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FTIR
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Development of Multifunctional Composites using Conductive Polymers

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1. Introduction

What happens to airframe structures when lighting strikes? It is estimated that each aircraft is struck by lightning once per year. The aircraft is subjected to lightning both during flight and on the ground. The lightning current flows safely through the surface of airframe structures, which are conventionally made of conductive metals, to the ground, as aircraft have no lightning conductor rods. Please rest assured of the operational safety of the aircraft.

Recently, carbon fiber-reinforced plastics (CFRP) have gained widespread use for aerospace and automotive structures because of their light weight. CFRPs are comprised of conductive carbon fibers and insulating polymers (e.g. epoxy), leading to lower conductivity compared to metals. Anisotropic characteristics and local variations of properties generally make the design and quality control tremendously complex and difficult.

Structural CFRPs with epoxy matrix are severely damaged when subjected to lightning strikes, as shown in Fig. 1. Therefore, metal films or meshes are attached to the surface of airframe CFRP structures to prevent lightning damage. The value of CFRPs as lightweight structures diminishes when adding metal layers as these layers increase weight and manufacturing costs. To tackle this problem, we conducted experiments applying intrinsically conductive polymers as the matrix of CFRPs [1,2].

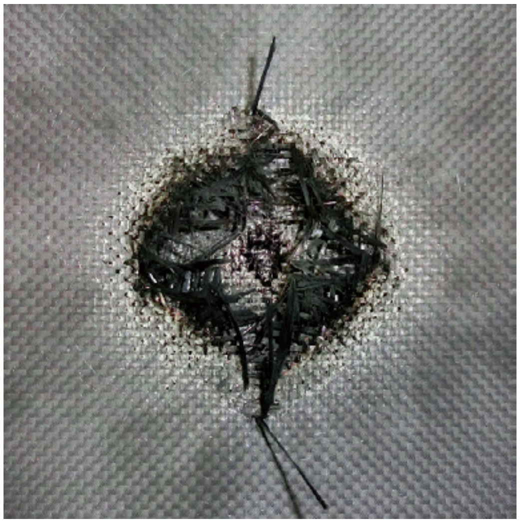

Fig. 1 Surface image of CFRP with epoxy matrix subjected to a lightning strike

2. Conductive Polymer Matrix

Intrinsically conductive polymer (ICP) can be used as a conductive materials in the same way as metals and carbon-based materials. ICPs (e.g. Polyacetylene, polypyrrole, polyaniline (PANI), poly (3,4-ethylenedioxythiophene) (PEDOT)) are insulating themselves, however, exhibit conductivity by doping with acid group. Specifically, PANI is stable in the air, affordable, highly conductive and available as a mass-production material, meaning that PANI is a well-balanced material. This study utilizes PANI as an ICP. Although ICPs are widely used in the electronics field, no examples for structural application have been found. It is necessary to develop a suitable manufacturing process for the CFRP application by considering the conductivity, mechanical properties, and manufacturability.

The doping process of PANI with acid groups is required to make PANI conductive (Fig. 2). Mixing of PANI with a curable polymer with high rigidity results in a matrix with high mechanical properties. We utilized dodecylbenzenesulfonic acid (DBSA) as a dopant and divinylbenzene (DVB) as a cross-linking polymer. The PANI/DBSA/DVB complex is doped and cured by heat application, resulting in a cured conductive polymer, which can be applied to CFRPs as a matrix.

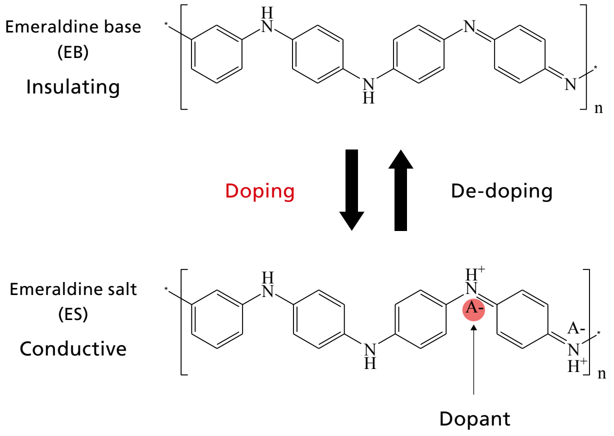

Fig. 2 Doping of PANI

3. Development of mixing process of conductive polymer complex

We developed a simultaneous doping and curing process for the PANI/DBSA/DVB complex to make a conductive polymer with high conductivity and good mechanical properties. In the process, DBSA is first mixed with PANI and the complex is doped partially (e.g. by applying heat), followed by the mixing of DVB and thermal curing of the whole complex [3,4]. As the first mixing and partial doping process of PANI/DBSA, thermal doping or shear mixing by roll-milling is applied with various parameters. The doping ratio for various processes is evaluated using FT-IR (Shimadzu Corporation, IRAffinity-1S, ATR spectroscopy).

FT-IR spectra are shown in Fig. 3 and the representative peaks are summarized in Table 1. In this figure or table, peaks corresponding to 1245 [cm^{-1}] (ν(C-N$^+$)) and 1100 [cm-^{1}] (δ(-NH$^+$=)) allowed us to evaluate the doping ratio. The relative peak strength (δ(-NH$^+$=)/ν(C-N$^+$)) was calculated based on the corresponding peaks in Fig. 3. The process with higher relative value was selected for the optimal mixing process of PANI/DBSA.

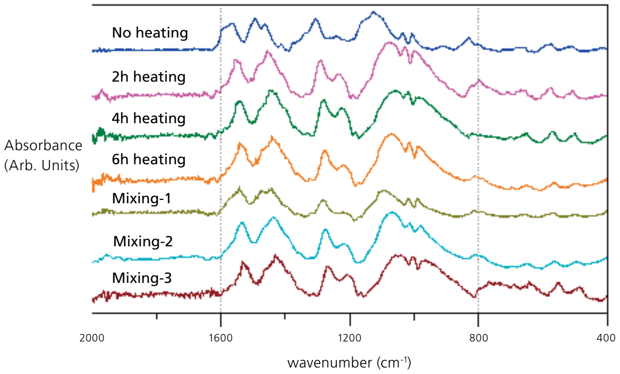

Fig. 3 Comparison of FT-IR spectrum of PANI/DBSA complex with various process conditions

Table 1 FT-IR peaks of PANI/DBSA (unit: cm^{-1})

	Q ($\nu_{C=N}$)	B ($\nu_{C=C}$)	ν (C-N)	ν (C-N$^+$)	δ (-NH$^+$=)	γ (C-H)
No heating	1568	1494	1304	1242	1126	839
2h heating	1556	1457	1292	1234	1078	796
4h heating	1542	1450	1282	1226	1056	813
6h heating	1536	1441	1275	1219	1071	812
Mixing-1	1543	1441	1281	1219	1094	813
Mixing-2	1534	1434	1274	1218	1067	807
Mixing-3	1533	1431	1267	1209	1031	768

ν (stretching vibration), δ (scissoring vibration), γ (bending vibration)

4. CFRP using conductive polymer matrix

In addition to investigating the mixing process as indicated in section 3, rheological properties, curing properties and mechanical properties of cured polymers were evaluated. We successfully developed a CFRP using a conductive polymer matrix. The lightning strike tests of the developed CFRP were performed using the same conditions as with the epoxy matrix (shown in Fig. 1). Fig. 4 indicates that almost no damage is found on the developed CFRP and demonstrates the significant improvement of resistance against a lightning strike by using a conductive polymer matrix.

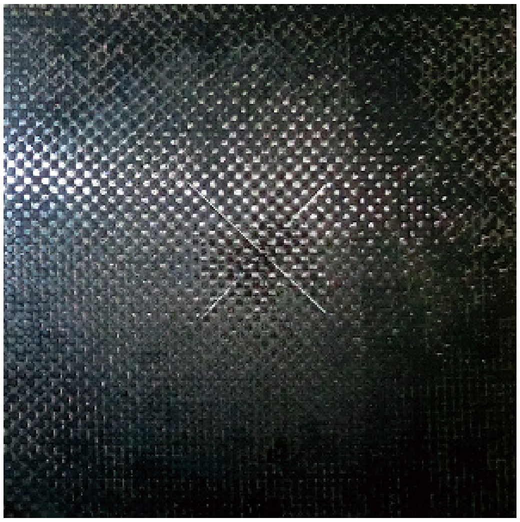

Fig. 4 Surface image of CFRP with conductive polymer matrix subjected to a lightning strike (X indicates the lightning entry point)

5. Summary

This article presents technology to mitigate lightning damages on CFRPs. It is expected that lightning strike protection layers for CFRP structures (e.g. metal mesh) can be reduced or removed, which greatly contributes to the reduction of weight, manufacturing cost, maintenance cost, and risk of galvanic corrosion. We have more tasks to improve the mechanical properties and the manufacturability of CFRPs with conductive polymer and continue to research this composite. It can be concluded that a CFRP with a conducive polymer matrix is one of the most attractive multifunctional structural materials.

References

[1] V. Kumar, T. Yokozeki, et al., "Mechanical and electrical properties of PANI-based conductive thermosetting composites", Journal of Reinforced Plastics and Composites, 34, 1298,(2015).

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FTIR Interferometers

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1. Introduction

Until around 1960, dispersive spectrometers that dispersed light with prisms and diffraction gratings were the prevailing technology in infrared spectroscopy, and Fourier transform infrared spectrometers that used interferometers were limited to very few special use cases. As fast Fourier transform (FFT) processing became easier thanks to improvements in the processing capabilities of computers, Fourier transform infrared spectroscopy, which can use brighter optical systems than dispersive spectrometers, increased in popularity and now prevails in mid-infrared spectroscopy.

This FTIR TALK LETTER provides a brief overview of the interferometer used in infrared spectroscopy.

2. Interference of Light

Before describing an interferometer, this article must first touch on the phenomenon of light interference. Light has the properties of a wave, and when two light beams of the same frequency overlap, the intensity of those beams changes. This phenomenon is called light interference, of which Young's interference experiment is a well-known example (Fig. 1).

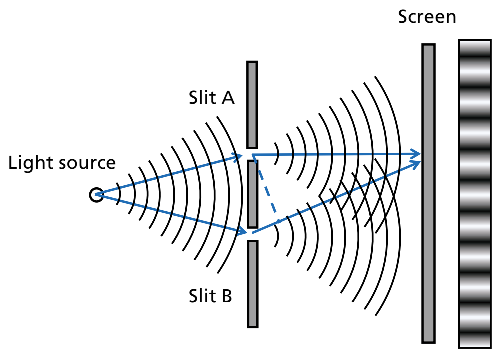

Fig. 1 Young's Interference Experiment

When two slits are placed in front of a single light source, and a screen placed behind the two slits, a pattern of bright and dark fringes appears on the screen. A bright fringe appears when the difference in distance from the two slits to the screen (optical path length difference) is equal to an integer multiple (n) of the light wavelength (λ), and a dark fringe appears when the optical path length difference is equal to an integer multiple of half the light wavelength ($\lambda/2$). This phenomenon arises because light has the properties of a wave, whereby when a single beam of light is split and then recombines, its intensity is dependent on the difference in path length between the two beams.

3. Interferometers Used in Infrared Spectrophotometers

This section covers interferometers used in infrared spectrophotometers. The interferometer most commonly used in Fourier transform infrared spectroscopy is called the Michelson interferometer. The Michelson interferometer is comprised of an optical material (beam splitter, BS) that transmits half the incoming infrared light and reflects the remaining half, and two total-reflection mirrors (Fig. 2), one of which is fixed (stationary mirror) and the other movable (moving mirror). In practical terms, infrared light from a light source that enters the interferometer is first split into two beams by the beam splitter, with one beam sent in the direction of the stationary mirror and the other in the direction of the moving mirror. Each beam is then reflected by each total-reflection mirror back to the beam splitter, where the returned light beams combine and are sent from the interferometer toward the detector.

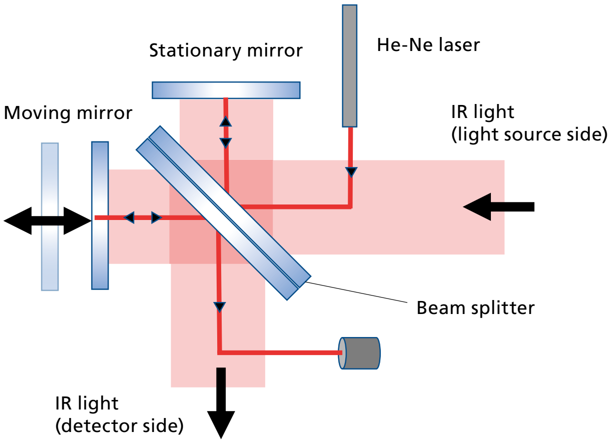

Fig. 2 Schematic Diagram of an Interferometer

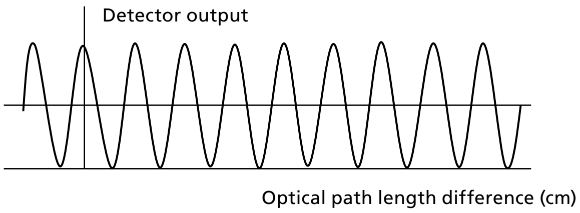

Fig. 3 Interference of Monochromatic Light

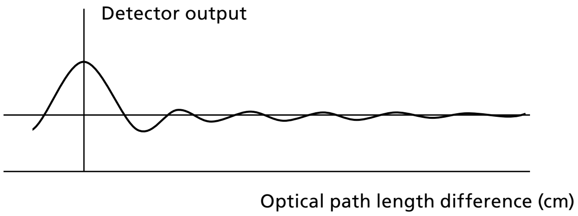

Fig. 4 Interference of White Light

This next section covers the detector output observed at the light receiving detector surface when the moving mirror is moved. First, to simplify conditions, the light incident on the detector is assumed to be monochromatic light of a single wavelength. When the moving mirror is moved, it creates an optical path length difference between light from the moving mirror and the stationary mirror. Light is reinforced and detector output is increased where the optical path length difference is an integer multiple of the light wavelength, and conversely, output is decreased where the optical path length difference is an integer multiple of half the light wavelength (Fig. 3). This is the same pattern of interference observed on the screen in Young's experiment and is a manifestation of the properties of light as a wave.

Next, the light incident on the detector is assumed to be white light (a mixture of light with a wide range of frequencies). Although moving the moving mirror repeatedly creates an increasing/decreasing detector output in accordance with the changing optical path length difference, as was observed with monochromatic light, with white light a maximum detector output occurs at the point of zero optical path length difference (called the center burst). The output amplitude quickly decreases as the optical path length difference moves away from zero (Fig. 4). When this effect is shown graphically with detector output signal on the vertical axis and optical path length difference on the horizontal axis, as shown in Fig. 4, it creates what is called an interferogram (IFG). (Interferograms normally do not show the direct current component.) The infrared spectra obtained by infrared spectroscopy are created by applying a discrete Fourier transform to IFGs.

4. IFG Sampling

The previous section "3. Interferometers Used in Infrared Spectrophotometers" described how to acquire an IFG. However, in order to apply a discrete Fourier transform, the data must be acquired at fixed intervals of optical path length difference. Also, for highly accurate spectra, this interval must be around several hundred nanometers and vary by ≤0.1 % (Fig. 5).

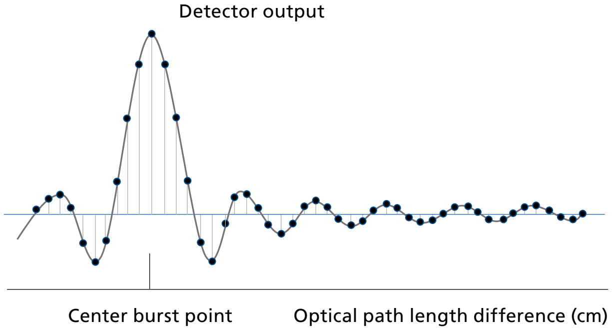

Fig. 5 IFG Sampling Example

Because of these strict requirements, a helium-neon (He-Ne) laser that produces monochromatic light at a stable wavelength is used in interferometers, as shown in Fig. 2, where light from the He-Ne laser enters the beam splitter (BS) and undergoes the same interference as infrared light. As with the monochromatic light shown in Fig. 3, the signal detected from a He-Ne laser increases and decreases at fixed intervals according to optical path length difference, where the optical path length difference is varied by moving the moving mirror. This signal is binarized and used to trigger data acquisition, thereby enabling the acquisition of IFGs at intervals of half the wavelength of light output by the He-Ne laser (approx. 314 nm). The highly stable wavelength of light produced by the He-Ne laser also enables an acquisition interval variability of ≤0.1 %. Recently, the wavelength stability of semiconductor lasers has improved, and some infrared spectrophotometers now use semiconductor lasers instead of He-Ne lasers.

5. IFG Reproducibility

This section covers IFG reproducibility. Even when sampling is undertaken as described in the previous section, IFG variation can occur and negatively affect reproducibility. Although this IFG variation has a variety of possible causes, the principal cause is believed to be variation in the orientation of the moving mirror. In the Michelson interferometer, variation in the angle of the moving mirror during mirror movement changes the position of reflected light and alters the area of light superimposed on light from the stationary mirror (Fig. 6). This changes the interference intensity of the IFG. This variation in the moving mirror angle also has an element of randomness, which negatively affects IFG reproducibility.

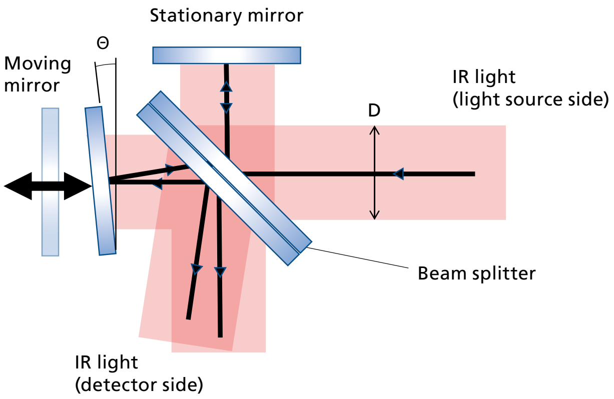

Fig. 6 Variation in Moving Mirror Orientation inside the Interferometer

In terms of actual numbers, the tilt θ of the moving mirror requires the following:

$$\theta < \frac{\lambda min}{20D}$$

where λmin is the minimum measured wavelength and D is the diameter of the infrared light beam. This makes the shift in the wavefront of infrared light beam caused by moving mirror tilt smaller than 1/10th. Based on this equation, when the minimum wavelength is 4,000 cm^{-1} and the infrared light beam diameter is 5 cm, θ becomes 2.5 μrad (0.5 arcsec). This value is sufficiently small compared to the normal amount of pitching in a linear guideway (10 arcsec) and has a major effect on measurements. For this reason, Shimadzu employs a mechanism (called dynamic alignment) that measures the variation in angle of the moving mirror and concurrently adjusts the angle of the stationary mirror in order to maintain the interference. In practical terms, the tilt of the moving mirror is measured, and this measured amount is transmitted as a signal to a piezo actuator positioned behind the stationary mirror. This mechanism works to maintain the stationary mirror at the same tilt as the moving mirror, reducing IFG variation (Fig. 7).

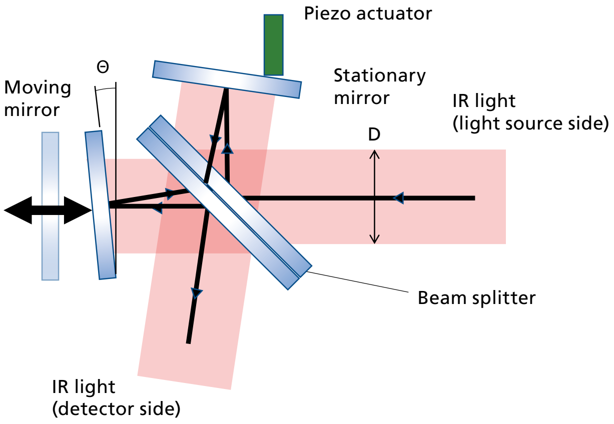

Fig. 7 Dynamic Alignment inside the Interferometer

6. Summary

This article described interferometers used in Fourier transform infrared spectrometers. Shimadzu uses He-Ne laser length measurement, dynamic alignment, and other technologies that allow its interferometers to be used at higher accuracies.

Although the interferometer was invented over 100 years ago and all avenues of development appear to be exhausted, interferometer technology continues to develop to this day. A particularly well-known example of this development is the laser interferometer gravitational-wave observatory (LIGO), essentially a large interferometer, which in 2015 became the first project to detect gravitational waves first predicted by Albert Einstein in 1916. This goes to show that, despite being old technology, the interferometer is still in ongoing development. We hope this article piqued your interest in the topic of interferometers.

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Fourier Transform Infrared Spectroscopy—FT-IR for Chemists
Editor: Jiro Hiraishi, Gakkai Shuppan Center
Fundamental and Practical FT-IR
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Analysis of Plastic Materials Using FTIR

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Plastic products of all shapes and sizes are around us. From containers and packaging to aircraft, plastics have an increasingly wide range of uses thanks to qualities that make them lighter than metal and glass and afford them with excellent durability. Nevertheless, there is also substantial discussion about reducing the impact of plastics on the environment, such as cutting back on the use of oil, the main raw material used in plastics, reducing carbon dioxide emissions, waste management, and the problem of microplastics.

Understanding plastic materials is important both for the development of new plastic materials and proper recycling practices. This article describes analytical procedures, analysis examples, and products that play a vital role in Fourier transform infrared (FTIR) spectrophotometry, a simple technique for analyzing plastics.

1. Introduction

The word plastic generally refers to synthetic resins, of which there are many different types. Given the prevailing viewpoint on cutting back on the use of oil, which is the main raw material of plastics, and recent serious concerns about microplastics, there are increasing efforts to promote the recycling of plastic products and to use biomass plastics and biodegradable plastics. Understanding the properties and chemical structure of plastics is important both for the development of new plastic materials and proper recycling practices. Gas chromatograph mass spectrometers (GC-MS), nuclear magnetic resonance (NMR) spectrometers, and a variety of other instruments are used to analyze plastics. Of these, FTIR spectrophotometers are in widespread use because they allow for simple analysis without the need for complex pretreatments such as dissolution in solvents.

2. Procedure for Plastic Analysis Using FTIR

A procedure for plastic analysis using FTIR is shown in Fig. 1.

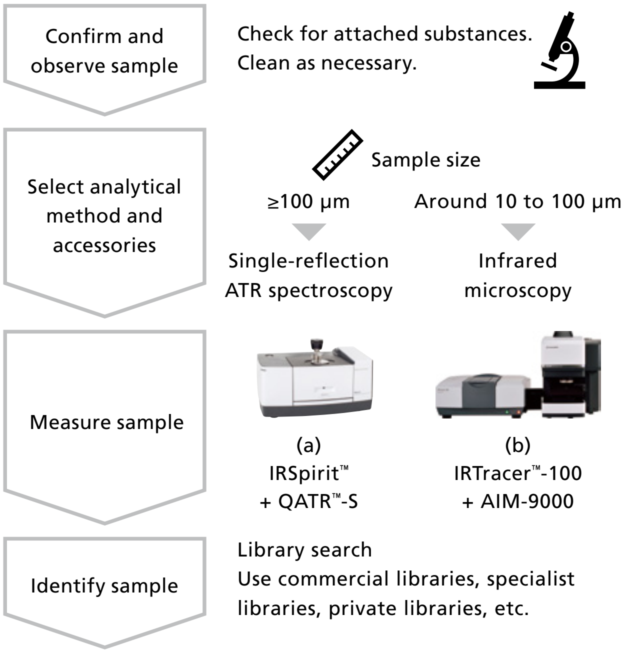


Fig. 1 Procedure for Plastic Analysis Using FTIR

First, the sample is observed and checked for attached substances. If needed, the sample is cleaned with water, ethanol, hydrogen peroxide solution, or another substance. (If a sample is a contaminant that appears plastic-like, it may be possible to estimate the specific source of the contaminant by forgoing cleaning and comparing measurements taken from areas with and without the attached substance.)

Next, an analytical technique is selected in light of the sample size. Samples of around 100 µm or larger are analyzed on a system that comprises a main FTIR unit connected to a single-reflection ATR attachment. Fig. 1 (a) shows an example system, where the QATR™-S single-reflection ATR attachment with integrated sample chamber is connected to the IRSpirit™ Fourier transform infrared spectrophotometer. One advantage of ATR is its prism located on the same plane as the top face of the instrument, which allows for the measurement of large flat samples as they are. In addition to the ATR attachment with integrated sample chamber, ATR attachments are also available with a built-in camera to allow sample observation and with heating during measurement. For more details, please refer to FTIR TALK LETTER vol. 32 and 33 that cover the various ATR attachments and other relevant information. Samples of around 10 to 100 μm are analyzed using an infrared microscope. (See Fig. 1 (b) for an example system.) Measurements are performed by transmission microscopy or by ATR microscopy based on the condition of the sample. Regarding microplastics, an area of recent interest, please refer to FTIR TALK LETTER vol. 35 for a detailed description of a microplastics analytical method.

After acquiring an infrared spectrum, the sample is identified using a library (database) search. Although libraries that come with the instrument and commercial libraries contain major plastics, specialized libraries for additives and damaged plastics or private libraries created by the user can also be used.

3. Example Analysis of Everyday Plastics

3.1 Example Analysis of Biomass Plastic

Japan is working to reduce its plastic usage and, from July 1, 2020, began charging for plastic shopping bags1). Charging for plastic shopping bags that retail stores once provided free-of-charge is expected to reduce excessive plastic usage. Based on their acceptable environmental performance, three types of plastic bags were made exempt from this change: (1) bags made of plastic 50 μm or thicker, (2) bags containing 100 % marine-degradable plastic, and (3) bags containing 25 % or more biomass material1).

There are now increasing cases of retail stores giving out free plastic bags that contain biomass materials. A single-reflection ATR spectroscopy was used to analyze plastic bags that display the biomass mark to indicate the use of biomass materials. The biomass marks displayed on respective plastic bags used for analysis are shown in Fig. 2, and the results obtained from analyzing each bag are shown in Fig. 3.

Fig. 2 Biomass Marks Displayed on Plastic Bags

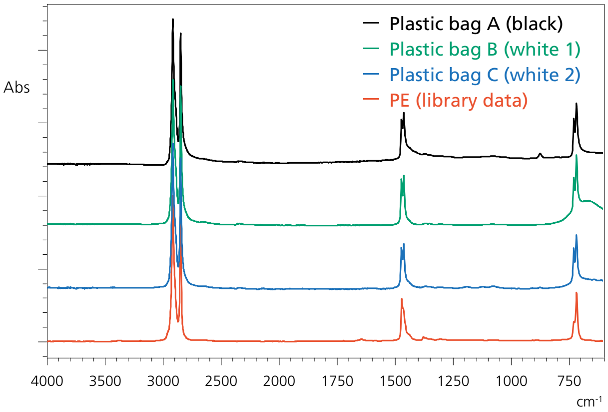


Fig. 3 Results Obtained from Analyzing Plastic Bags

The "biomass 30" mark indicates the product contains at least 30 % and under 35 % biomass as raw materials. The major peaks from all three bags matched the library data for polyethylene (PE) where PE is the bottom line shown in Fig. 3, which confirmed that all bags are made of polyethylene as indicated by their labeling. These results showed polyethylene made with biomass material has a chemical structure identical to that of normal polyethylene, and the spectral patterns of the two types of plastic match each other.

3.2 Example Analysis of Biodegradable Plastic

"Biomass plastic" refers to plastic that contains biomass materials and does not indicate different chemical properties, while "biodegradable plastic" refers to plastic with the ability to degrade under biological exposure and be broken down by microorganisms into, eventually, just water and carbon dioxide. Fig. 4 shows the results obtained from using a single-reflection ATR spectroscopy to analyze a plastic bag marked as biodegradable and referencing this data against a library database.

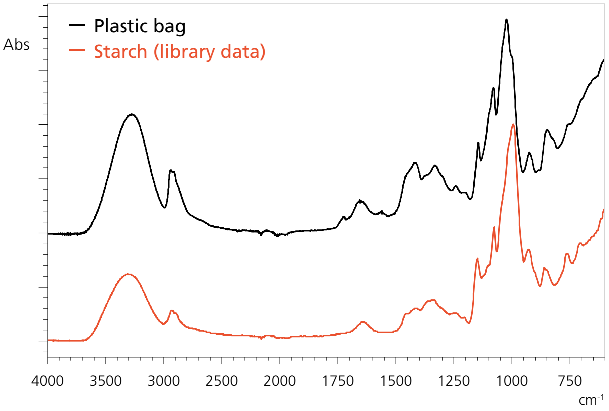


Fig. 4 Library Search Results for a Biodegradable Plastic Bag

The measured plastic bag spectrum resembled the library data for starch. Esterification and other processes are used to create commercial plastic products with a starch-based backbone structure, and the sample plastic bag appears to be produced from this same material. FTIR analysis was successfully used to determine the type of material used in a biodegradable plastic without material labeling.

Biodegradable plastics are also sometimes used in straws given free with coffee and other beverages. A straw made from a hydroxyalkanoate plastic (PHBH) and a straw made from normal polypropylene (PP) were analyzed with the single-reflection ATR spectroscopy. Fig. 5 shows the straws and Fig. 6 shows the spectra obtained by this ATR spectroscopy.

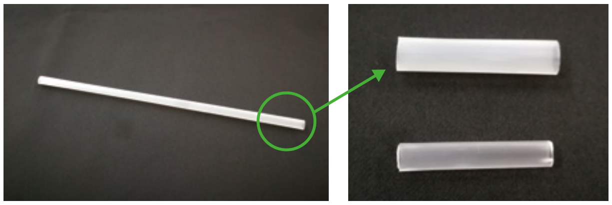


Fig. 5 PHBH Straw (Top Right) and PP Straw (Bottom Right)

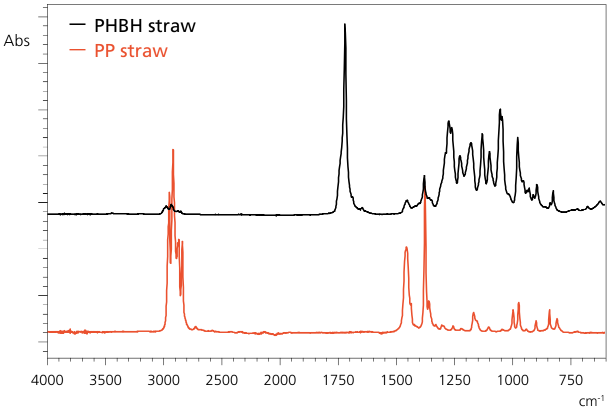


Fig. 6 Results Obtained from Analyzing the PHBH Straw and PP Straw

PHBH is a biomass-derived biodegradable plastic and at least as degradable as cellulose. The PHBH straw appears slightly cloudy compared to the polypropylene straw, but has the same surface luster as a normal straw and is indistinguishable based on appearance alone. Comparing ATR spectra revealed a substantial difference in spectral pattern between the two straws, showing the PHBH straw and PP straw can be easily distinguished by FTIR analysis. Because biodegradable plastics are unsuitable for recycling, this finding showed that FTIR-based analysis is an effective means of separating biodegradable plastics to prevent their inclusion with recycled plastics.

4. Example Analysis of Damaged Plastic

Plastics can undergo deformation, discoloration, or degradation from environmental factors such as heat and ultraviolet (UV) rays. This section describes an example analysis of damaged plastic using a window shade that has yellowed after extended exposure to sunlight. The window shade was installed indoors at a window, and ATR analysis was performed on a window-facing surface and an internal part cut from the window shade. The analyzed sample is shown in Fig. 7 and the results obtained from analysis are shown in Fig. 8.

Fig. 7 Window Shade

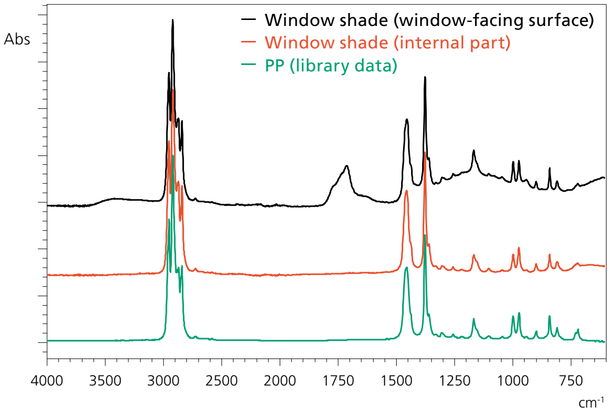


Fig. 8 Results Obtained from Analyzing the Window Shade Overlaid with Polypropylene

The internal part not exposed to light matched well with polypropylene (PP) from the library, but the window-facing surface did not match PP at around 1750 cm^{-1}. The window-facing spectral data was also referenced against the UV-damaged plastics library (results shown in Fig. 9).

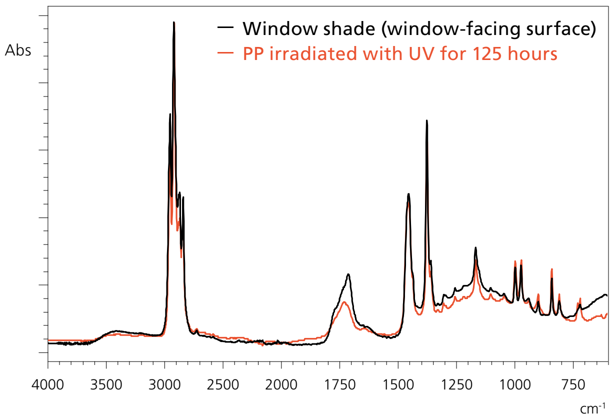


Fig. 9 Library Search Result for Window Shade (Window-Facing Surface)

Polypropylene exposed to 125 hours of UV rays showed the same absorption as the window shade (window-facing surface) at around 1750 cm^{-1} and the spectral shapes were also matched at low wavenumbers. Therefore, it can be inferred that yellowing of the window shade (window-facing surface) was due to damage caused by extended exposure to UV rays.

The UV-damaged plastics library is a proprietary Shimadzu library that contains the infrared spectra of plastics subjected to UV damage. Fourteen different plastics were irradiated with UV rays for up to 550 hours (equivalent to approx. 10 years of UV exposure) in a super accelerated weathering tester manufactured by Iwasaki Electric Co., Ltd.

Degraded plastics often produce an infrared spectral pattern different from that of normal products, as observed with the window shade, and are difficult to identify by referencing commercial spectral libraries. Using libraries that are populated with spectra obtained from plastics degraded by UV irradiation allows for highly accurate sample identification that reflects the degraded state of the sample plastic.

This analysis was performed using Plastic Analyzer, a system specifically developed for plastics analysis. The Plastic Analyzer consists of the IRSpirit and QATR-S shown in Fig. 1 (a), macro programs for analysis, and a method package that includes both the thermal-damaged plastics library and UV-damaged plastics library. For more details, refer to the product brochure (C103-E130) or Application News No. A647. Plastic Analyzer is an effective system for quality control, contaminants analysis, and damage analysis of plastics.

5. Summary

This article described procedures and examples of FTIR analysis of plastics. Using the single-reflection ATR attachment enables easy identification of various plastics. By using specialized libraries, even samples with spectral patterns altered by degradation can be identified with precision. Please consider Shimadzu's FTIR products for plastics analysis.

Reference

1) Fee-Incurring Plastic Checkout Bags Starts on July 1, 2020 (METI/Japanese Ministry of Economy, Trade and Industry) (Accessed: November 12, 2020)
https://www.meti.go.jp/policy/recycle/plasticbag/plasticbag_top.html

Fourier Transform Infrared Spectrophotometer Plastics Analysis System

Plastic Analyzer

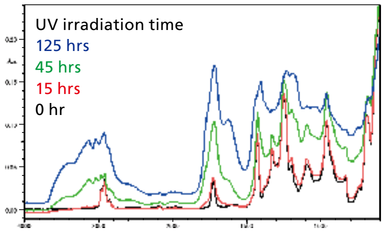
Plastic Analyzer consists of the following:

- IRSpirit Fourier transform infrared spectrophotometer
- QATR-S single-reflection ATR attachment
- Plastic Analyzer method package
  1. UV-Damaged Plastics Library 2. Thermal-Damaged Plastics Library 3. Macro Program for IR Pilot™/Parameter Files

The Plastic Analyzer method package includes an FTIR spectral library for plastics degraded by UV rays and heat. Utilizing searches of this library demonstrates its effectiveness in the analysis of unknown samples that are difficult to identify with standard libraries. Examples include plastics degraded by exposure to UV rays as well as contaminants and defective items altered by heating.

UV-Damaged Plastics Library

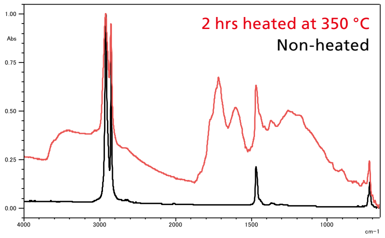
This library includes more than 300 spectra from the UV degradation of 14 types of plastic. This proprietary Shimadzu library includes the IR spectra for plastics degraded by UV rays for the equivalent of approximately 10 years using a super accelerated weathering chamber from Iwasaki Electric Co., Ltd.



Hard PVC Spectra Listed in
the UV-Damaged Plastics Library

Thermal-Damaged Plastics Library

This library includes more than 100 spectra from the degradation of 13 types of plastic heated to between 200 and 400 °C. This proprietary Shimadzu library contains IR spectra for plastics degraded by heating, acquired through measurements at the Hamamatsu Technical Support Center at the Industrial Research Institute of Shizuoka Prefecture.



Polyethylene Spectra Listed in
the Thermal-damaged Plastics Library

Microplastic Analysis for Microscopic Sizes

Fourier Transform Infrared Spectrophotometer IRTracer™-100 (left)
Infrared Microscope AIM-9000 (right)

Infrared Microscope AIM-9000

For microplastic analysis of several tens to several hundred micrometers in size, it is possible to quickly determine the plastic components and additive components by using an infrared microscope capable of qualitative analysis of organic substances and some inorganic substances.

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