### **Equipment for Collaborative Research in 2023**

#### 1. Materials Synthesis Station

#### 1-1 Electron Beam Lithography & Ion Milling System

Building No. 2, Room 211

Microfabrication at a sub-micron scale can be performed by a 50-kV thermionic electron gun to obtain micro-sized structures of approximately 100 nm. Photolithography in combination with electron beam lithography equipment using a photomask aligner with large-area processing can be used to fabricate devices. An attached secondary ion mass spectrometer enables depth profile measurement using ion milling.

ELIONIX: ELS-7500 (2003)



### 1-2 Multi-Target Reactive Sputtering (Ion Beam Sputtering)

Building No. 2, Room 111

This multi-target reactive sputtering (ion beam sputtering) system enables the preparation of thin films/multi-layer samples without Ar plasma damage. This sputtering system consists of three ion source guns for sputtering and one ion milling gun for cleaning. There are six sputtering targets that can be installed in the deposition chamber.

Toei Scientific Industrial Co., Ltd: 3000HC (1989)

### 1-3 Reflection High-Energy Electron Diffraction System

Building No. 2, Room 111

This system consists of a main deposition chamber and a load lock chamber. In the main deposition chamber, epitaxial thin films/metallic superlattice samples can be developed while monitoring the film growth by reflection high-energy electron diffraction (RHEED). This RHEED system has a 30 kV-accelerated electron gun and a 150 mm-diameter screen. Moreover, a PC software for monitoring and analyzing RHEED patterns is available.

(1997)

### 1-4 Multi-Ion Vapor Deposition System

Building No. 2, Room 104

This is a thin-film deposition system equipped with magnetron sputtering cathodes. This deposition system also consists of an electron cyclotron resonance ion source for Ar milling and surface modification. In addition, a Freeman ion source enables ion implantation and dynamic mixing of samples, and its maximum acceleration voltage is 50 kV.

ULVAC: MB98 (1999)







### 1-5 Multi-Layer Chemical Vapor Deposition Reactor

Building No. 2, Room 111

This is an apparatus for fabricating thin films by chemical vapor deposition (DVD) using electron cyclotron resonance (ECR) plasma. The high density and high ionization rate of the plasma in this equipment enables a high deposition rate, excellent step coverage, and low-temperature synthesis of thin films.

Sumitomo Metal Industries, Ltd.: ES-037 (1988)

### 1-6 Hot Working (Forging) Simulator

Building No. 2, Room B06A

Heat treatment under various conditions, high-temperature tensile/compressive deformation, powder sintering, and material phase transformation measurement can be performed using this equipment.

Fuji Electronic Industrial Co., Ltd.: Thermecmastor-Z (2009)

#### 1-7 Spark Plasma Sintering: SPS-1050

Building No. 2, Room B01

This is a powder consolidation machine that uses the spark plasma sintering method. The raw material powder is filled in the conductive mold and is compressed by the top and bottom punches. A large pulsed current flowing through the moldgenerates heat for the sintering. Owing to the rapid heating effect, the grain growth can be suppressed and fine-structured compact specimens can be obtained.

Sumiseki Materials Co., Ltd: DR.SINTER Model SPS-1050 (1992)

### 1-8 Spark Plasma Sintering: SPS-3.20, Mark IV

Building No. 2, Room B04

This is a powder consolidation machine that uses the spark plasma sintering method. The raw material powder is filled in the conductive mold and is compressed by the top and bottom punches. A large pulsed current flowing through the mold generates heat for the sintering. Owing to the rapid heating effect, grain growth can be suppressed and fine-structured compact specimens can be obtained. Compared with the SPS-1050, a larger current (8000 A) and a higher load (20 ton) can be applied in this equipment; this is useful for preparing large compact specimens.

SPS SYNTEX INC: DR.SINTER Model SPS-3.20 Mark IV (2005)









# 1-9 Electron Beam Melting Furnace Building No. 2, Room B05

This device is used for melting refractory materials using an electron beam on a water-cooled copper hearth ( $\varphi 100 \times t10$  mm) under high vacuum conditions ( $10^{-4}$  Pa).

JEOL Ltd.: (1995)

### 1-10 Gas Atomization

Building No. 3, Room 307

This device can make rapidly-quenched metal powder in air or in an inert atmosphere. The molten metal is pulverized and quenched by a high-pressure gas stream. After atomization, the powder is collected in a vacuum chamber that is isolated from air. Thus, easily oxidized metal powders such as titanium- and zirconium-based alloys can be produced safely.

Makabe Giken Co,.Ltd.: RQP1 (2022)

### 1-11 High-Frequency Induction Tilt Casting

Building No. 3, Room 307

This device can prepare a master alloy ingot by tilt-casting into a mold by high frequency induction heating under inert gas atmosphere. The amount of an alloy that can be prepared at one time is about 70 g in terms of iron. By selecting the optimum crucible material, the maximum temperature can be reached up to  $1500 \,^{\circ}\text{C}$ .

Makabe Giken Co, Ltd.: VF-HMF100 (2007)

### 1-12 Single Roll Melt Spinning

Building No. 2, Room B06A

This device is used for ultra-rapid cooling of molten metals in a vacuum or an inert gas atmosphere. An alloy is melted in the quartz nozzle and spun by the high-speed rotating copper roller. The melting temperature of the alloy must be below 1200 °C. This machine can also be used as a copper mold casting machine.

NISSIN GIKEN Corporation: NEV-A04 (1999)







### 2. Performance Evaluation Station

### 2-1 Magnetic Property Measurement System

Building No. 2,Room 107

This measurement system can be used to characterize the magnetic properties of magnetic materials (mainly thin-film samples). The maximum magnetic field is 2 T. The magnetization can be measured.

TAMAKAWA CO.,LTD: TM-VSM2614HGC-KIT (2014)

### 2-2 X-ray Diffractometer (Micro-Area Type)

Building No. 2, Room 103

This X-ray diffractometer can analyze fine precipitation in specimens using a micro X-ray collection device, CBO-f.

Rigaku: UltimaIV/MAJ (2009)

### 2-3 X-ray Diffractometer (Horizontal Sample Setting Type)

Building No. 2, Room 103

A high-performance detector (D/teX Ultra) enables a high-speed analysis, up to 100 times compared with a conventional scintillation counter. In addition, a trace element analysis can be performed. Texture measurement can be performed using the multi-purpose sample stage.

Rigaku: UltimaIV/SG (2012)

### 2-4 X-ray Photoelectron Spectrometer (XPS)

Building No. 2, Room 106

The composition of a sample can be analyzed by measuring the energy and intensity of photoelectrons generated under irradiation of X-rays. The position of peak energy determines the chemical state, and the surface with a depth of several nanometers can also be analyzed. The sample transfer container system enables the analysis of chemically-treated samples without exposure to the atmosphere.

SHIMAZDU Co.: KRATOS AXIS-Ultra DLD (2009)







### 2-5 Field Emission Scanning Electron Microscope (FE-SEM)

Building No. 2, Room 217

High-resolution observations on the order of 1 nm in secondary electron images can be achieved by using the FE electron gun. This equipment can analyze complex surfaces, such as tensile-fractured surfaces. Insulation materials can be observed using various observation modes without pre-treatment.

JEOL: JSM-7500F (2009)

### 2-6 Field Emission Electron Probe Micro-Analyzer (FE-EPMA)

Building No. 2, Room 106

FE-EPMA with a small probe of 40 nm and a high resolution of 3 nm enables high-precision composition analysis using a wavelength dispersive X-ray spectrometer, which can detect elements from boron to uranium. The attached EBSD system analyzes the crystal orientation of specimens.

JEOL: JXA-8530F (2009)

### 2-7 Scanning Electron Microscope (Tungsten Filament) (W-SEM)

Building No. 2, Room 107

This thermionic-emission gun with a tungsten filament provides high-resolution secondary electron images (SEI) and back-scattered electron (BSE) images of metallic and inorganic materials. The chemical compositions of samples can be determined using the attached EDX system. The maximum size of a specimen that can be installed in the analysis chamber is 200 mm in diameter and 80 mm in thickness.

Hitachi High Technologies: S-3400N (2008)

#### 2-8 Superconducting Quantum Interference Device (SQUID) Magnetometer

Building No. 2, Room 107

Quantum Design's MPMS magnetometry provides users with a superconducting quantum interference device, SQUID, which has a sensitivity  $\leq 10^{-6}$  emu under a temperature range of 2–400 K and a magnetic field control below 5 T. AC magnetic measurements can also be performed in a frequency range of 0.01–1500 Hz.

Quantum Design: MPMS-5S (1997)







### 2-9 Differencial Scanning Calorimetry (DSC)

Building No. 3, Room 301

This device is a high-performance differential scanning calorimeter in which the temperature program can be precisely reproduced. The lowest temperature can reach the cryogenic temperature range, and the maximum controlled cooling rate exceeds the glass-forming critical cooling rate of metallic glass. Ultra-high-speed scanning of 750 °C/min in a measurement range of -170–750 °C can be performed in the double furnace system. In addition, because the endothermic and calorific values are directly measured, the sensitivity does not depend on the temperature. The accuracy and reproducibility of temperature and calorific values are excellent.

Perkin Elmer: DSC8500 (2010)



#### 2-10 Conventional Type Thermal Analysis Measurement System (DTA, DSC, TMA)

Building No. 2, Room 215

Differential thermal analysis (DTA), differential scanning calorimetry (DSC), and thermomechanical analysis (TMA) between the sample and reference are monitored against time or temperature, while the temperature of the sample is programmed in a specified atmosphere. A aluminum, platinum, or alumina crucible can be used while monitoring the DTA and DSC properties of the sample. The system delivers an extended temperature range from ambient temperature to 1300 °C for DTA, to 1200 °C for DSC and to 1500 °C for TMA. The cooling unit can be used to measure the DSC in a temperature ranging from -150 °C to 500 °C.

SIISeiko Instruments Inc.: EXSTAR 6000 series, TMA/SS 6200, TG/DTA 6300, DSC 6200 6300 (2007)

### 2-11 Single Crystal X-ray Diffractometer (rotating anode X-ray source)

Building No. 2, Room 103

Single crystal X-ray diffraction is commonly used for the precise determination of crystal structure. The obtained structural information on the distribution of constituent atoms, bond distances, and angles enables us to understand the physico-chemical properties of materials. A classical four-circle goniometer with a scintillation counter (Rigaku AFC7R) and a modern single crystal diffractometer with an imaging plate (IP) (Rigaku R-AXIS) are installed at the Cooperative Research and Development Center for Advanced Materials together with a powerful high-frequency 18kW X-ray generator (ultraX 18). A high temperature sample heating system is also equipped and this allows us to analyze the structural change at high temperatures.

Rigaku: AFC-7R and R-AXIS IV++ with UltraX 18 (2012)





List of equipments in Cooperative Research and Development Center for Advanced Materials as of April 2023

## 2-12 Micro X-ray Diffractometer (rotating anode X-ray source, position sensitive proportional counter)

Building No. 2, Room 103

The characterization of microstructure requires an X-ray diffraction (XRD) analysis in the small area of a large sample. This micro X-ray diffraction ( $\mu$ XRD) analysis utilizes a narrow X-ray beam to perform localized X-ray diffraction measurements of a very small area. This method requires a powerful high-frequency 18 kW X-ray generator (ultraX 18) and dedicated incident beam collimators that reduce the emitted X-rays. The  $\mu$ XRD technique is usually applied to the characterization of small spots on samples with strong composition gradients. This technique is also useful in the analysis of contaminations, inclusions, petrological samples, test pieces of alloy samples, and patterned wafers.

Rigaku: RINT-2000 with UltraX 18 (2016)

### 2-13 Wide and Small Angle X-ray Diffractometer (rotating anode X-ray source)

Building No. 2, Room 103

This device with a 18 kW rotating anode X-ray source, is compatible with the small-angle scattering method (SAXS) and the powder diffraction method (XRD). SAXS method, which measures X-ray scattering intensity with a scattering angle of several degrees or less, allows us to analyze the particle shape and size together with their distribution. In addition, the common XRD method is a powerful method to identify the structure of substances by combining a high-intensity radiation source, a monochromator and a scintillation counter. As an option, it is also possible to analyze the structure of a thin film using a parallel beam optical system.

Rigaku: UltraX18VB3-450 (2020)

### 2-14 Vibrating Sample Magnetometer (VSM)

Building No. 2, Room B05

Magnetic properties of bulk, powder, and thin-film materials can be measured with high sensitivity in a temperature range of 77 K to 1200 K using this magnetometer. The maximum applicable magnetic field is 1.5 T and the magnetization range is  $10^{-3}$  to  $10^2$  emu. Magnetization curves at an arbitrary temperature, thermal magnetization curves under a constant magnetic field, temperature changes during magnetization, spontaneous magnetization, and Curie and Neel temperatures can also be obtained. The measurement of magnetostriction by using Strain-Gauge under applying magnetic field is also possible.

Toei Scientific Industrial: VSM-5 (1995)







### 2-15 Laue X-ray Back Scattering by Digital CCD Camera

Building No. 2, Room 110

The constituent instruments of this system are 1) X-ray generator, 2) Laue back scattering optical system, 3) Sample stage, 4) Cooling-type CCD digital camera, 5) PC, and 6) Orientation-analyzing software. A tungsten target is used as the X-ray tube and the maximum load power is 3 kW. A four-axis goniometer is commonly used in the electric discharge machine. Diffraction spots can be immediately obtained on the PC using the CCD digital camera.

Rigaku: RASCO-IIBLA (2014)

### 2-16 X-ray Diffraction Apparatus for Crystallographic Orientation Measurements

Building No. 2, Room 103

This experimental facility is useful for measuring the crystallographic orientation of bulk crystalline samples such as crystal blocks and wafers. By combining with various sample holders, it is possible to determine the cutting orientation of various single crystals such as quartz, sapphire, Si, and GaAs.

Rigaku: 2991F2 (2021)

### 2-17 Seebeck Coefficient/Electrical Resistivity Measurement System

Building No. 3, Room 308

The Seebeck coefficient and electrical resistivity can be measured by the steady direct-current method and by the four-point probe method. The operating temperature ranges from room temperature to 1000 °C. The available sample shape is a rod or a prism with a length of  $5 \sim 2$  mm.

Advance Riko: ZEM-3 (2015)

### 2-18 Laser flash apparatus for thermal diffusivity and conductivity measurements

Building No. 3, Room 308

The Laser Flash Apparatus is useful to measure the thermal characteristics (thermal diffusivity, specific heat capacity, thermal conductivity) of solid materials such as metals, ceramics, glass, carbon, and plastics. Since it is easier to measure than the steady-state method and the data obtained is so reliable, it should be the most effective method to obtain the thermal conductivity.

Advance Riko: TC-7000S (2020)









### 2-19 Static particle image analyzer

Building No. 3, Room 307

This device can accurately evaluate the morphology of powder particles by static image analysis. A series of operations from sample dispersion to measurement can be performed automatically. The imaging optical system can be selected from transmission or incident-light mode. The binarized image and characteristic parameters are automatically recorded. It can handle many types of powder samples such as metals, ceramics, and organic substances.

Malvern Panalytical: Morphologi 4 (2021)

### 2-20 Flat / Cross Section Ion Milling System

Building No. 3, Room 207

This apparatus allows us to polish the sample using the sputtering phenomena by argon ion beam irradiation. In the planar milling mode, it is possible to remove the surface contaminated layer, fine scratches, and strain of the sample specimen. In the cross-section milling mode, focused argon ion beam processing provides a sound cross-section surface for microstructural observation. We can obtain high-quality image and analysis via scanning electron microscope (SEM) and backscattered electron diffraction (EBSD) using the specimen polished by the apparatus.

Hitachi High-Tech Corporation: IM4000PLUS (2022)





### **3.** Crystal Preparation Station

### **3-1 Solidification Control Equipment from Liquid Phase**

Building No. 2, Room B04

This equipment can obtain single crystals of metals, semiconductors, and oxide and fluoride compounds by the Czochralski process or by the vertical Bridgeman process. The state of melted materials can be monitored in-situ. The materials are melted by a high-frequency induction heating system or by a resistance heating system under an arbitrary atmosphere.

Celec: 89026 (1990)

### 3-2 Single Crystal Growth Equipment by Resistance Heating

Technical Center II, Room 105

This equipment can obtain single crystals of metals, semiconductors, and oxide and fluoride compounds by the Czochralski process or by the vertical Bridgeman process in horizontal magnetic fields (maximum of 0.4 T). The materials are melted by a resistance heating system with a double zone-type carbon heater under an arbitrary atmosphere.

Celec (1989)

### 3-3 IR Image Furnace for Floating Zone Melting

Building No. 3, Room 208

This equipment can obtain highly purified single-crystalline materials by the floating zone melting method using an infrared (IR) beam focused from a halogen lamp. It can melt materials under an arbitrary atmosphere and can be used to obtain single crystals of metals, alloys, intermetallic compounds, and oxides. The equipment can obtain highly purified materials because the materials are melted without the use of a reaction container.

Asgal: FZ-SS35WV (1989)

### 3-4 Electron Beam Furnace for Floating Zone Melting

Building No. 3, Room 208

This equipment can obtain highly purified single-crystalline materials by the floating zone melting method using electron beams. The charged materials can be heated up to  $3000 \,^{\circ}$ C in vacuum.

JEOL: JEBS-3B (1974)









### 3-5 Crystal Growth Furnace with HF-Inductive Heating System

Building No. 3, Room 208

This equipment can obtain single-crystals of metals, semiconductors, and oxide and fluoride compounds using a high-frequency (HF)-inductive heating system. The charged materials are melted either by the Czochralski process, the vertical Bridgeman process within a crucible, or the floating zone melting without a crucible.

Kokusai Electric Inc.: DP-20MP (1981)

### **3-6 Tungsten Resistivity Element Furnace for Vacuum Heating**

Building No. 3, Room 208

This furnace can be used to heat and melt samples at a high temperature (up to 1900 °C) using a tungsten mesh heater in vacuum.

ULVAC: FHW-50-special specification (1971)

#### **3-7 High-Frequency Induction Furnace**

Multi-Use laboratry, Room 104

This furnace is capable of melting quantities of various metals (1 kg in the case of iron) in a high vacuum of  $2.0 \times 10^{-3}$  Pa. The charged metals are melted in an appropriate crucible in vacuum or an inert gas atmosphere; they are then cast into a mold by inclining the crucible. The furnace can be used to prepare ingots of raw metals, mother alloys, and metallic glasses.

DIAVAC: VMF-1-11 (2004)

### **3-8 Small vacuum melting furnaces**

Building No. 2, Room 301

This furnace is capable of melting quantities of various metals (0.4 kg in the case of iron) in a high vacuum of  $6.7 \times 10-3$  Pa. The charged metals are melted in an appropriate crucible in vacuum or an inert gas atmosphere; they are then cast into a mold by inclining the crucible. The furnace can be used to prepare ingots of raw metals, mother alloys, and metallic glasses.

NISSIN GIKEN: NEV-M04S (2021)







### 3-9 High-Temperature Floating Zone Furnace for Composite Ceramics

Building No. 2, Room B08

This equipment can be used to heat and melt samples at a high temperature (up to above 2000 °C) using a xenon lamp heating system without a crucible. The equipment is used extensively for single-crystal growth and ceramic synthesis at a high melting temperature.

Asgal: FZ-20065XHVG (1998)

### 3-10 Vacuum Arc Melting Furnace for Small-sized Ingot

Building No. 3, Room 203

The arc-melting furnace can melt charged materials and fabricate polycrystalline material ingots. The material is melted by means of an electrical arc, which is generated conventionally by a power of 2 to 6 kW. It is used extensively to melt and cast metals, alloys, intermetallic compounds, and some oxides, by increasing the temperature to above 3,000 °C.

obtainable sample size: [disk-shaped sample] max  $\phi$  30 mm×10 mmt, [rod-shaped sample]  $\phi$  8-10 mm×90 mmL

DIAVAC: ACM-01 (1986)





### 3-11 Vacuum Arc Melting Furnace for Large-sized Ingot

Building No. 3, Room 203

This is a larger version to Vacuum Arc Melting Furnace for Small-sized Ingot (Max output: 300 A) (listed up as # 3-10), and equipped with a turbo-molecular pump to evacuate the chamber. We can obtain refractory alloy ingots with a dimension of 50 mm in diameter and 10 mm in thickness.

DIAVAC: ACM-S14TMP-M (2020)

### 3-12 Arc-Melting Furnace with Horizontal Traveling Hearth

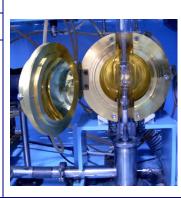
Building No. 3, Room 203

This arc-melting furnace exhibits a performance similar to that of a conventional furnace (such as the one listed in 3-10). In addition, the furnace is attached with a system that shifts the copper hearth horizontally at a speed of 5-60 mm/h. Therefore, it can fabricate long rod-shaped specimens with elongated and coarsened grains.

obtainable sample size: [rod-shaped sample]  $\phi 10 \text{ mm} \times 290 \text{ mmL}$ 

DIAVAC: ACM-S-6 (1985)





### 3-13 Programmable Furnace with MoSi<sub>2</sub> Heater

Building No. 3, Room 116

This furnace, which is equipped with a  $MoSi_2$  heater, can heat up to a maximum temperature of 1600 °C in air. The furnace can also be used for the synthesis of complex oxides by reaction sintering and for the fabrication of various sintered compacts.

NECCO: 850-M-120×120 (1998)

### 3-14 Programmable Furnace for Flux Growth

Building No. 3, Room 117

This furnace is used for the flux growth of single-crystalline compounds. It can be used to prepare fine single-crystals by controlling heat under a flowing gas atmosphere.

IZUMI TEC: IZU-KGSS-1500 (2008)

### 3-15 µ -PD Apparatus for Small-Diameter Crystal Growth

Building No. 3, Room 208

The materials charged in a crucible are melted by a high-frequency induction heating system. The bottom of the crucible has a hole with a size of several millimeters, and the charged material is drawn down slowly after contacting a seed crystal. Thus, the apparatus can form a single-crystalline material on a thin stick with a diameter of several millimeters. The cross-section of the stick-shaped single-crystal can be controlled by changing the shape of the hole at the bottom of the crucible.

TDK: µPD-HT (2006)





