. Processing without air exposure is possible.

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### 32. SEM

[Maker] JEOL

[Model] JSM-7800F

[Use for] SEM observation, EDS/WDS/EBSD measurement

[Outline] High resolution observation (0.8 nm @15 kV, 1.2 nm @1 kV) is possible with low acceleration voltage (0.01-30 kV). Soft x-ray emission from lithium can be detected using a newly developed WDS. Sample transfer without air exposure is possible.



#### [Maker] ULVAC-PHI

[Model] PHI710

[Use for] Analysis of the elemental composition and chemical bonding state of the solid sample surface

[Outline] This system is capable of analyzing the elemental composition and chemical bonding state by irradiating a sample surface with a focused electron beam and analyzing the kinetic energy of the Auger electrons emitted from the surface. The analysis at high spatial resolution up to 8 nm and high magnification up to 500,000 is available without a sample drift. Samples can be introduced into the analysis chamber from an Ar-filled glove box and/or a dry room via transfer vessel without moisture/air exposure.

### 34. Cross-section polisher

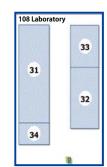
[Maker] JEOL

[Model] IB-09020CP

[Use for] Preparation for pristine cross sections of a specimen for SEM and AES. [Outline] The cross-section polisher is a device used to prepare pristine cross sections of a specimen using an ion beam and a shielding plate. The processing condition can be confirmed by monitoring the CCD camera in real time. A cooling system for the specimen stage is available. Non-atmospheric exposure analysis is possible with the transfer-vessel.

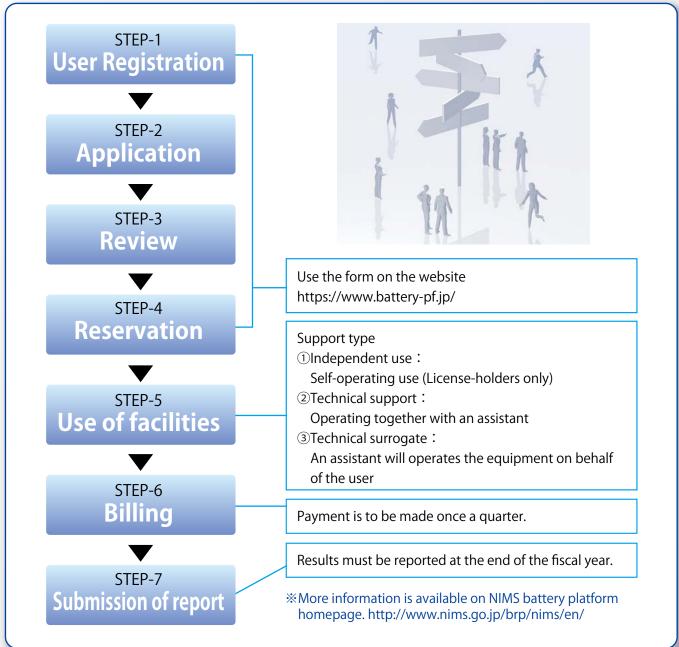








### Application process



### **Related organizations**

### AIST Kansai

#### **Evaluation of medium-sized batteries**

[Nondestructive internal observation apparatus for medium-sized battery] •Industrial X-ray CT scanner

[Properties analysis apparatus for medium-sized battery] • Laser Raman microscope



ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY (AIST)

National Institute of Advanced Industrial Science and Technology

#### AIST Battery Research Platform

Calvet calorimeter

Thermal analyzers

1-8-31 Midorigaoka, Ikeda, Osaka 563-8577, Japan TEL:+81-72-751-9550, FAX:+81-72-751-9629 https://unit.aist.go.jp/riecen/

### WASEDA University

#### Electrochemical impedance spectroscopy

[Impedance measurement apparatus for condition analysis of battery]

Properties evaluation by designing and analysis of equivalent circuit based on measured results of electrochemical impedance of battery and electrode

#### WASEDA University

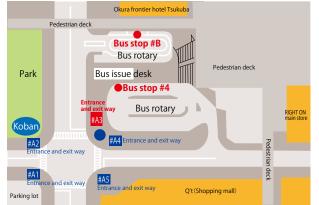


WASEDA Battery Research Platform Smart Energy System Innovation Center (SEnSIC) Waseda University Research Organization for Nano & Life Innovation

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# Access from Tsukuba Station NIMS Shuttle Bus / Route Bus



NIMS shuttle bus

Get out to the Tsukuba Center Bus Terminal from the station through Exit #A3. Take the NIMS shuttle bus at "Bus stop #B".

Kanto-railway bus

Get out to the Tsukuba Center Bus Terminal from the station through Exit #A3. Take an Arakawaoki-bound local bus at the bus stop #4.

Get off at "Busshitsu Zairyo Kenkyu Kiko". (7 minutes ride and 1 minute walk)



### NIMS Battery Research Platform Secretariat

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NanoGREEN/WPI-MANA Bldg.