



Solving Problems in R&D and Production with Toray's Chemical Analysis Service

Part 1. General Overview: The Attractiveness of Toray Research Center (10 min.)

Part 2. Latest Innovative Analysis Methods & Material Detective Work (20 min.)



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Part 1: The Attractiveness of Toray Research Center

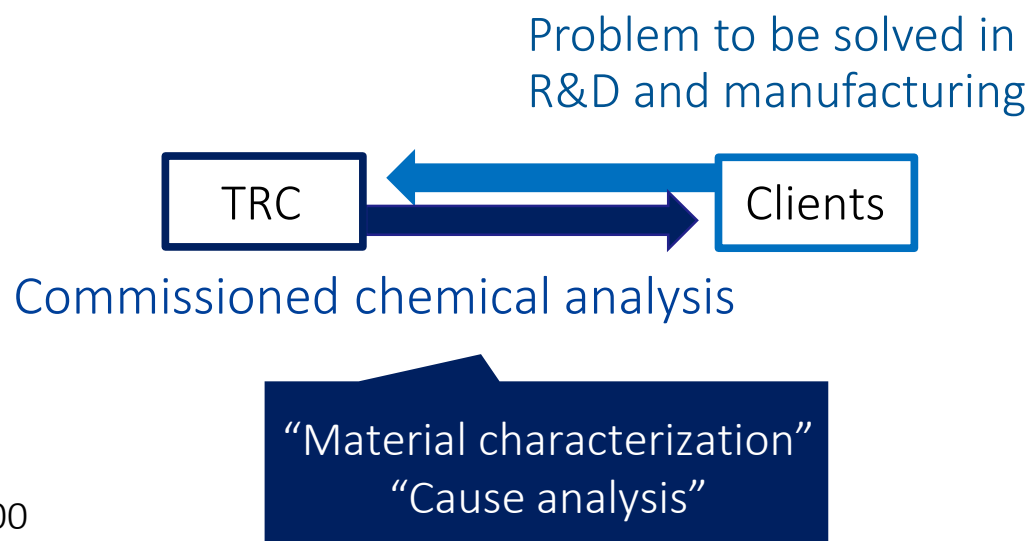


Toray Research Center / Concept & Function

Your partner for problem-solving in the fields of R&D and manufacturing



- Number of Employees: 400
(20% have a Ph.D.)
- Annual Sales: 65 MEUR
(as of 2024)



Company

University

Public Institute

- Number: 1,100 organizations annually
(Number of requests: 12,000 annually)

No.1 advanced chemical analysis service in Japan!

Business field

Materials



Organics, Polymers, Inorganics, Ceramics, Metals, Films, Nano Materials, FRP, Paint Coating, Adhesion, Electronic Materials, Catalysts, Gas Analysis, Combustion

Lithium-ion Battery, Fuel Cell, Solar Cell



Batteries

Semiconductors & Packaging



LSI, IC & Memory, Power Semiconductor, Discrete Device, Lithography, Compound, Semiconductor, LED, Ion Implantation, Packaging, TSV, Electronic Materials, Metals

Lithium-ion Battery, Fuel Cell, Exhaust Gas, Catalyst, Combustion Gas, Paint Coating, FRP, Polymers, Catalysts, Power Semiconductor



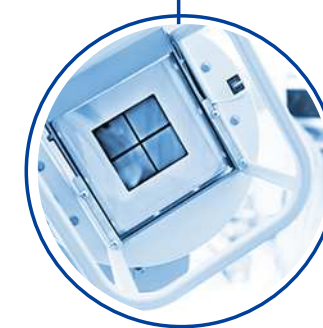
Automobiles & Aerospace

Displays & IT Equipment



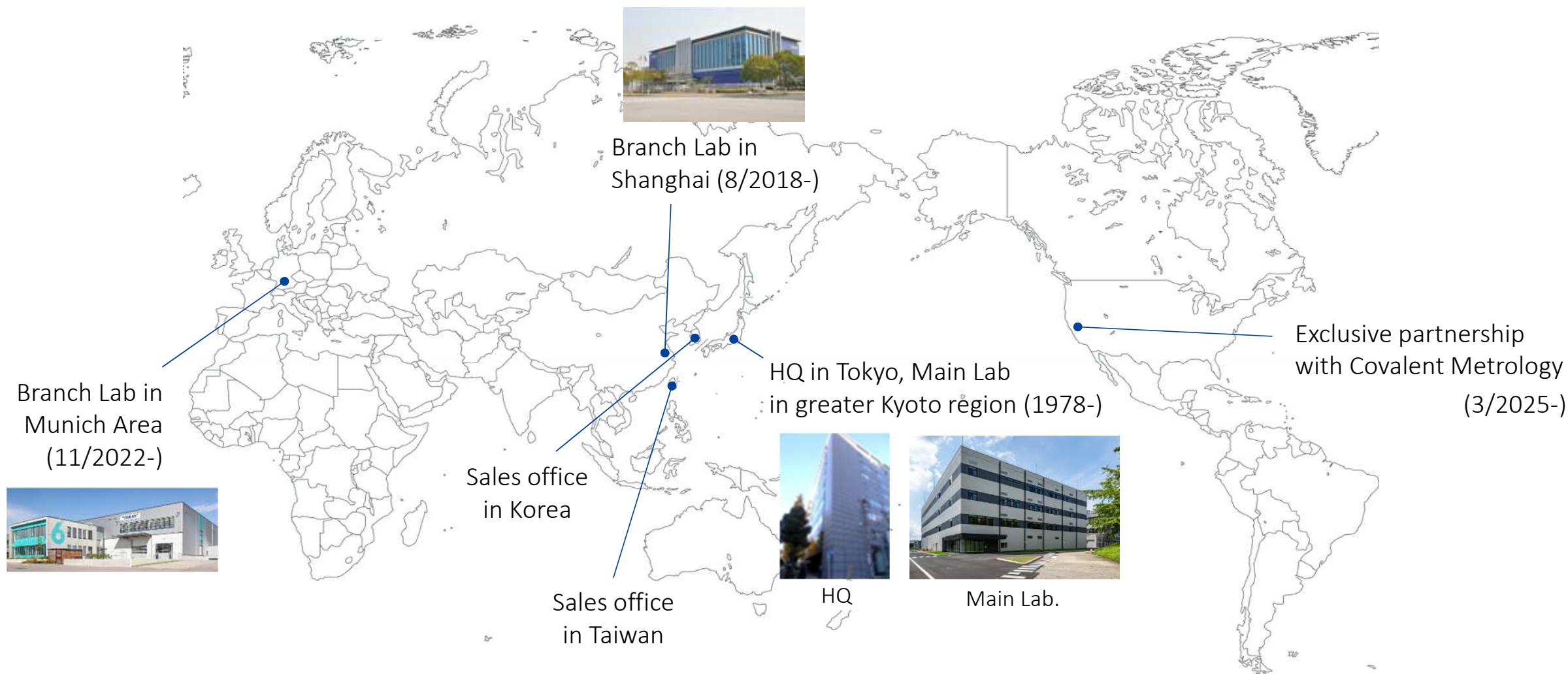
LCD, OLED, μ LED and QD device, Printer, Electronic Materials

Cosmetics, Food, Biomass, Biological Tissue, Biological Product, Diagnostic Agent, Pharmaceutical, Tissue Engineering & Regenerative Medicine, Medical Device

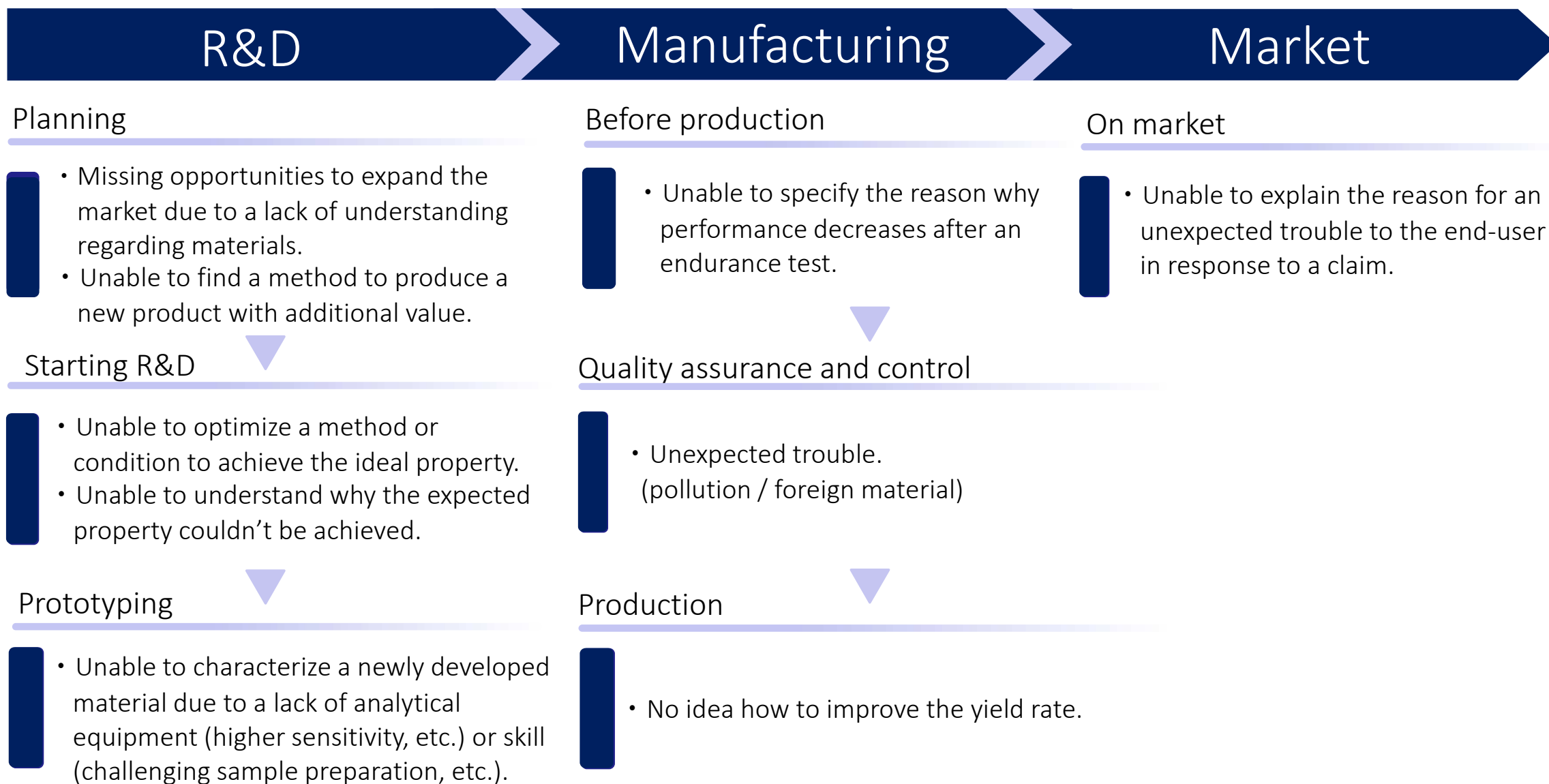


Life Science

Toray Research Center (TRC) / Location



Problems to be solved



Problem solving at TRC

R&D

Manufacturing

Market

Case 1

Unexpected trouble
(foreign substance)



Problem to be solved

Black-colored foreign substance in resin after molding.

Understanding the situation

The customer suspected that the foreign substance might be coming from a metal pipe that is part of the manufacturing instrument. As the first step, the composition of the foreign substance was investigated, which revealed that it was not derived from the metal. Hence, TRC thoroughly discussed the situation with the customer and then made a hypothesis.

Analysis

Based on the hypothesis, analyses of additives in the resin were performed. As a result, the foreign substance was confirmed to be an organic material that was accidentally produced through a reaction with a catalyst added during the production of the virgin resin.

Achievement

No further foreign material was found after the replacement of the catalyst.

How to achieve problem solving

TRC's competence 1: Experience over 45 years

TRC pioneered and developed a new market, “R&D and manufacturing support through commissioned analysis” in Japan. We have been supporting clients across a wide range of fields since 1978.



How to achieve problem solving

TRC's competence 2: Analytical instrument

Data are acquired using:

Wide variety / State-of-the-art / Original instruments



How to achieve problem solving

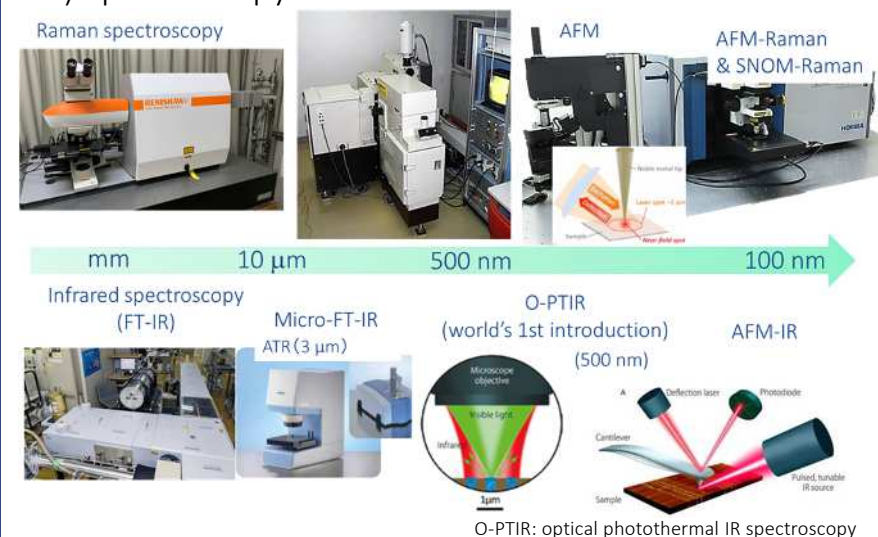
TRC's competence 2: Wide variety of analytical instruments

Representative analytical instruments in Japan's main laboratory

- | | | | |
|------------------------|-------------------------|--------------------------|---------------|
| • AAS | • GD-OES | • PIXE | • XPS |
| • AEM | • GPC | • Raman | • XRD |
| • CE | • HFS | • RBS | • XRF |
| • CL | • Hg Probe | • Rheological Properties | • XRM, et al. |
| • CLSM | • IC | • SEM | |
| • Composition Analysis | • ICP-MS | • SIMS | |
| • Cs-corrected STEM | • ICP-OES | • SPM | |
| • EA | • ITC | • SRA | |
| • EBSD | • LA-ICP-MS | • TD-NMR | |
| • EGA | • LC/MS | • TA | |
| • ELISA | • LC-NMR | • TEM | |
| • EPMA | • MALDI-MS | • Thermal diffusivity | |
| • ESR | • Mechanical Properties | • TOF-SIMS | |
| • FT-IR | • Nano-SIMS | • TPD-MS | |
| • GC | • NMR | • UV-VIS | |
| • GC/MS | • NMR (solid-state) | • Vapor Pressure | |
| • GDMS | • NRA | • XAFS | |

Comprehensiveness of resolution

Ex.) Spectroscopy

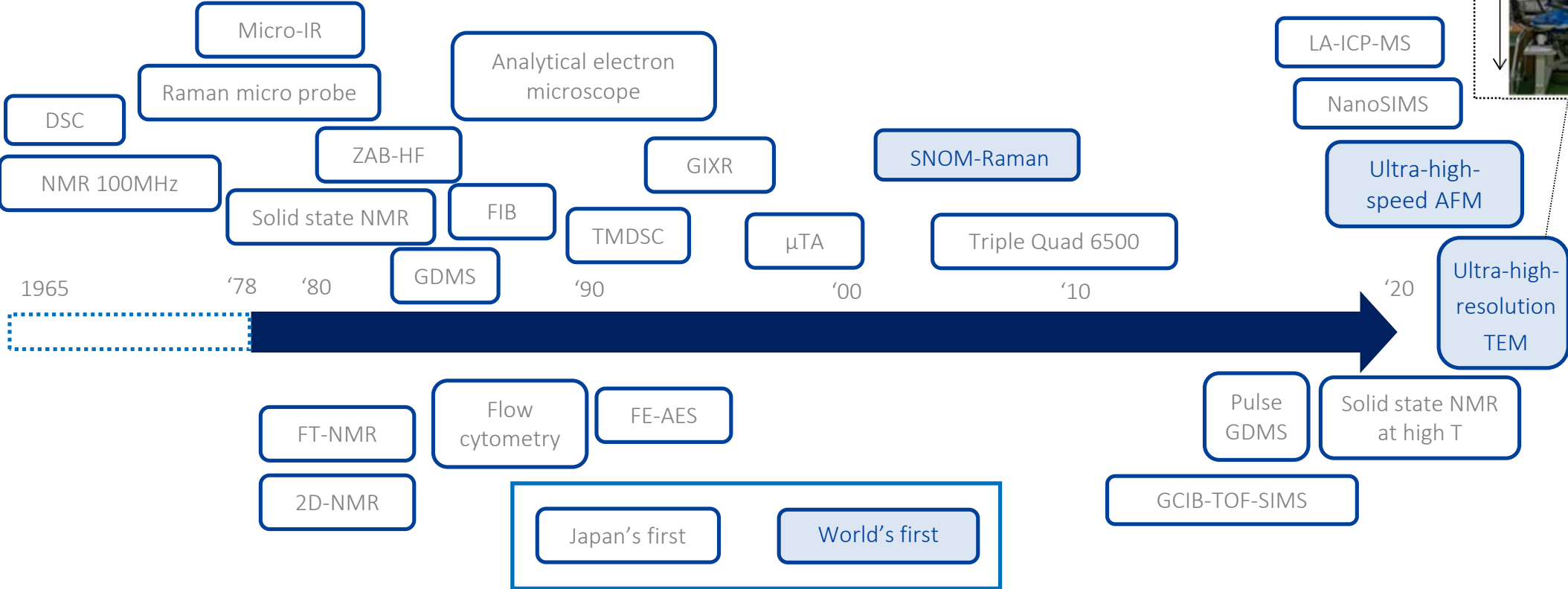


Top-level versatility under one roof

How to achieve problem solving

TRC's competence 2: State-of-the-art analytical instruments

History of system installations

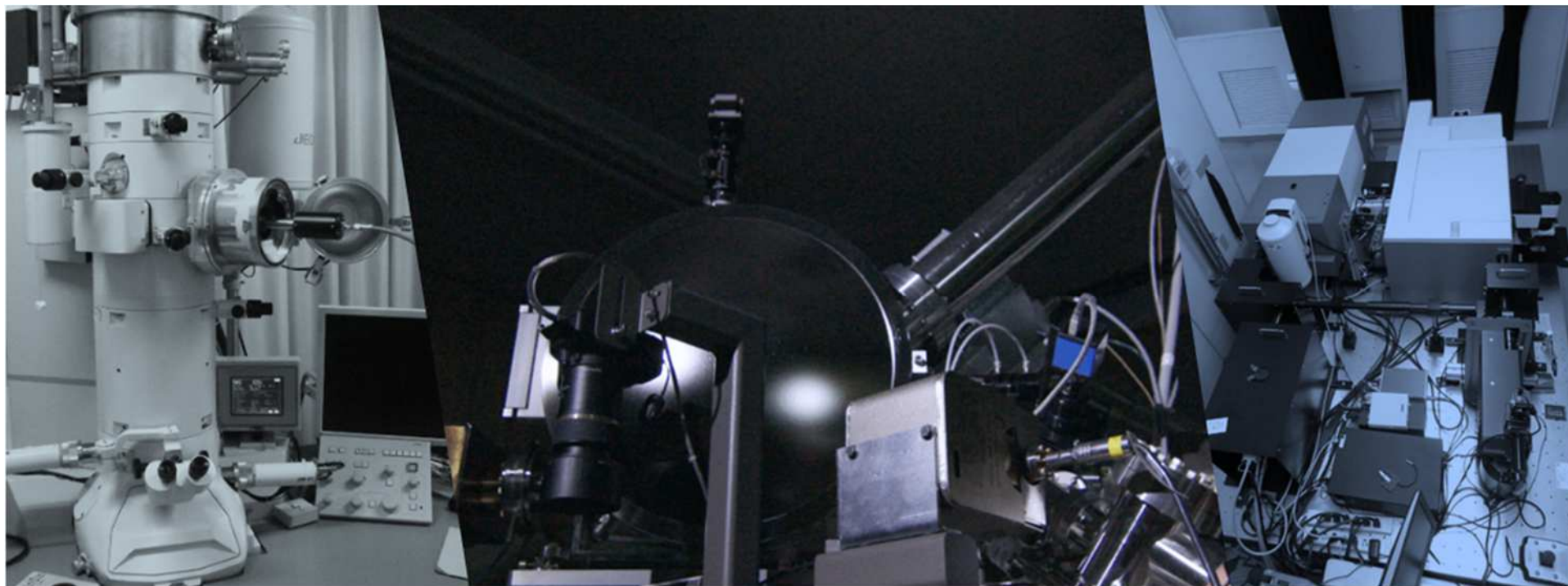


Provision of cutting-edge analysis

How to achieve problem solving

TRC's competence 3: Original analytical instruments

Joint development with manufactures of analytical instruments



STEM-CL

SNOM-RAMAN

Leading analytical chemistry

How to achieve problem solving

TRC's competence 3: Analytical skills

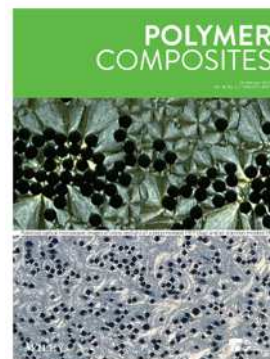
Data are acquired by highly skilled researchers



Numerous awards for analytical skills from external organizations

Example 1: Awards from the Japan Society for Analytical Chemistry, Japan Society of Applied Physics, etc.

Example 2: Latest accreditation to ISO/IEC 17025:2017 as the first Japanese company for impurity analysis in semiconductors by SIMS



Example 3: Latest selection for a journal cover image. Yuki Yoshida et al. February 2025. <https://doi.org/10.1002/pc.29114>. (License from John Wiley and Sons)

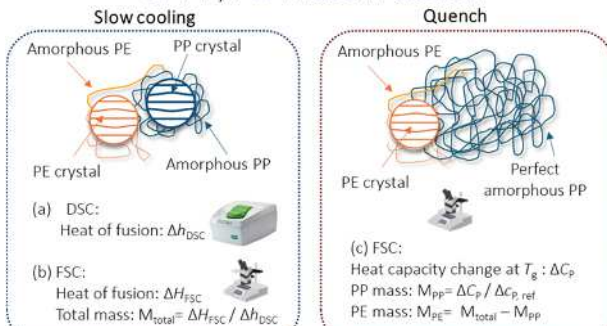
How to achieve problem solving

TRC's competence 3: Analytical skills

Development of innovative methodologies

Composition

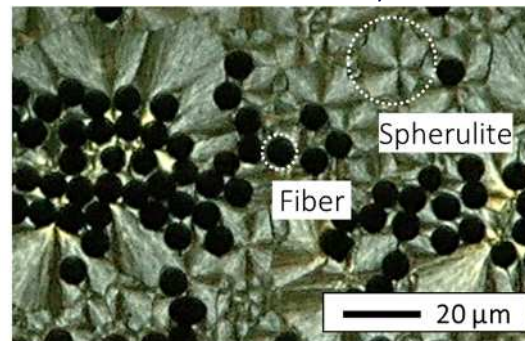
Eco-friendly and rapid determination of PP/PE mass fraction



Publication: Furushima et al. J Appl Polym Sci. 2024

Morphology

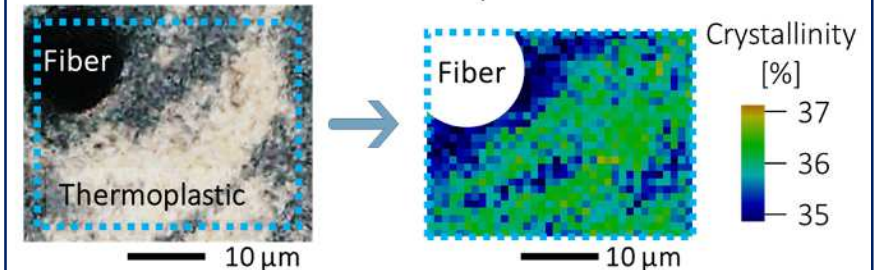
Macroscopic crystalline structure of F RTP observed by POM



Publication: Yoshida et al. Polym Compos. 2024

Chemical structure

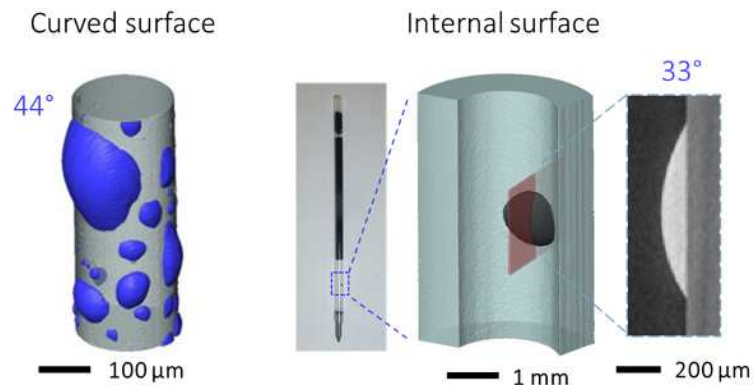
Microscopic distribution of polymer higher-order structure by μ -Raman spectroscopy



Publication: Yoshida et al. Polym Compos. 2025

Surface property

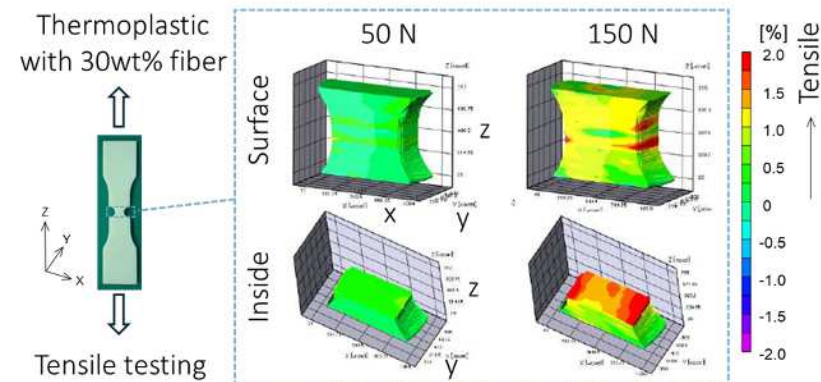
Micro-scale contact angle measurement by Xray-CT



IP right: Patent number; JP7572335, country; Japan

Mechanical property

In-situ 3D strain distribution monitoring by Xray-CT with DIC



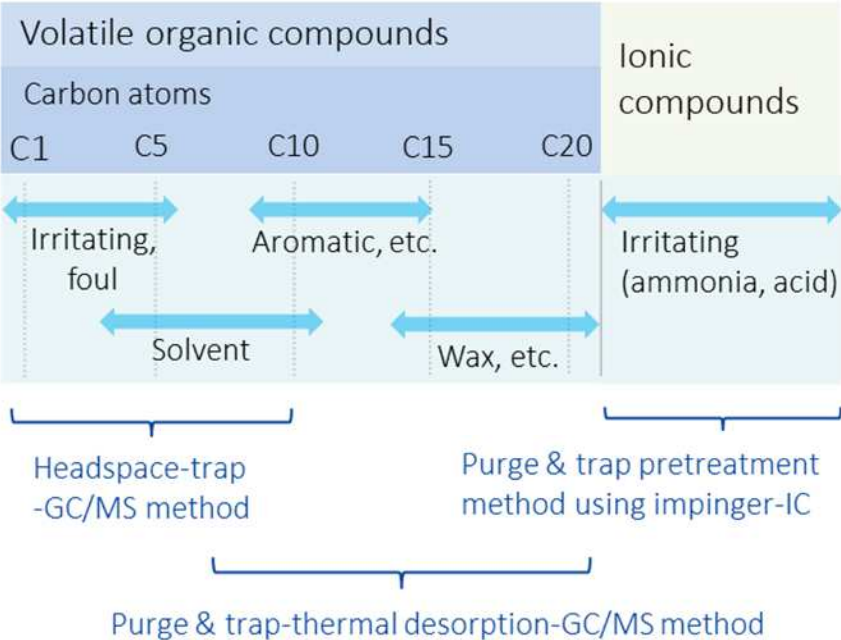
Funded from Japan's national research institute

Example of TRC's chemical analyses

Topic 1: Assessment of odor

TRC's competence
1. Portfolio covering every type of odor

Types of odor and suitable analytical methods



2. Original system



GC/HRMS
(Mass resolution;
More than 10 times higher
than the standard one.)

3. Advanced data analysis

Multivariate analysis, deconvolution, search through database of over 140,000 types of odor.

4. Licensed olfactory measurement operator



Detection and characterization of odor based on human perception.

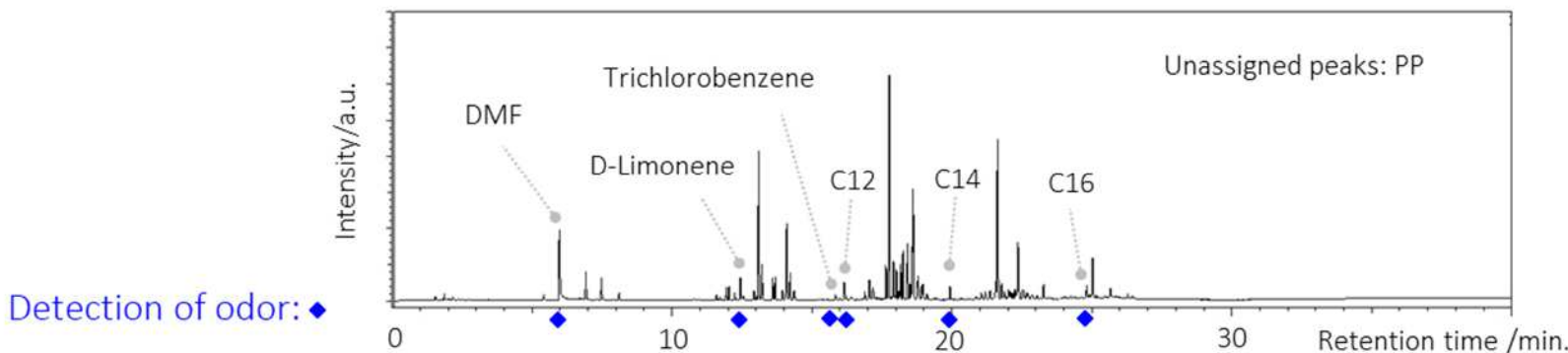
Strength in specification of root cause of odor

Example of TRC's chemical analyses

Sample: recycled PP

Fresh and oily odor, presumably deriving from volatile organic compounds, while the usage at 40°C.

Mass spectrum of the gas emitted from the r-PP



Specification of the root cause of the odor

* Calc.=Amount/threshold

Compound	Amount	Type of odor	Odor detection threshold by human sense	Contribution to the odor*
N,N-Dimethylformamide	240 ng/g	Weak amine	1.8 ppm	133
D-Limonene	38 ng/g	Citrus	0.038 ppm	1000
Trichlorobenzene	12 ng/g	Aromatic, unpleasant	ca. 1 ppm	12
Linear alkane (C12/14/16)	88 ng/g	Oil	0.11 ppm	800

The root cause was assigned to D-Limonene and linear alkanes

Example of TRC's chemical analyses

Topic 2: Cause analysis of deteriorated mechanical property of recycled GFRP

Sample	Virgin GF	Virgin PA6	r-PA6	Tensile strength
Virgin GFRP	30%	70%	0%	187 MPa
Recycled GFRP	30%	19%	51%	137 MPa

Possible factor 1) Strength of resin and fiber

Analysis by Nano-indentation

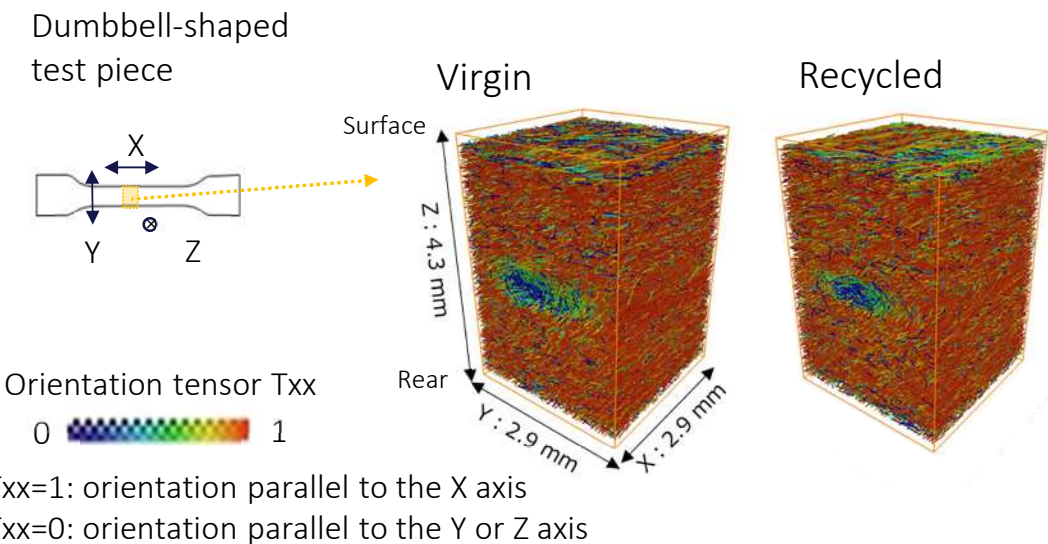
Multiple fibers and various points of the resins were measured.

Sample	Elastic modulus / GPa	
	GF	PA6
Virgin	55.7 ± 8.5	2.7 ± 0.4
Recycled	56.0 ± 8.8	2.8 ± 0.6

No significant differences

2) Fiber orientation

By Xray-CT + Image analysis



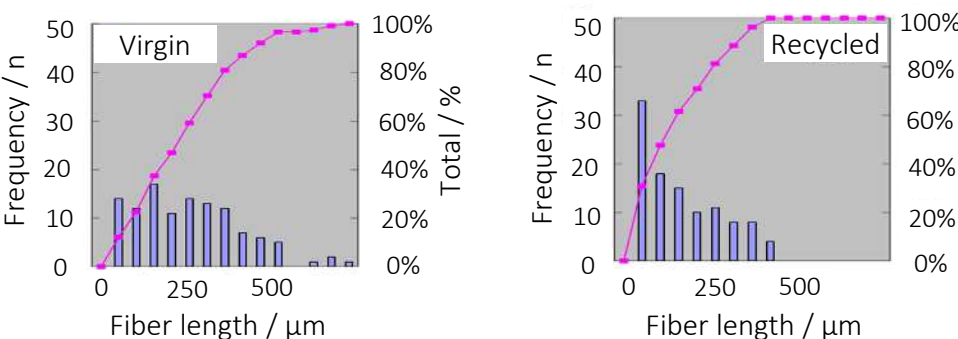
No significant difference

Example of TRC's chemical analyses

3) Fiber length

The fibers were extracted from the resin by a solvent.

By optical microscope

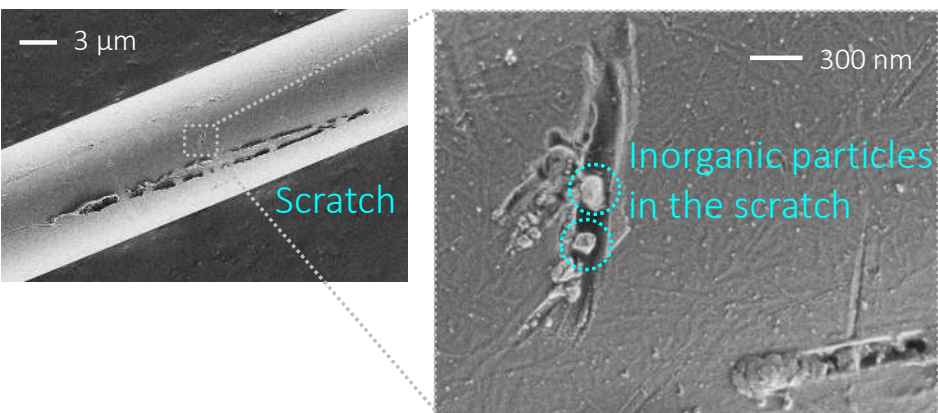


Shorter length could be one of the reasons for the deteriorated mechanical property.

Root cause analysis Why did the fiber length become shortened?

Surface of the shortened fiber

By SEM



Inorganic particles, which are assumed to derive from the recycled resin, might have damaged the fibers and triggered their shortening during the compounding process.

Possible measures

- A. Adoption of longer fibers for the recycled resin, taking fiber breakage into consideration.
- B. Elimination of the inorganic particles from the recycled resin, before mixing with the fibers.

Customer voice

The reason to choose TRC

1. Proper understanding of analysis request
2. Proposal of suitable analysis approach
3. Analysis with sophisticated skills
4. Discussion on the obtained data
5. Experience and know-how accumulated through over 45 years of business

Branch Labs in Europe and China

Branch Lab in Munich area,
Germany, launched in Nov. 2022.



List of analytical instruments

- Xray-CT
- Optical microscope
- SEM-EDX
- DSC
- Raman
- Macro-IR
- Micro-Imaging-IR
- TGA
- DMA
- Microtome
- Polishing machine
- Temperature control stage for microscopy, and others.



Branch Lab in Shanghai area,
China, launched in Nov. 2018.



List of analytical instruments

- Optical microscope
- Macro-IR
- Micro-Imaging-IR
- GCIB-XPS
- GC
- GC/MS
- IC
- TD-GC/MS
- SEM-EDX
- TEM
- BIB
- , and others.



Part 2: Latest Innovative Analysis Methods & Material Detective Work

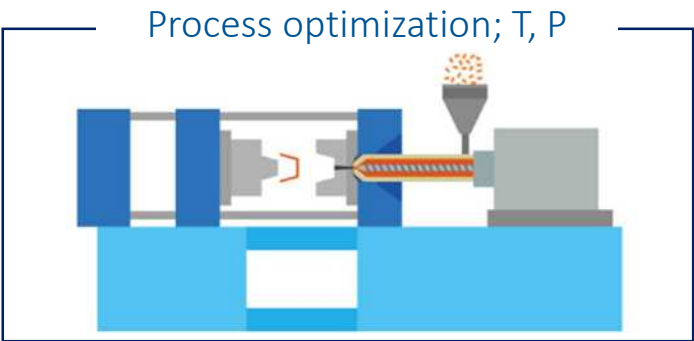


Topic:

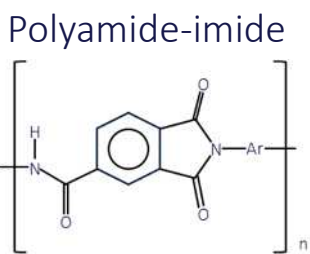
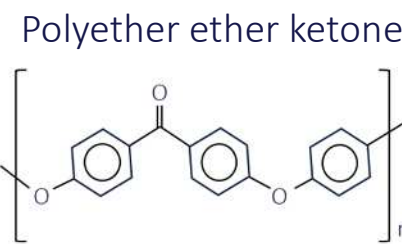
- A. Characterization of Molecular Weight Distribution of Super-Engineering Plastics by GPC
- B. Identifying Chemical Composition of Tiny Foreign Substances for Quality Control
- C. Optimization of Molding Process of Thermosetting Plastics by Fast-Scanning Calorimetry
- D. Reverse Engineering of Plastic Parts to Boost R&D
- E. Root Cause Analysis of Discoloration

A. Characterization of Molecular Weight Distribution of Super-Engineering Plastics by GPC

MWD: critical factor to affect the molding conditions and to ensure the product performance



Challenge Assessment of super-engineering plastics (SEP), due to the low-solubility

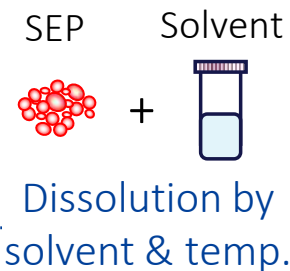


High solvent resistance

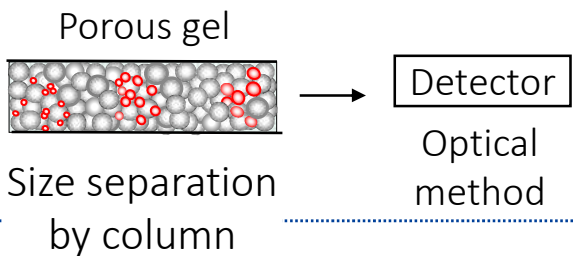
A. Key Point: Dissolution of SEP without decreasing its MW

Flow of our GPC measurement

1. Sample preparation



2. Measurement

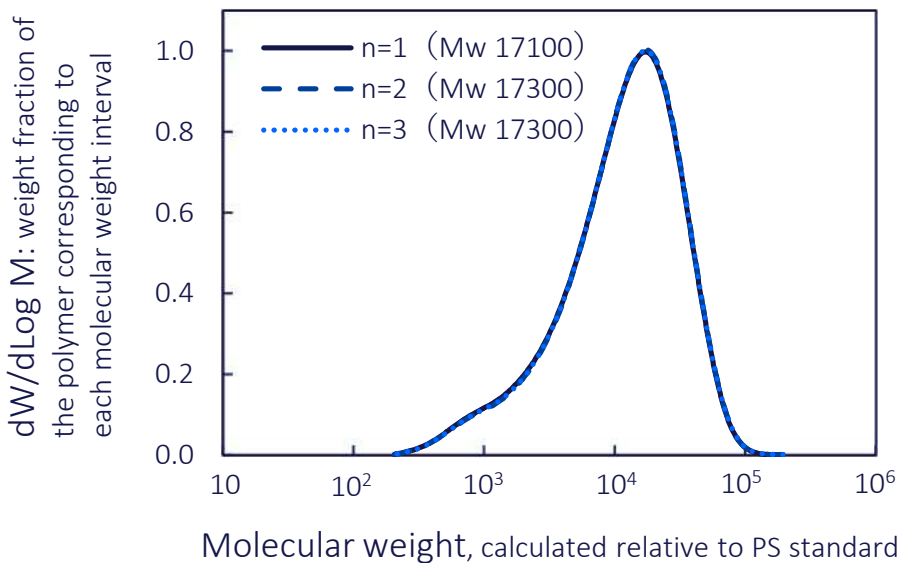


TRC competence: expertise & equipment versatility enabling the suitable selection

3. Data output

‘Differential molecular weight distribution’

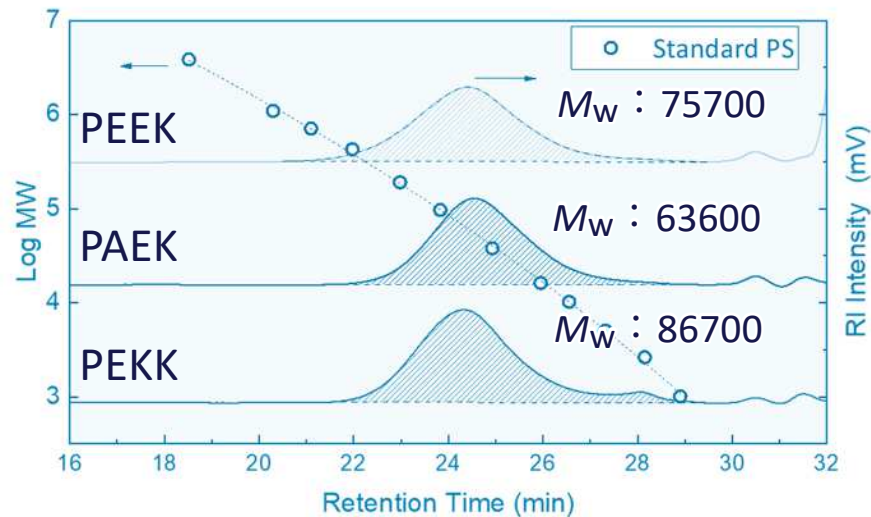
Ex.) Sample: Poly phenylene sulfide



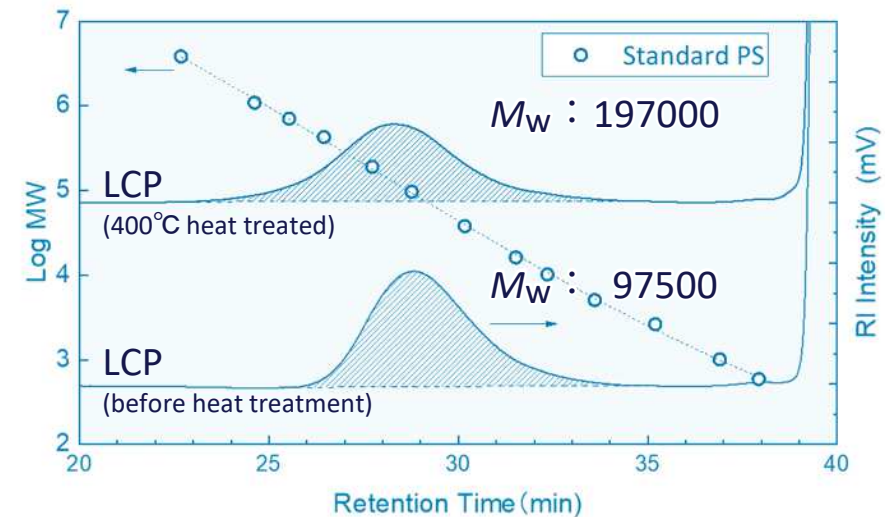
Accurate and reliable

A. Unveilment of Molecular Structure

Application Ex. 1) Understanding virgin resin



2) Assessing degradation behavior & degree



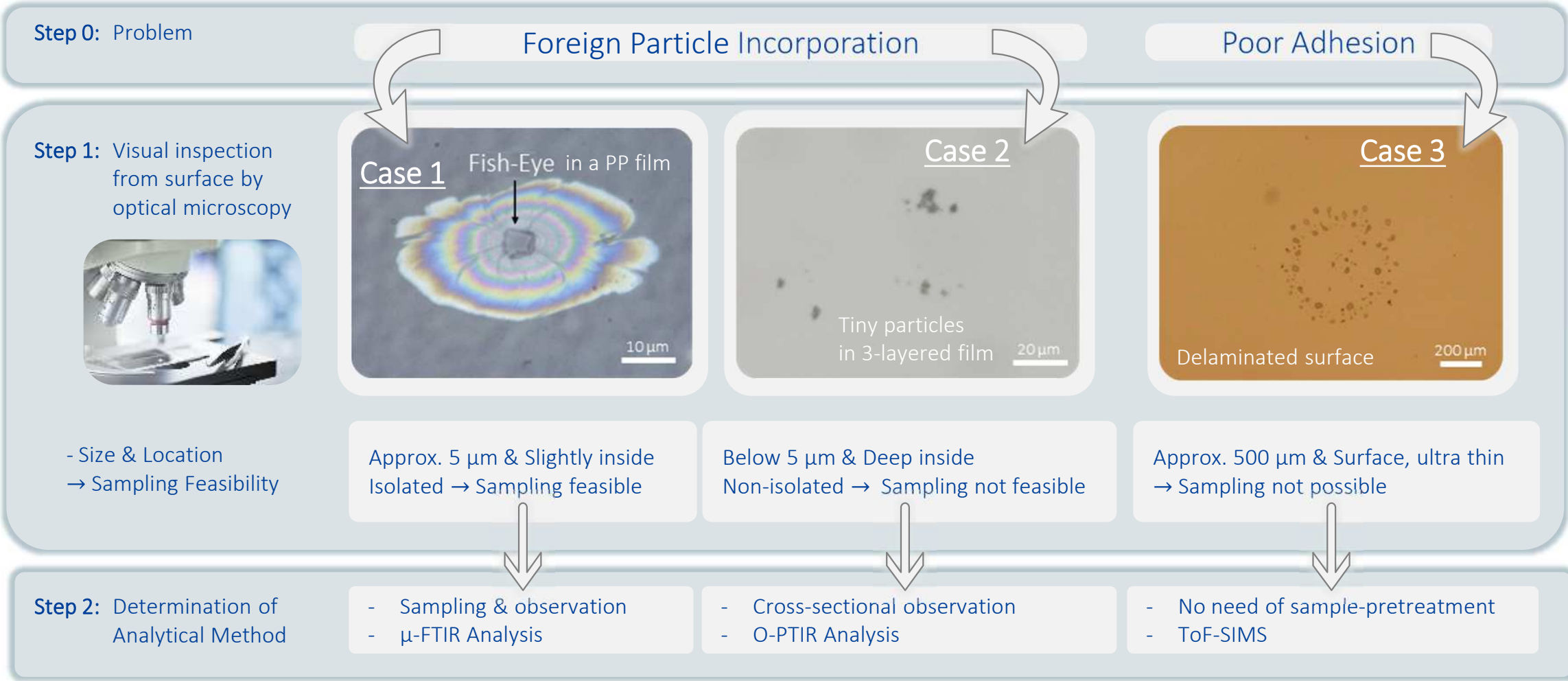
MW: Molecular Weight, M_w : Weight average molecular weight, RI intensity: Detected amount, Retention Time: Time required to pass through the column

‘Exemplary list of SEPs we can measure’

- Polyamide imide
- Polyether imide
- Polyphenylene sulfide
- Polysulfone
- Polyether sulfone
- Liquid crystal polymer
- Polyether ether ketone
- Polyaryl ether ketone
- Polyether nitrile

B. Identifying Chemical Composition of Tiny Foreign Substances for Quality Control

Case study Three Exemplary Cases



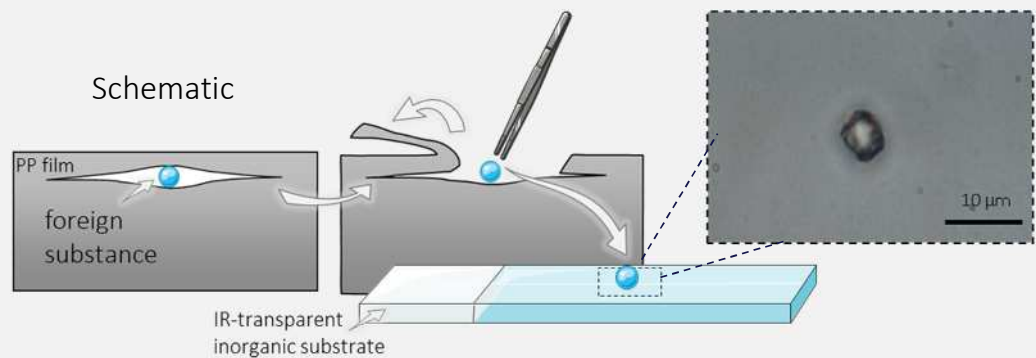
Continuing to the following pages.

B. Identifying Chemical Composition of Tiny Foreign Substances for Quality Control

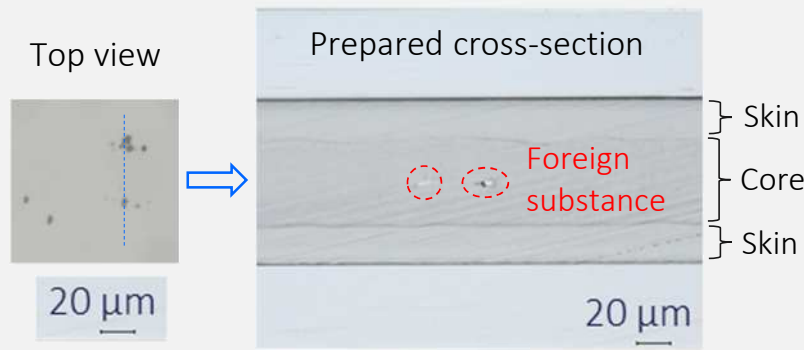
Case 1 & 2

Step 3:
Sample
preparation
&
Morphological
characterization

Case 1 Micro-sampling

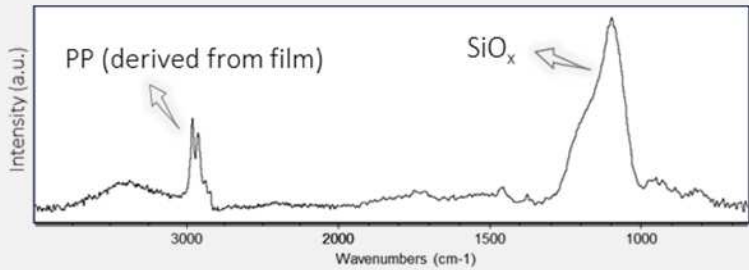


Case 2 Pin-point cross-sectioning



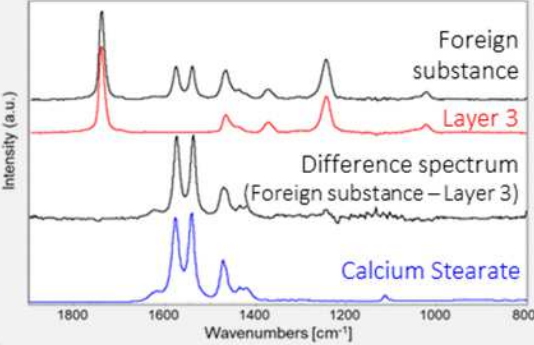
Step 4:
Analysis

IR Spectrum – Spatial resolution: micrometer



The foreign substance is hypothesized to be silica, potentially originating from filler agglomerates or external contaminants.

O-PTIR Spectra –Spatial resolution: sub-micrometer



The foreign material likely stems from contamination with higher fatty acid salts (e.g., lubricants).

B. Identifying Chemical Composition of Tiny Foreign Substances for Quality Control

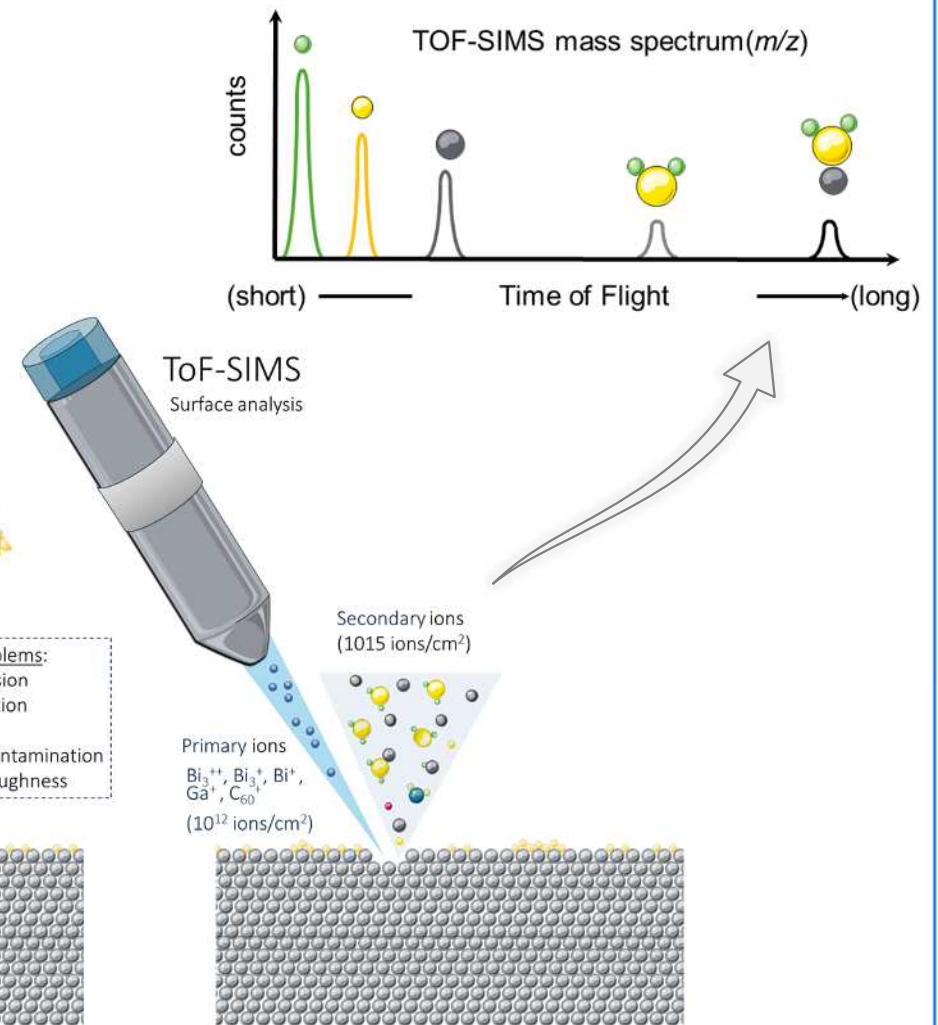
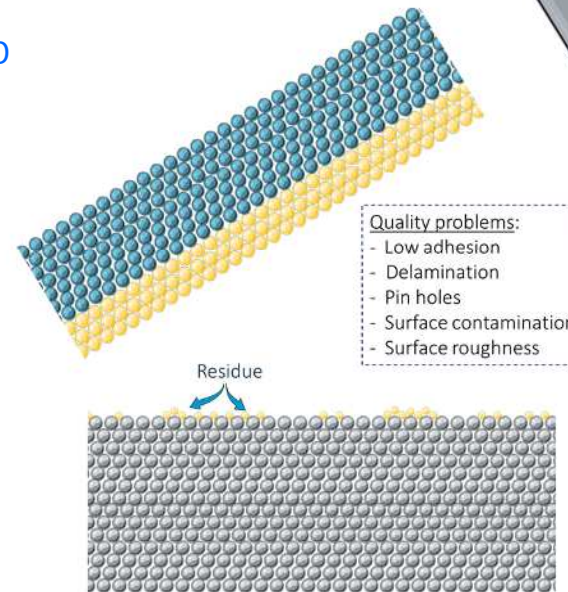
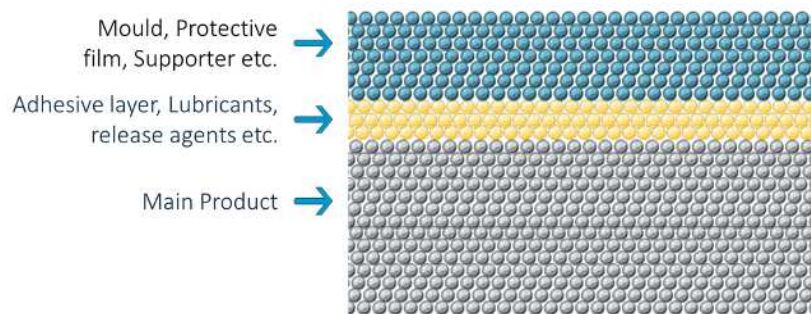
Method with the highest detection sensitivity

Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS)

Irradiate the sample surface with primary ions to generate secondary ions for analysis. The primary ion dose is kept low to minimize surface damage, allowing the detection of molecular ions and fragment ions that reflect the molecular structure.

Detection sensitivity: ppm – ppb

Depth resolution: nm



B. Identifying Chemical Composition of Tiny Foreign Substances for Quality Control

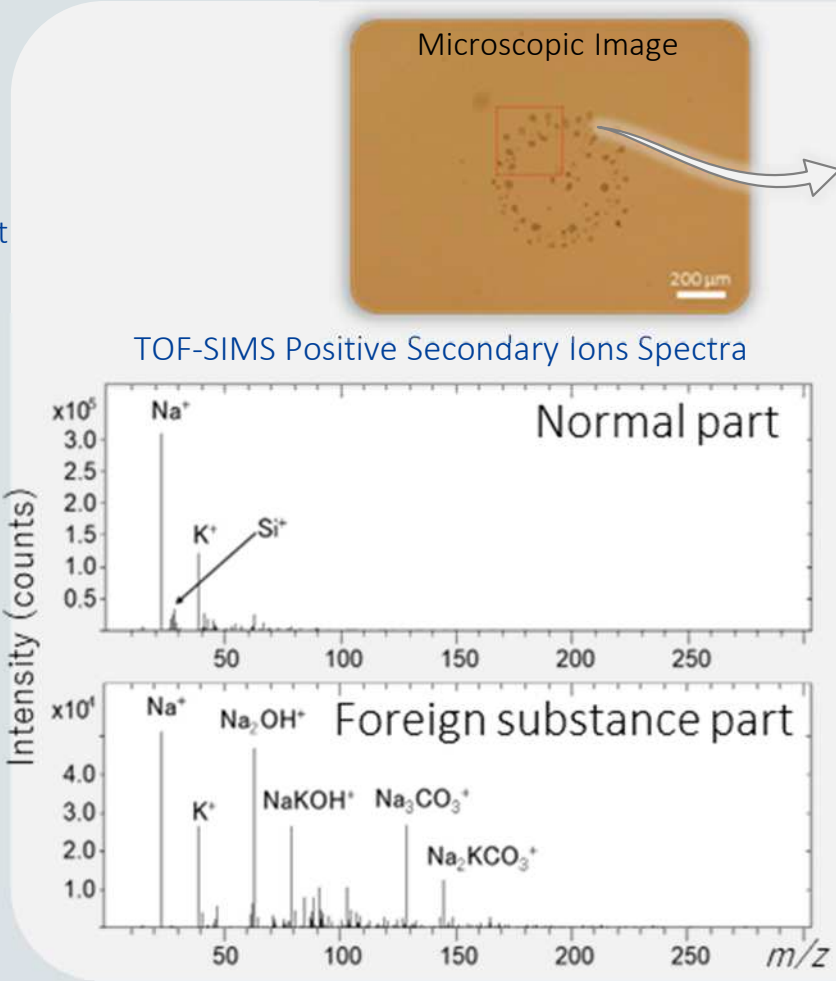
Case 3

Step 3:

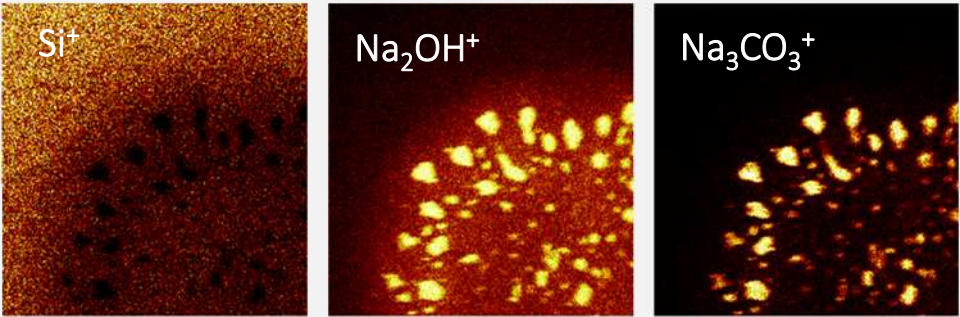
Unnecessity
of sample-
pretreatment

Step 4:

Analysis



TOF-SIMS Ion Imaging



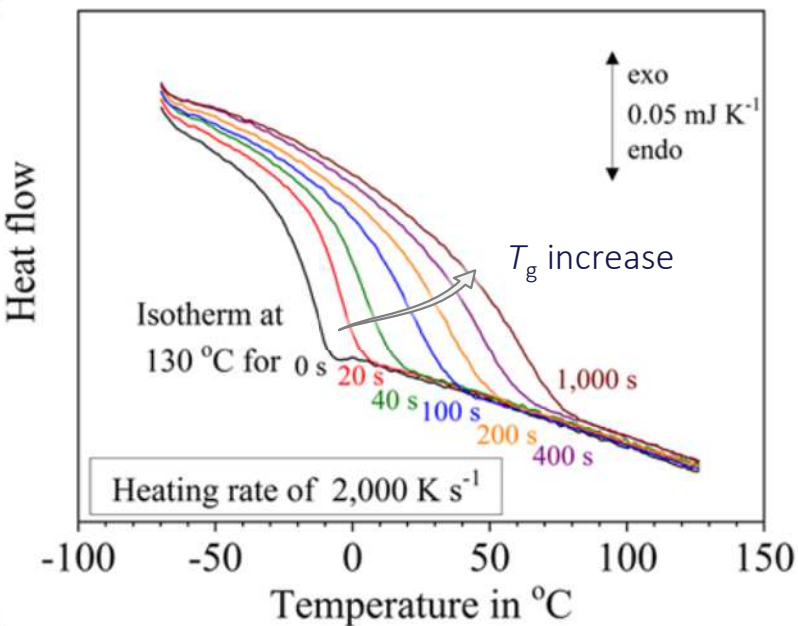
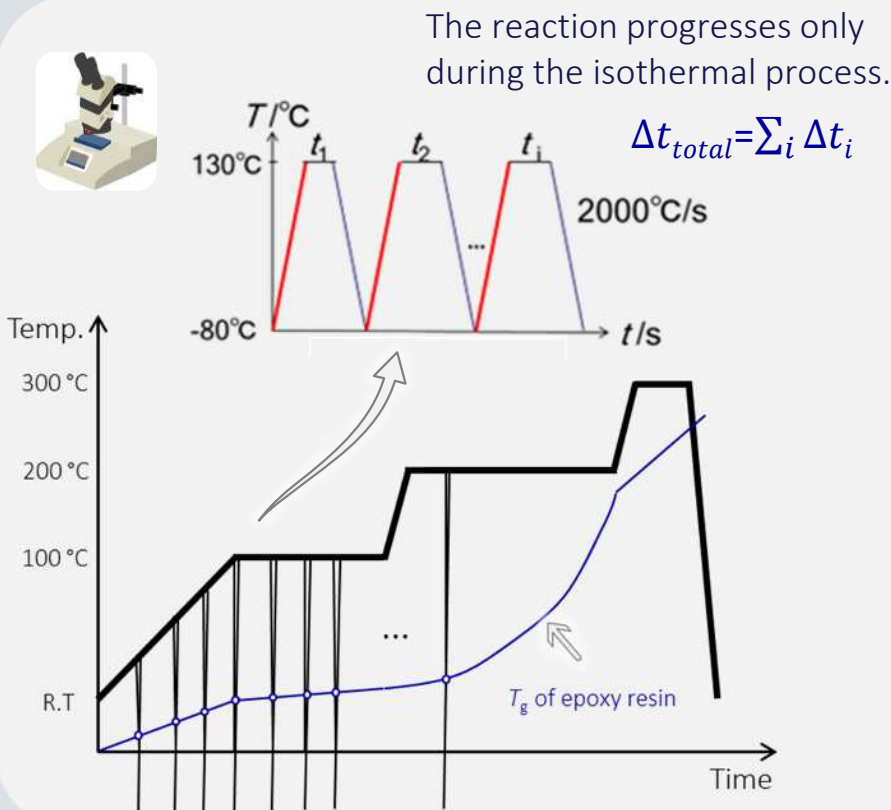
Conclusion

Presumably, sodium carbonate.
Ion imaging reveals the presence of adhesions both in the vicinity of the foreign material and within the area containing the material itself.

C. Optimization of Molding Process of Thermosetting Plastics by FSC

- Determine the temperature profile for the epoxy curing process

Thermal Profile: Monitoring Glass Transition (T_g) Shift at 130° C



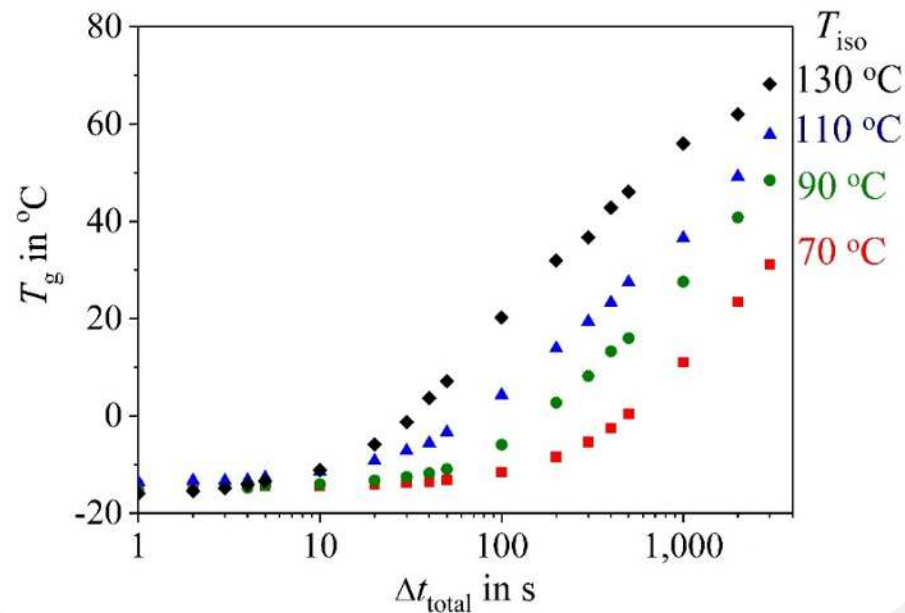
The step-like T_g signal shifts to higher temperatures as curing time increases, indicating progress in the curing process.

C. Optimization of Molding Process of Thermosetting Plastics by FSC

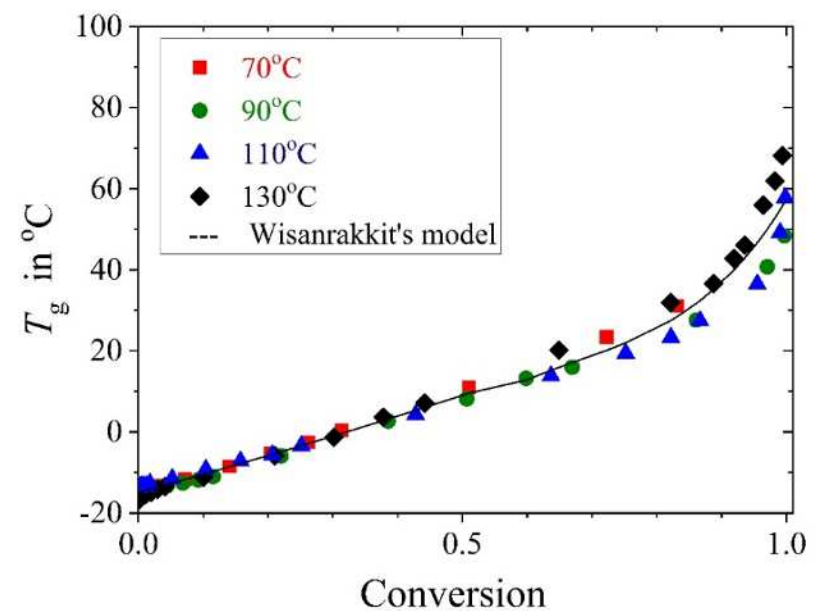
- Determine the temperature profile for the epoxy curing process

Thermal Profile: Monitoring Glass Transition (T_g) Shift at 130 °C

The change in T_g can be accurately measured by precisely controlling time and temperature.



The correlation between T_g and conversion makes T_g a reliable indicator for monitoring the curing progress.



D. Reverse Engineering of Plastic Parts to Boost R&D

Why reverse engineering ?

- R&D

Why is the competitor's product better than ours?

- Quality control

How can we control the quality of our product?

- Patent search

Is a competitor's product in conflict with our patent?



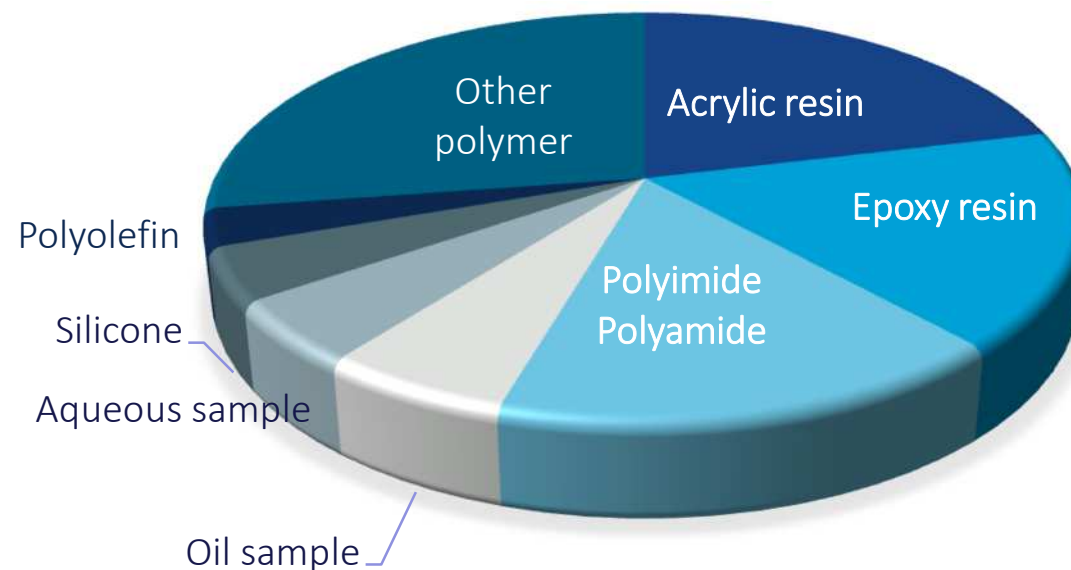
Our experience

more than **4000**



This is the number of organic materials that we have performed the compositional analysis from 2014 to 2023.

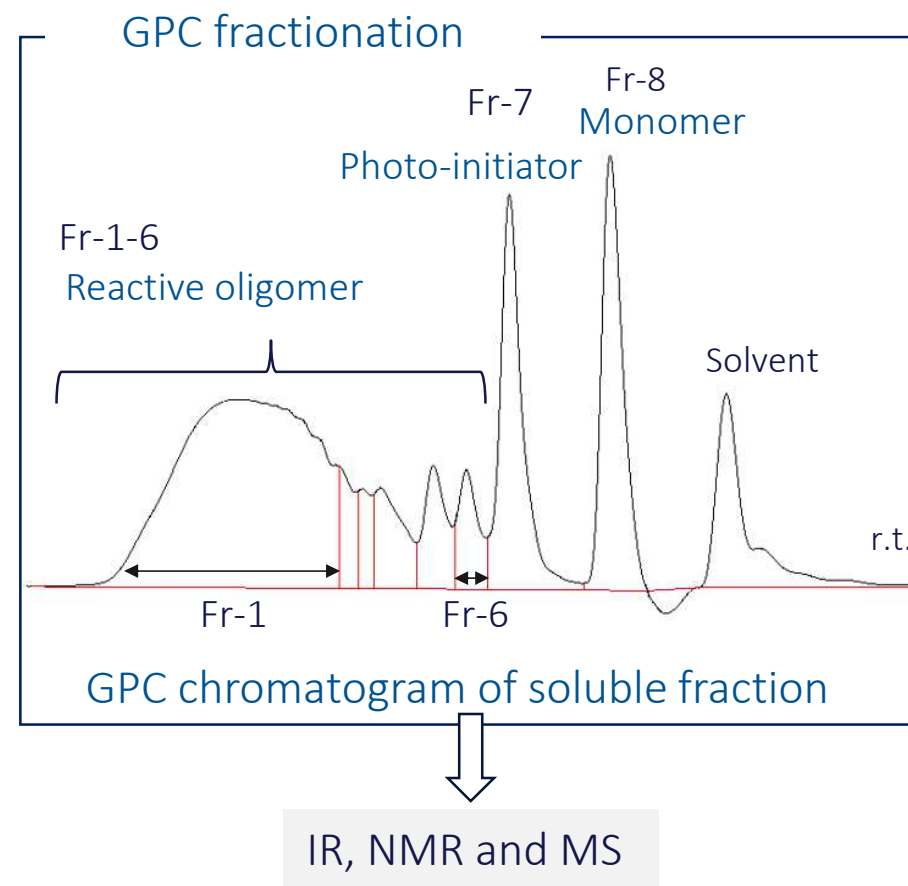
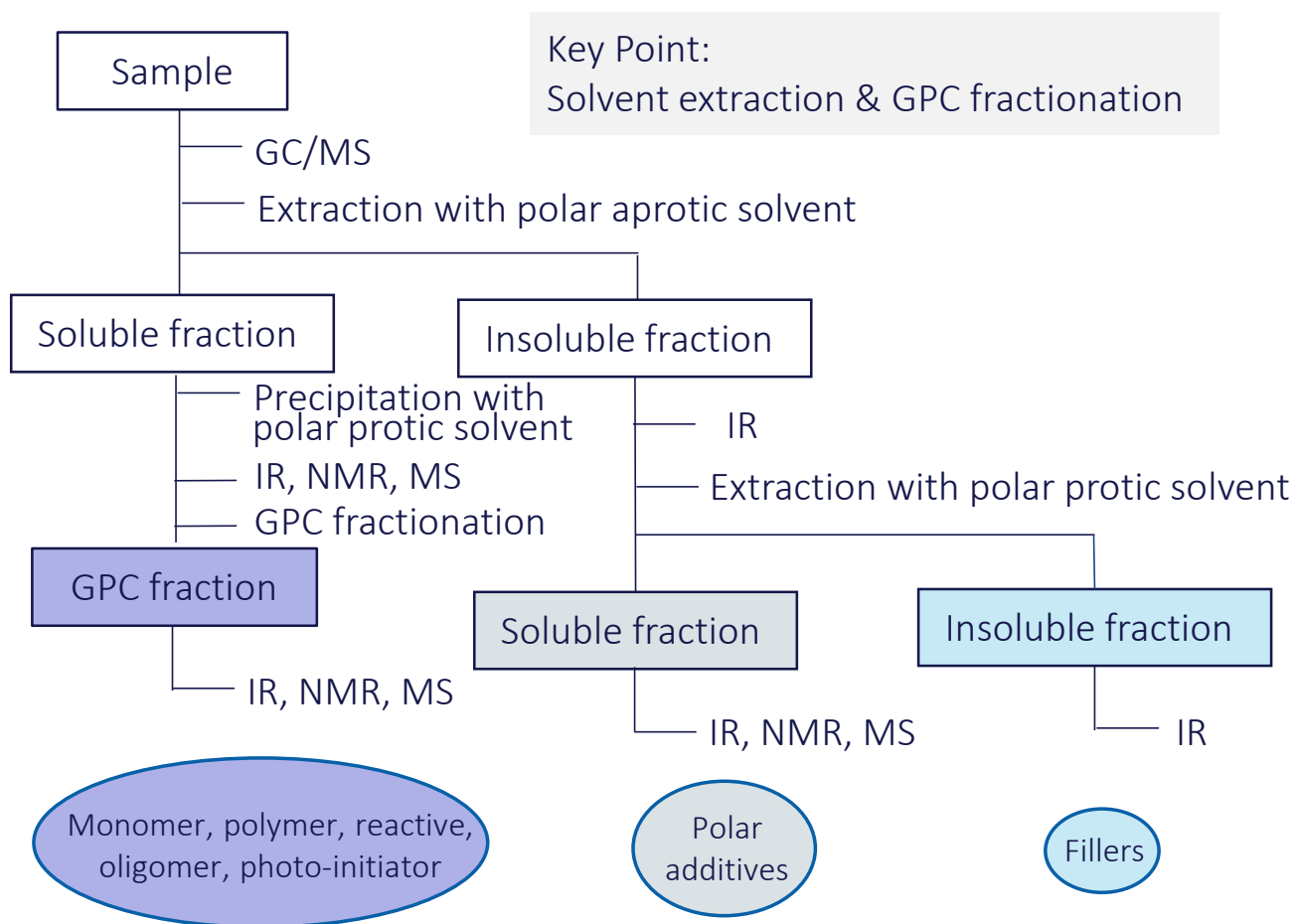
Polymer type



D. Organic Composition Analysis of Uncured UV Resin

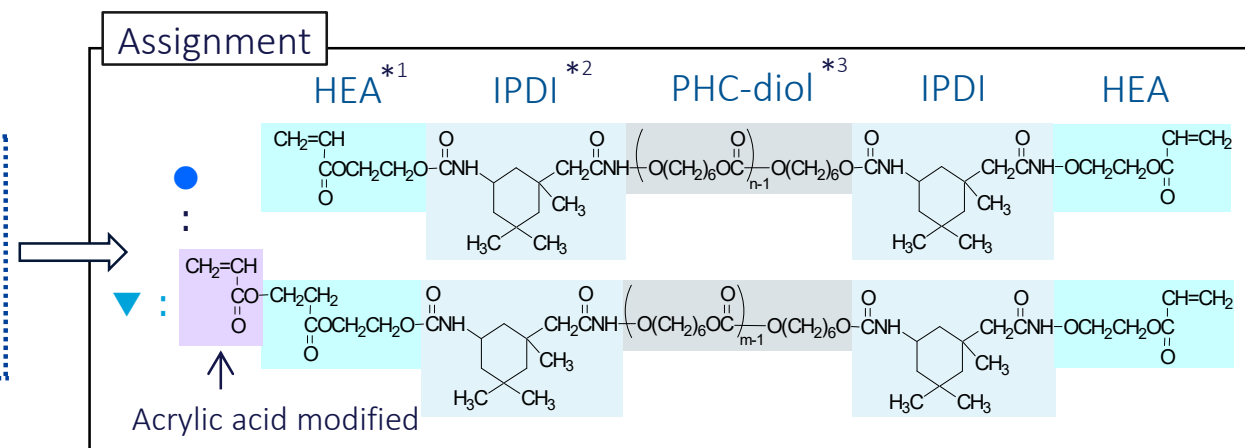
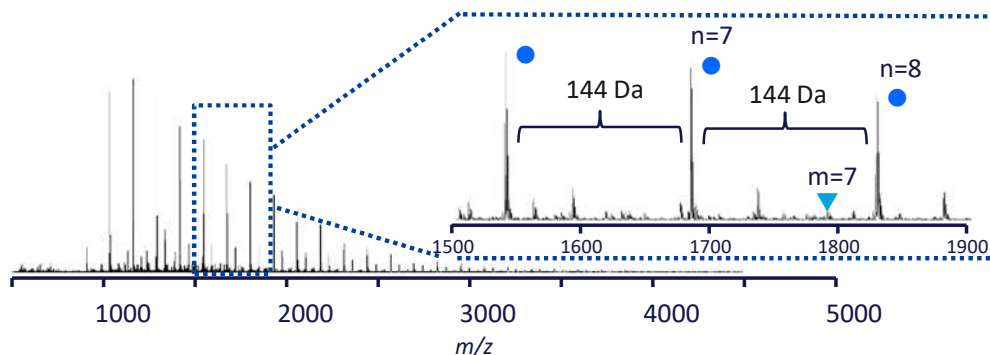
Analysis procedure

UV curable resin includes solvent, monomer, reactive oligomer, polymer, photo-initiator, additives and fillers.



D. Result of Reverse Engineering of Uncured UV Resin

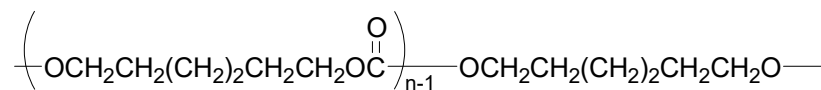
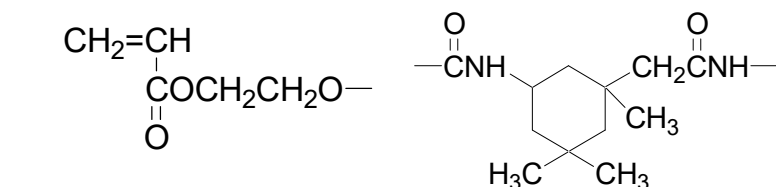
Ex.) Matrix Assisted Laser Desorption/Ionization-Mass Spectroscopy (MALDI-MS) of Fr-1



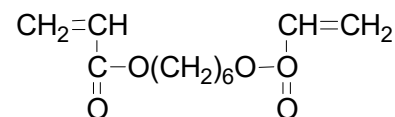
*1) 2-Hydroxyethyl Acrylate, *2) Isophorone Diisocyanate, *3) Polycarbonate Diol

Composition of uncured UV resin

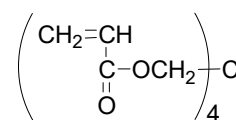
- Urethane acrylate (HEA, IPDI, PHC diol) 61%



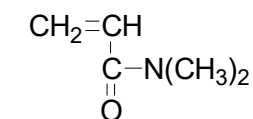
- Hexamethylene glycol diacrylate 22%



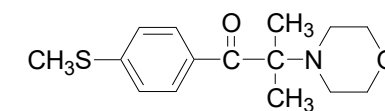
- Pentaerythritol tetraacrylate 5%



- N,N-Dimethylacrylamide 10%

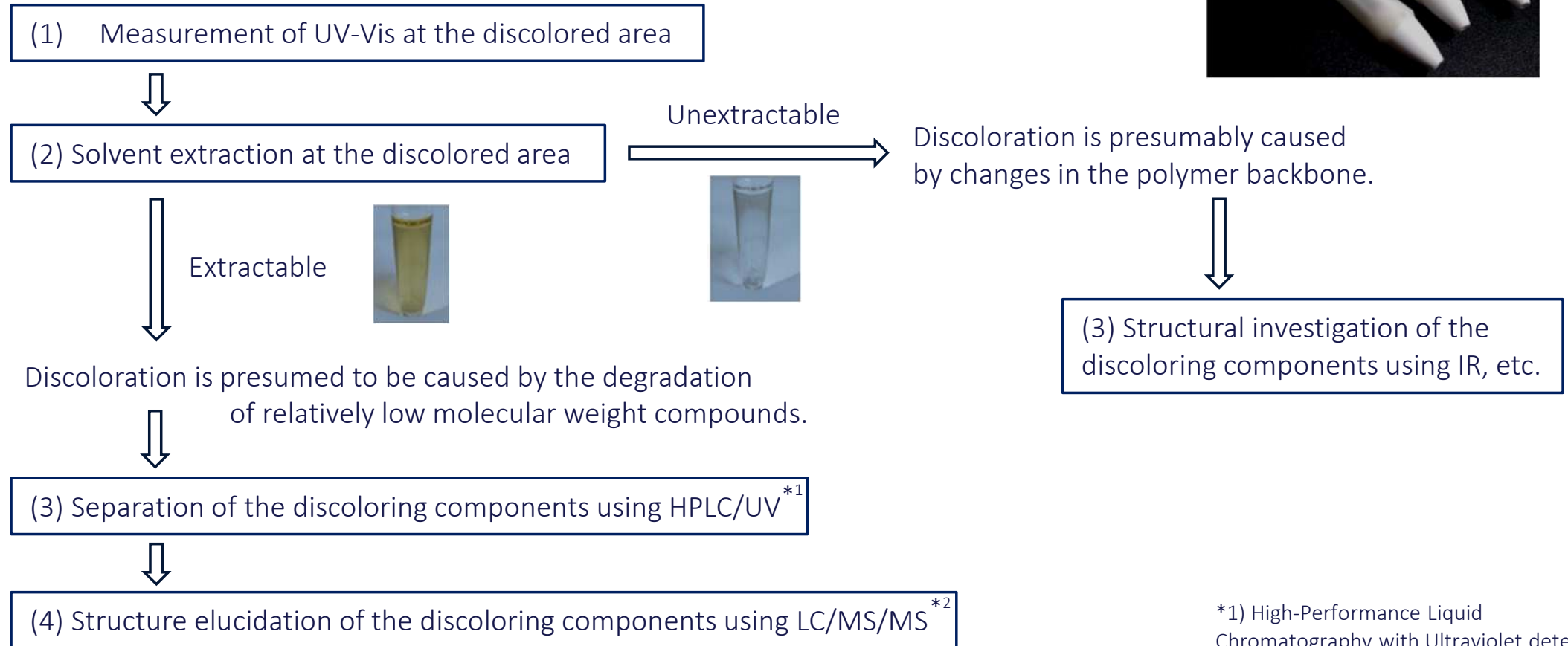


- Initiator 2%



E. Root Cause Analysis of Discoloration

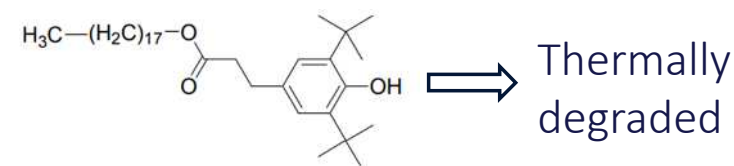
Approach for discoloration analysis



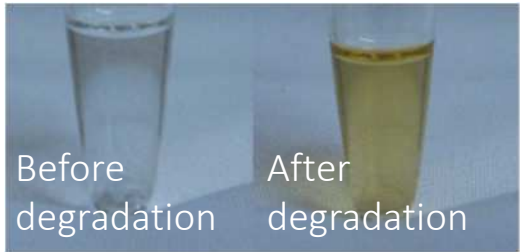
*1) High-Performance Liquid Chromatography with Ultraviolet detection
*2) Liquid Chromatography-tandem Mass Spectroscopy

E. Discoloration Caused by Degradation of Additive

Sample Phenolic antioxidant



Solvent extraction

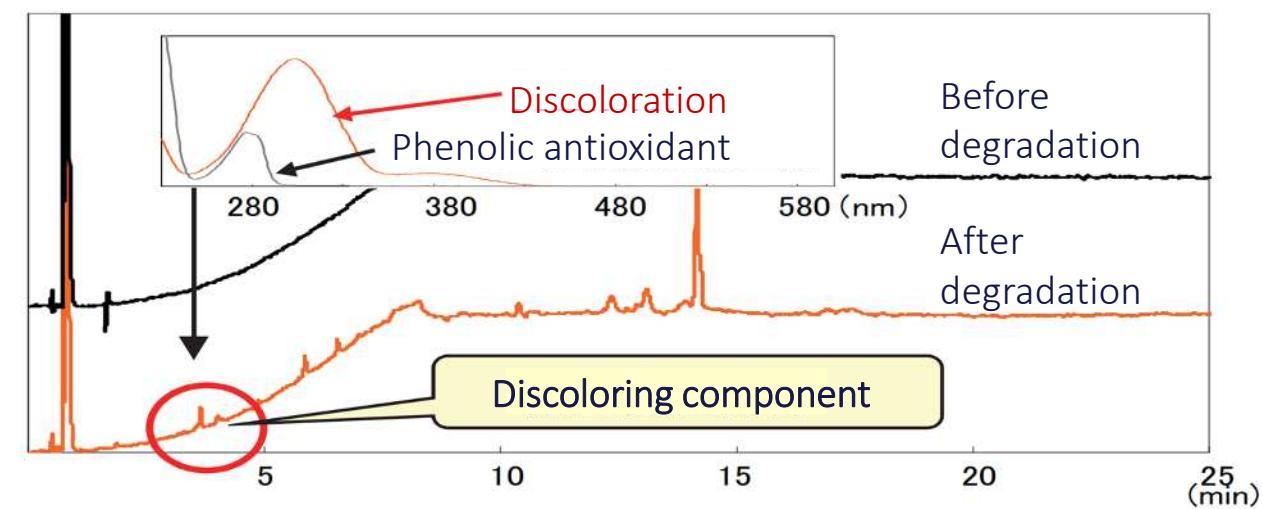


Extraction of substances



Low molecular weight compounds

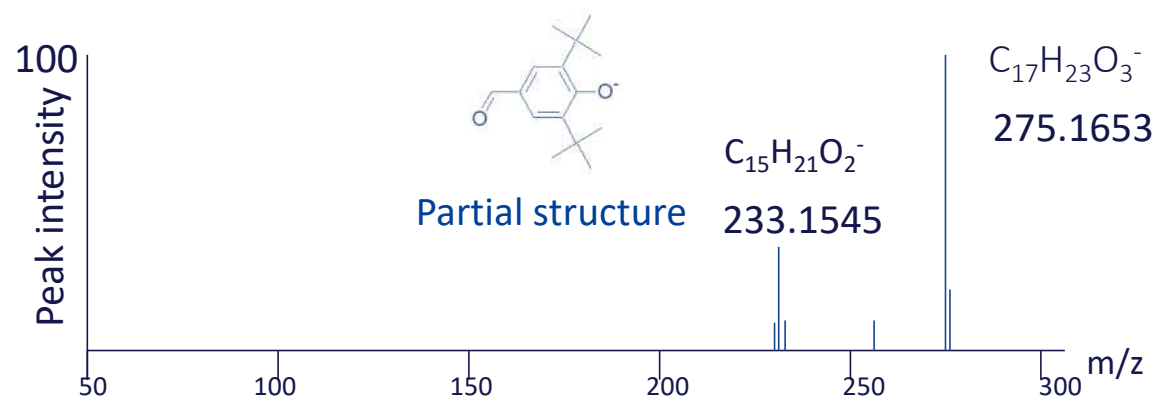
Liquid Chromatograph with Ultraviolet Detection



\Rightarrow High resolution LC/MS/MS analysis

E. High Resolution LC/MS/MS Analysis

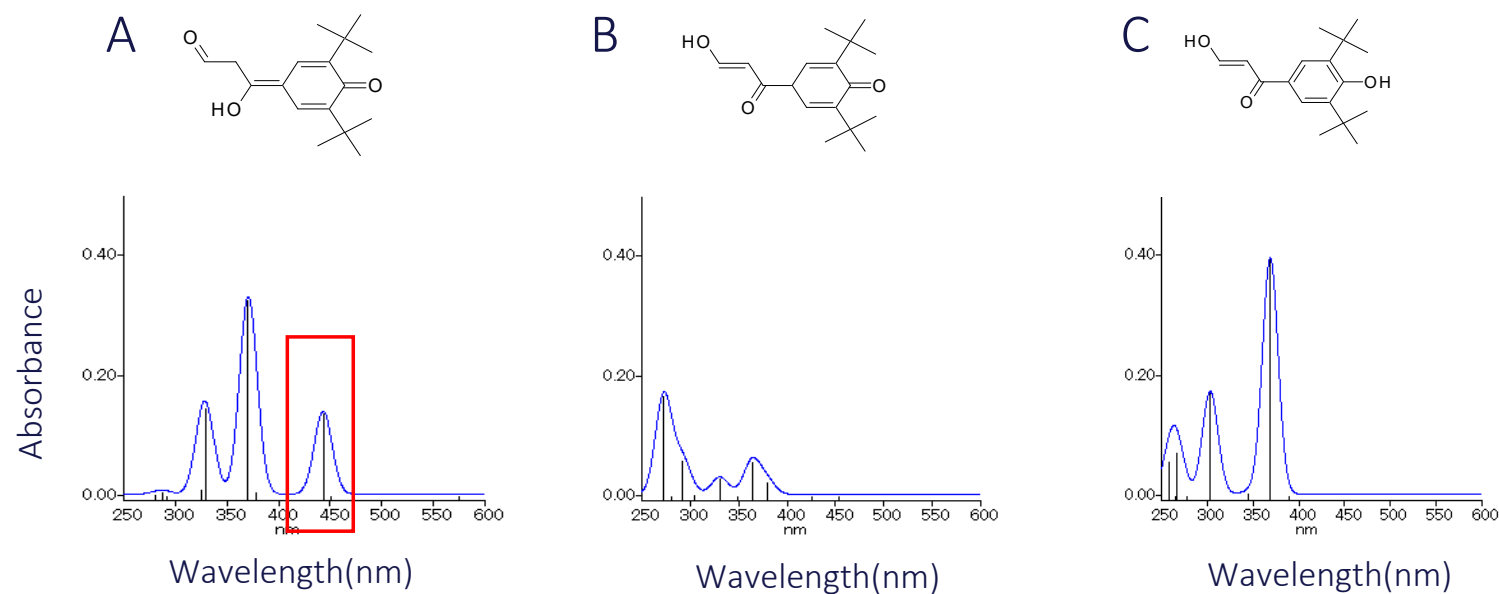
MS/MS analysis



Candidate structure



Simulation
(Molecular orbital method)



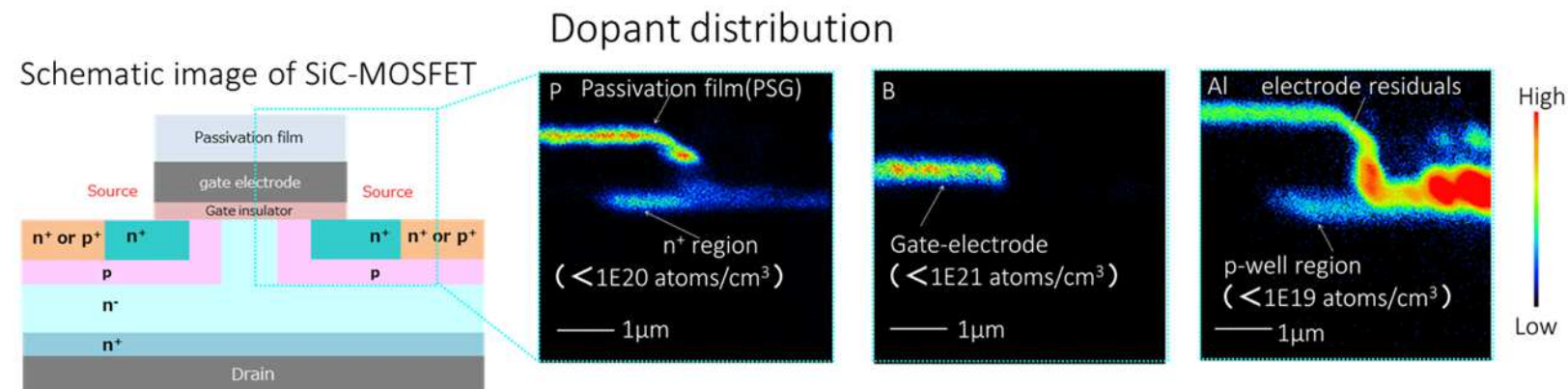
Wrap-up



Finally

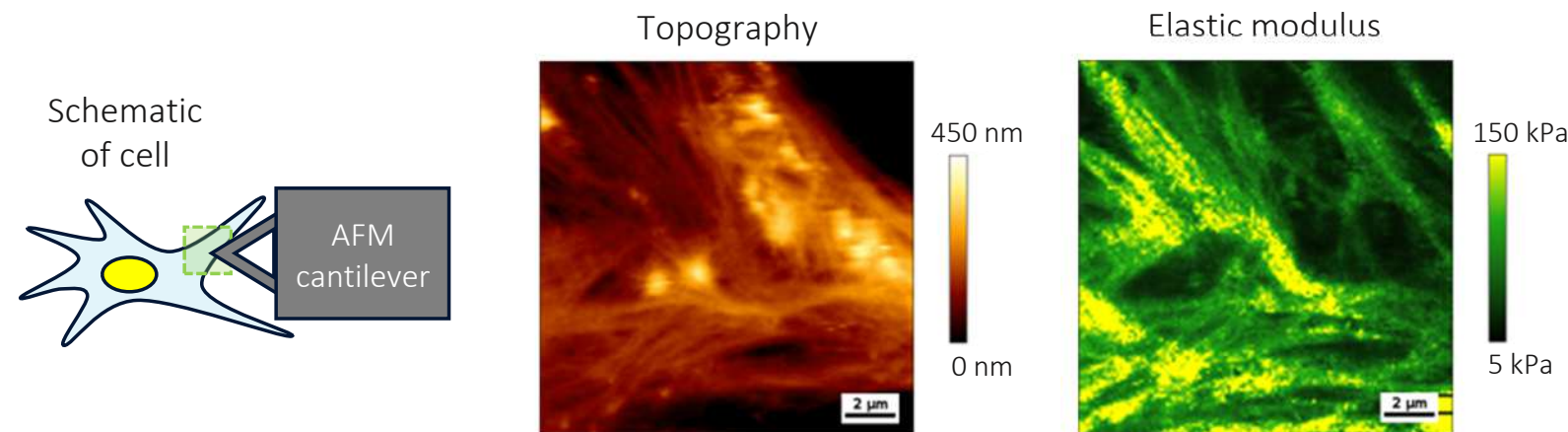
Semiconductor

Dopant distribution
by nano-SIMS



Life-science

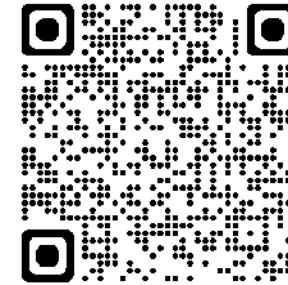
Imaging of living cell
by high-speed AFM



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