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Rapid analysis of carbon fiber reinforced plastic using DART-MS

ASMS 2014 TP 782

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Introduction

DART (Direct Analysis in Real Time) can ionize and analyze samples directly under atmospheric pressure, independent of the sample forms. Then it is also possible to measure in form as it is, without sample preparation. Qualitative analysis of target compounds can be conducted very fast and easily by combining DART with LCMS-2020/8030 which have ultra high-speed scanning and ultra high-speed polarity switching.
Carbon-fiber-reinforced plastics, CFRP is the fiber-reinforced plastic which used carbon fiber for the reinforced material, which is only called carbon resin or carbon in many cases. An epoxy resin is mainly used for a base material in CFRP. While CFRP is widely used taking advantage of strength and lightness, most approaches which measure CFRP with analytical instruments were not tried, triggered by the difficulty of the preparation.
DART (Direct Analysis in Real Time), a direct atmospheric pressure ionization source, is capable of analyzing samples with little or no sample preparation. Here, rapid analysis of carbon fiber reinforced plastic was carried out using DART combined with a mass spectrometer.

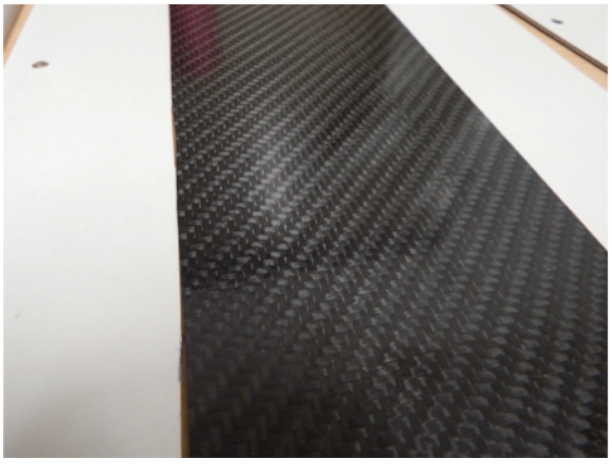

Figure 1 CFRP：carbon-fiber-reinforced plastic

Methods and Materials

Thermosetting polyimide (carbon-fiber-reinforced plastics) and thermoplastic polyimide (control sample) were privately manufactured. After cutting a sample in a suitable size, it applied DART-MS analysis. They were introduced to the DART gas using tweezers. The DART-OS ion source (IonSense, MA, USA) was interfaced onto the single quadrupole mass spectrometer LCMS-8030 (Shimadzu, Kyoto Japan). Ultra-fast polarity switching was utilized on the mass spectrometer to collect full scan data.
LCMS-8030 can achieve the polarity switching time of 15msec and the scanning speed of up to 15,000u/sec, therefore the loop time can be set at less than 1 second despite the relatively large scanning range of 50-1,000u.

MS condition (LCMS-8030; Shimadzu Corporation)

Ionization	: DART (Direct Analysis in Real Time)

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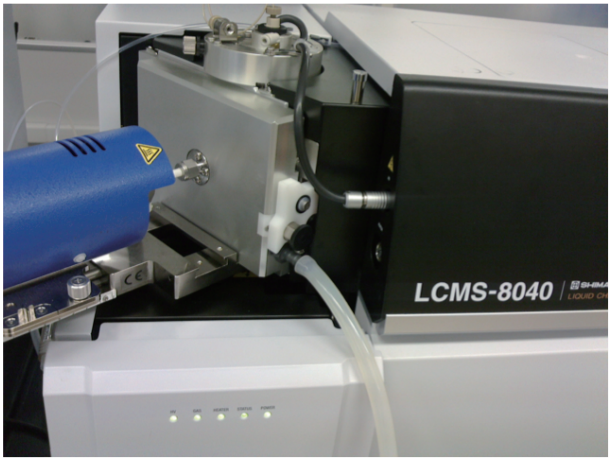

High Speed Mass Spectrometer

UFswitching
High-Speed Polarity Switching 15msec
UFscanning
High-Speed Scanning 15,000u/sec

Figure 2 DART-OS ion source (IonSense) & triple quadrupole LCMS (Shimadzu)

Result

3 CFRP samples were analyzed by DART-MS. Mass chromatograms of each sample were shown in Figure 3 and mass spectra in Figure 4.

Sample

#1 thermoplastic polyimide (control)
#2 thermosetting polyimide (molded; dried)
#3 thermosetting polyimide (immediately after molded; wet state with solvent)

Analytical Condition

Heater Temperature (DART) : 300°C
Measuring mode (MS) : Positive/Negative scanning simultaneously

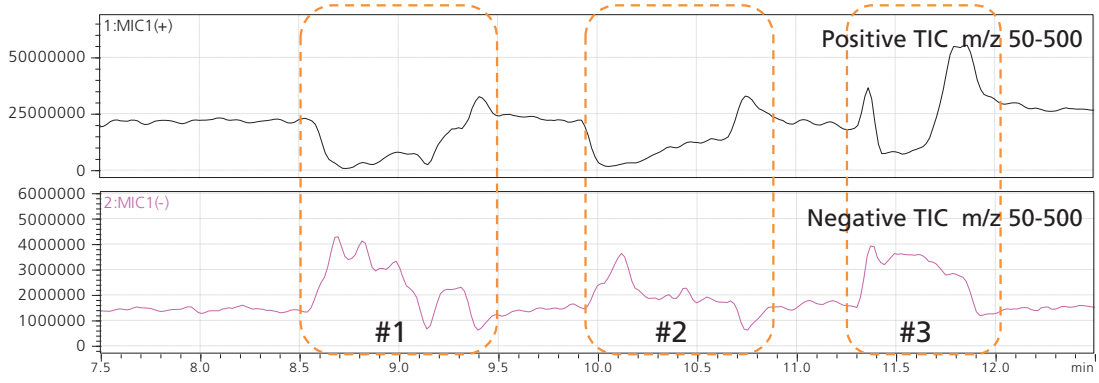

Figure 3 TIC chromatogram of CFRP samples #1, #2, #3

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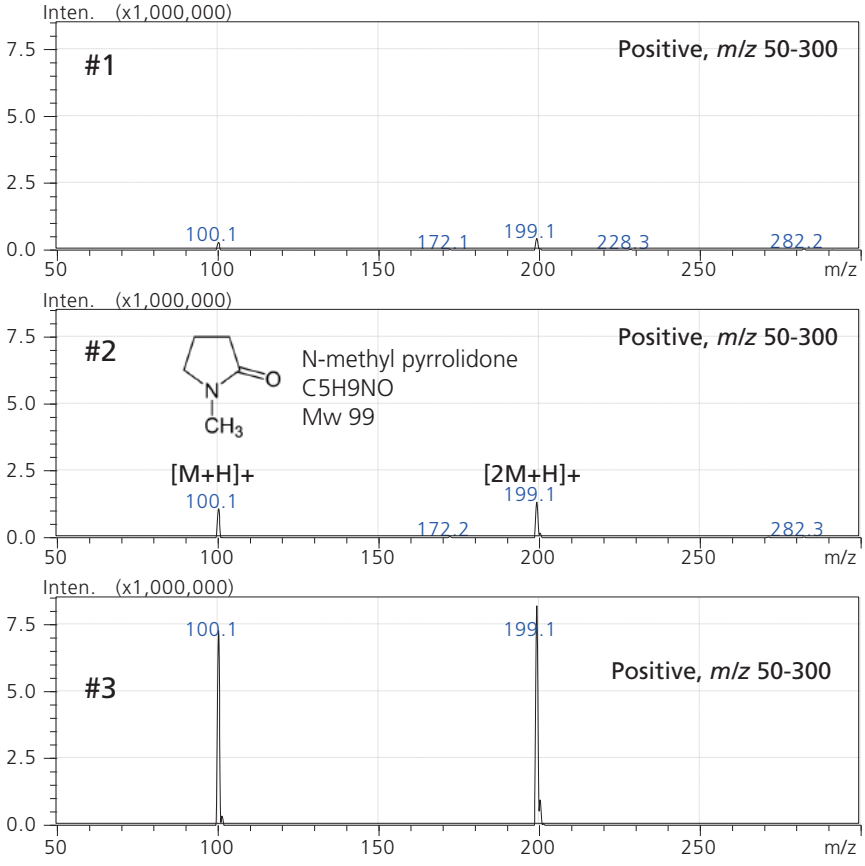

Figure 4 DART-MS spectra of each sample

Since the thermosetting polyimide used for this measurement was molded using the organic solvent (N-methyl pyrrolidone, C5H9NO, molecular weight 99), molecular related ions of N-methyl pyrrolidone, [M+H]+ (m/z 100) and [2M+H]+ (m/z 199), were detected very strongly in the mass spectrum of #1. The mass spectrum of #2 also showed the same ions that intensity was intentionally detected strongly compared with #3 although intensity was weak compared with #1. Even if it raised the heating gas temperature of DART to high temperature (up to 500°C), MS signal considered to originate in the structural information of CFRP was not able to be obtained.

Then, the optional heating mechanism, ionRocket (Bio Chromato, Inc.; Figure 5), in which a sample could be heated directly was developed to the sample stage of DART, and analysis of CFRP was verified by heating the sample directly up to 600°C.

Sample

#4 thermosetting polyimide (molded; dried)
#5 thermoplastic polyimide (control)

Analytical Condition

Heater Temperature (DART)	: 400°C
Temperature control (ionRocket)	: 0-1min room temp., 4min 600°C
Measuring mode (MS)	: Positive scanning

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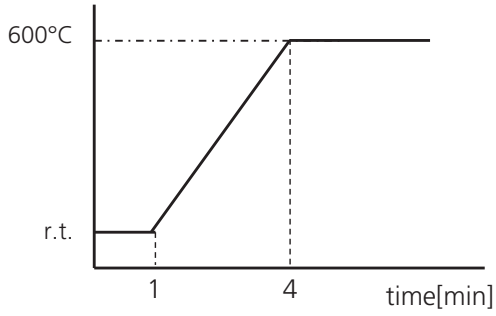

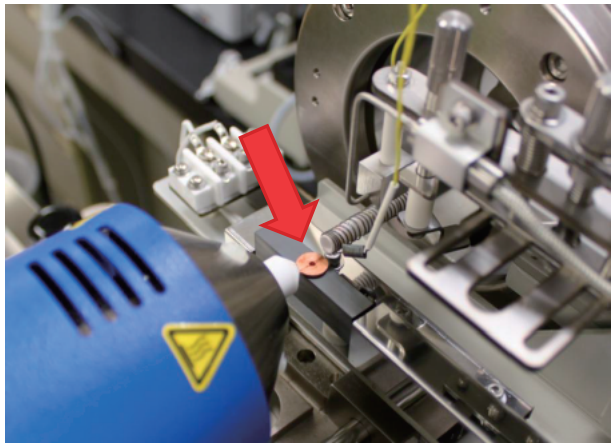

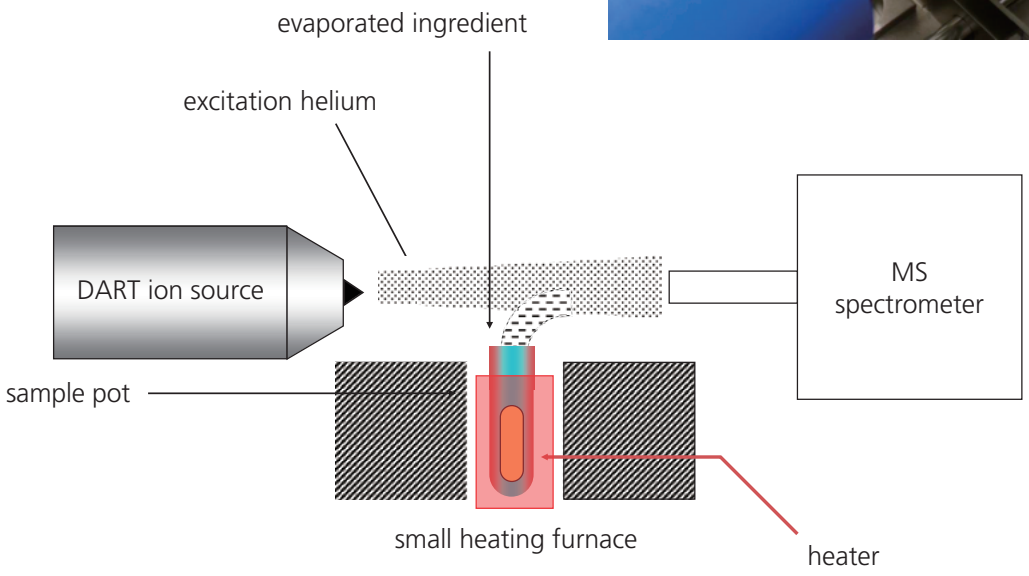

Figure 5 DART-MS system integrated with ionRocket

When heating temperature was set to 600°C, the rudder shape signals of 28u (C2H4) interval was appeared around m/z 900. This signal was more notably detected with the thermosetting polyimide sample than the thermoplastic sample. Since the sample was heated at high temperature, it was considered that the thermal decomposition of resin started, the thermal decomposition ingredient of polyimide clustered, and possibly the structures of the rudder signals of equal interval were generated.

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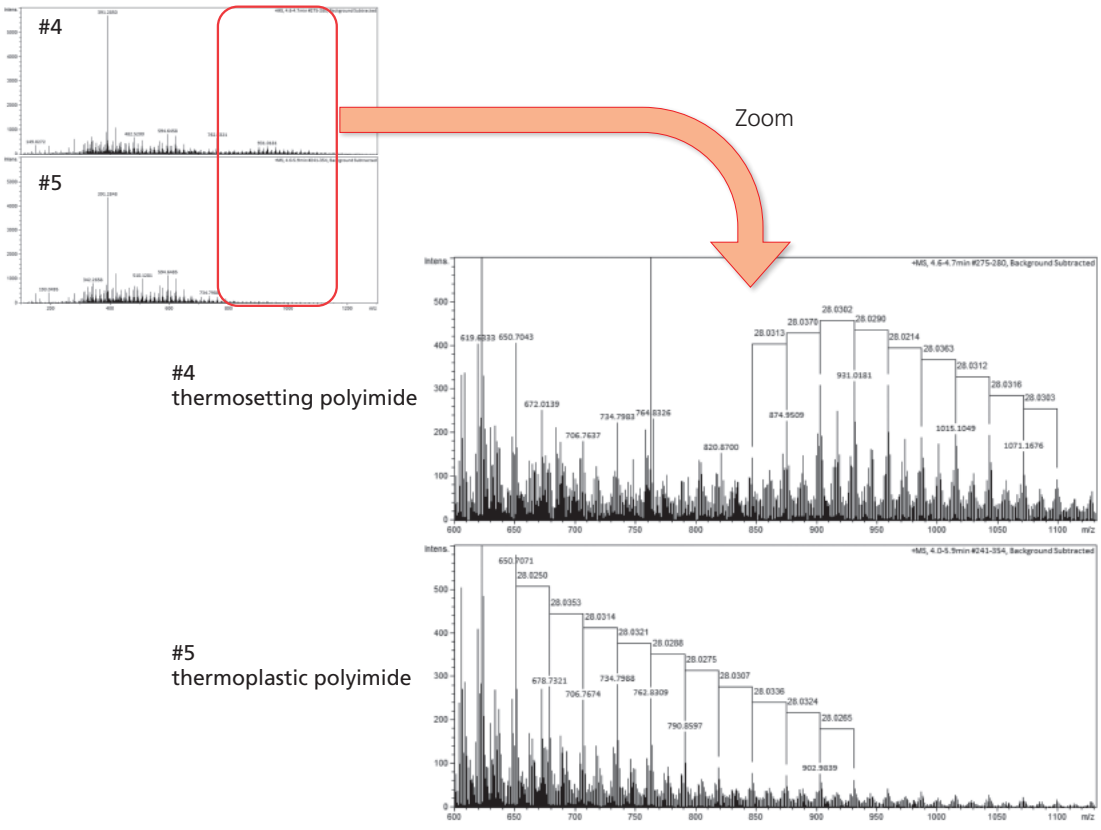

Figure 6 DART-MS with ionRocket spectra of each sample

Conclusions

The result of having analyzed the carbon fiber plastic CFRP (thermosetting polyimide and thermoplastic polyimide) using DART-MS,

a. residue of the solvent used in fabrication was able to be checked by direct analysis of CFRP by DART.
b. analyzing CFRP by DART and the heating option ionRocket, the difference between thermosetting polyimide and thermoplastic polyimide was able to be found out.

Acknowledgment

We are deeply grateful to Mr. Yuichi Ishida, Japan Aerospace Exploration Agency (JAXA), offered the CFRP sample used for this experiment.

First Edition: June, 2014

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