

Characterization of Fluoropolymers Using FTIR and TG-DTA to Support the Growth of 5G

Risa Fuji and Atsuko Naganishi

User Benefits

- ◆ Evaluate structural changes and thermal stability of materials during heating by using FTIR and TG-DTA techniques.
- ◆ Enhance the efficiency of measurements while heating the sample using the GladiATR™ high-performance ATR.
- ◆ The DTG-60H simultaneous thermogravimetric differential thermal analyzer detects minute changes in mass, allowing use with a wide variety of materials.

Introduction

5G (fifth-generation mobile communications system) is a new mobile communications system featuring high speed, high capacity, low latency, and multiple simultaneous connections. The adoption of 5G will enable the instantaneous review of vast amounts of big data, such as data collected by IoT devices, and AI-based analysis and training of AI with this data. This, in turn, will accelerate the development of remote healthcare services, home healthcare, automated driving, and congestion forecasting and introduce new technologies and services into our lives.

The growth of 5G requires that smartphones and other terminal devices use high-frequency printed circuit boards (PCBs) with good electrical characteristics. This need has created a growing interest in new materials that can replace FR-4 (a PCB substrate material made of glass cloth impregnated with epoxy resin) and glass polyimide substrates that use polyimides. Two typical examples of these replacement materials are liquid crystal polymers (LCPs) and fluoropolymers. This article describes using Fourier-transform infrared spectroscopy (FTIR) to evaluate structural changes in fluoropolymers upon heating and thermogravimetry-differential thermal analysis (TG-DTA) to evaluate the change in mass of fluoropolymers upon heating.

Fluoropolymers

Fluoropolymer is a generic term for synthetic plastics obtained by the polymerization of fluorine-containing olefins. Due to their low dielectric constant (permittivity), low hygroscopicity, and excellent insulating and thermal characteristics, fluoropolymers are utilized in high-frequency PCBs where they support the growth of 5G.

One of the fluoropolymers, polytetrafluoroethylene (PTFE), was used in this experiment. Fig. 1 shows the structure of PTFE.

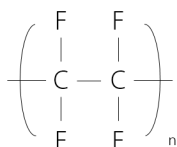


Fig. 1 PTFE Structure

Evaluating Structural Changes upon Heating by FTIR

FTIR was used to evaluate structural changes in PTFE upon heating. Measurements were taken using the IRTracer-100 shown in Fig. 2 and the GladiATR high-performance ATR accessory (PIKE Technologies) shown in Fig. 3. The measurement conditions used are shown in Table 1.

The GladiATR is an ATR accessory that provides high energy throughput, high applied pressure, a wide wavenumber range, and an optional crystal plate compatible with high temperatures. Two heating plates with maximum temperatures of 210 °C and 300 °C are available for the GladiATR; the 300 °C plate was used in this experiment. A programmable temperature controller that can be controlled from a computer is also available for the GladiATR.



Fig. 2 IRTracer™-100



Fig. 3 GladiATR™

Table 1 Measurement Conditions

Equipment:	IRTracer-100 GladiATR (diamond prism)
Resolution:	4 cm ⁻¹
Accumulation:	40
Wavenumber Range:	4,000 to 400 cm ⁻¹
Apodization Function:	SqrTriangle
Detector:	DLATGS

PTFE was heated at 200 °C for 30 minutes and at 300 °C for 60 minutes, 120 minutes, and 180 minutes and analyzed by FTIR for structural changes. Fig. 4 shows an overlay of each recorded infrared spectrum.

The main peaks and peaks indicative of oxidation were unchanged even when PTFE was heated at 300 °C for 180 minutes. This confirms that the chemical structure of PTFE was unchanged upon heating, a result that underscores the excellent thermal resistance of PTFE.

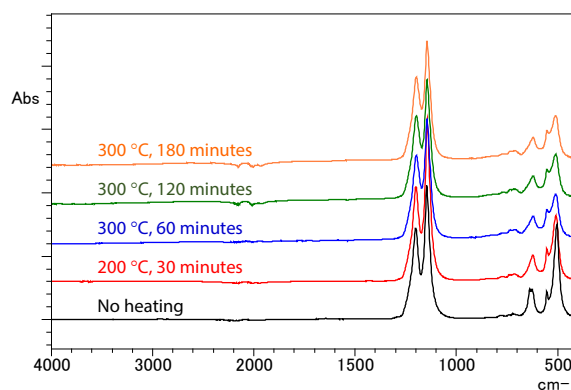


Fig. 4 Infrared Spectra of PTFE

Evaluating Change in Mass upon Heating by TG-DTA

TG-DTA was used to evaluate changes in PTFE mass upon heating. The evaluation was performed using the DTG-60H shown in Fig. 5 and the measurement conditions shown in Table 2.

The DTG-60H can evaluate evaporation, decomposition, gas adsorption and desorption, and dehydration by altering the temperature of a sample at a fixed rate and measuring the resulting change in mass and differential temperature. The DTG-60H takes measurements across a wide temperature range from ambient to 1,500 °C.

PTFE was heated from 30 to 700 °C, and changes in mass and differential temperature were recorded. Fig. 6 shows the results of the TG measurement of PTFE in air.



Fig. 5 DTG-60H

The TG curve shows PTFE lost 5 % of its mass by 522.2 °C and 10 % of its mass by 533.9 °C. The differential temperature curve also shows that PTFE melted at 331.6 °C. The mass of PTFE remained unchanged up to around 500 °C, indicating that PTFE has a higher decomposition temperature and is more thermally stable than most plastics.

Table 2 Measurement Conditions

Equipment:	DTG-60H
Sample Weight:	9.870 mg
Atmosphere Gas:	Air
Gas Flowrate:	100 mL/min
Measured Temperature Range:	30 to 700 °C

Conclusion

Multiple aspects of the temperature characteristics of PTFE were evaluated by using FTIR to assess structural changes upon heating and using TG-DTA to assess changes in mass and differential temperature upon heating.

Analyzing thermal degradation by FTIR and decomposition temperature and decomposition behavior by TG-DTA is effective for evaluating new materials that can potentially be used as substrates in terminal devices and help grow 5G.

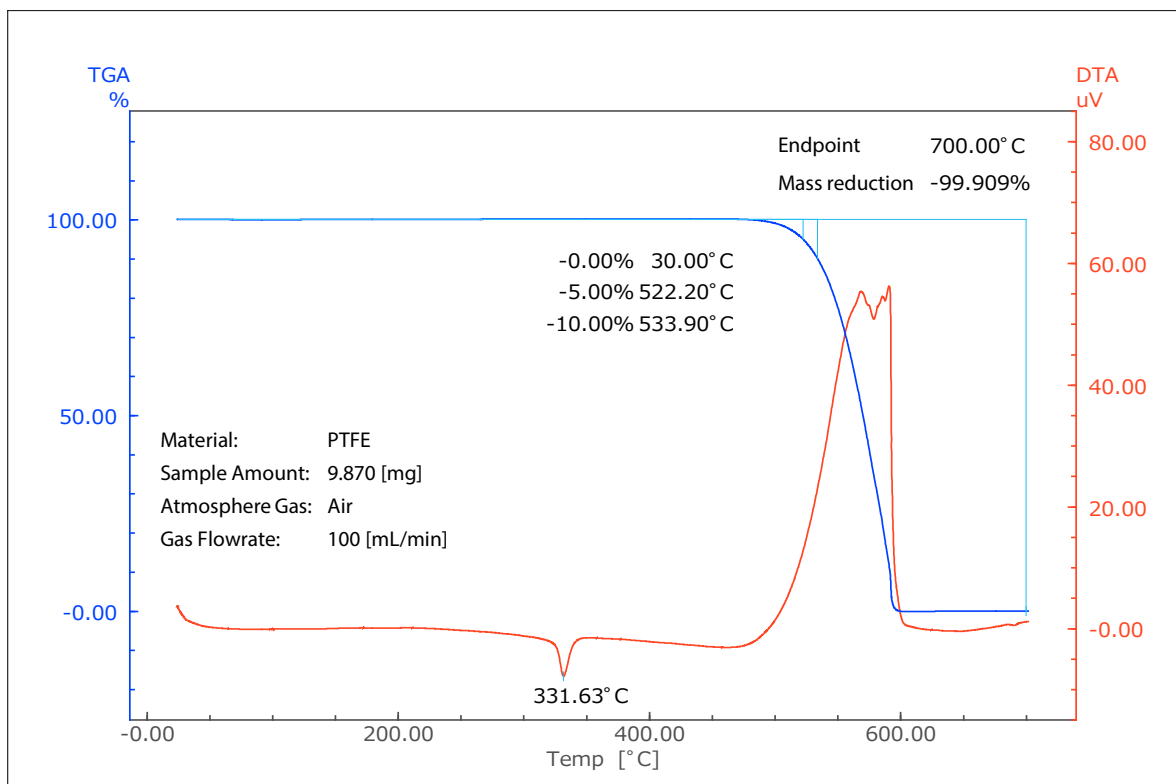


Fig. 6 TG-DTA Measurements from PTFE in Air

IRTracer is a trademark of Shimadzu Corporation in Japan and other countries.
GladiATR is a trademark of PIKE Technologies.



Shimadzu Corporation

www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.

This publication may contain references to products that are not available in your country. Please contact us to check the availability of these products in your country.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. See <http://www.shimadzu.com/about/trademarks/index.html> for details.

Third party trademarks and trade names may be used in this publication to refer to either the entities or their products/services, whether or not they are used with trademark symbol "TM" or "®".

Shimadzu disclaims any proprietary interest in trademarks and trade names other than its own.

The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

First Edition: Dec.2020
Revision A:Mar.2023