CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

ANALYSIS OF ACRYLATE POLYMERS BY DIRECT PYROLYSIS/FT-IR

Acrylate polymers fall into a class of polymers which depolymerize when heated to extreme temperatures. This aspect of acrylate polymers makes them especially ammenable to analysis by pyrolysis/FT-IR.

Figure 1 shows a sketch of the pyrolysis/FT-IR interface used in this work. The interface is a cylindrical cell, with ZnSe windows on each end. The windows are held in place by knurled nuts which can be easily removed for cleaning the windows. A port in the center of the cell allows the Pyroprobe to be inserted so that the filament is directly below the infrared beam. When the sample is pyrolyzed, the gaseous pyrolysate diffuses immediately into the beam. Additional ports are located on the cell for sweep gas inlet and exit.

The direct pyrolysis/FT-IR spectrum of poly(methylmethacrylate), (PMMA), is shown in Figure 2. Pyrolysis of the sample at 650° C for 30 seconds produced a spectrum which is identical to that of the methyl methacrylate monomer. Some bands are shifted slightly because of the elevated temperature at which the spectrum was obtained. When acrylate polymers are pyrolyzed, they tend to unzip or depolymerize, forming almost exclusively monomer units. This accounts for the IR spectrum of PMMA resembling the methylmethacrylate monomer. One of the most evident bands in the spectrum is the C-O stretch at 1680 cm^{-1}.

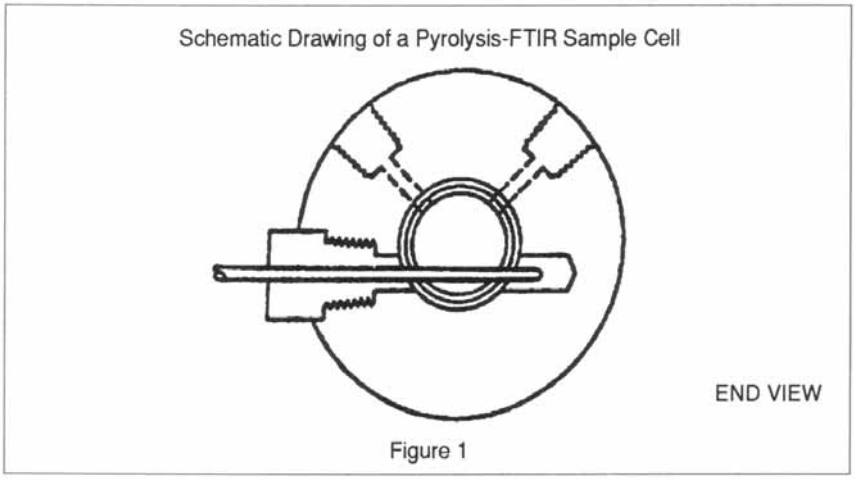

Figure 1

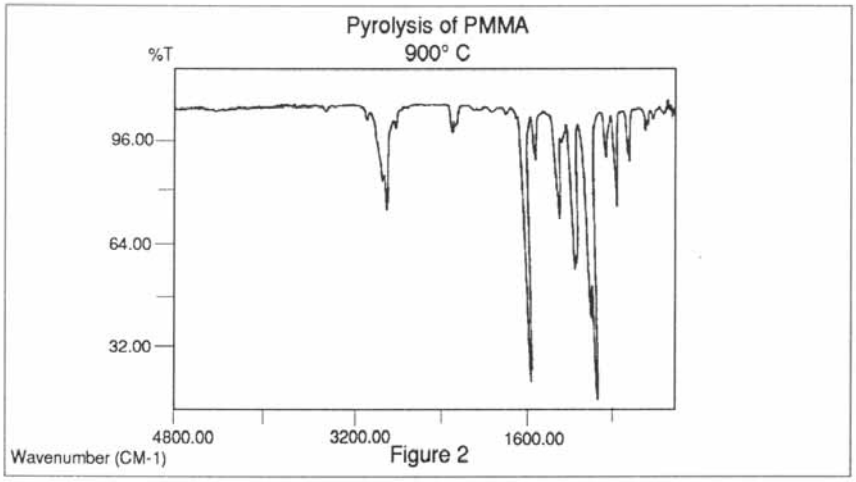

Figure 2

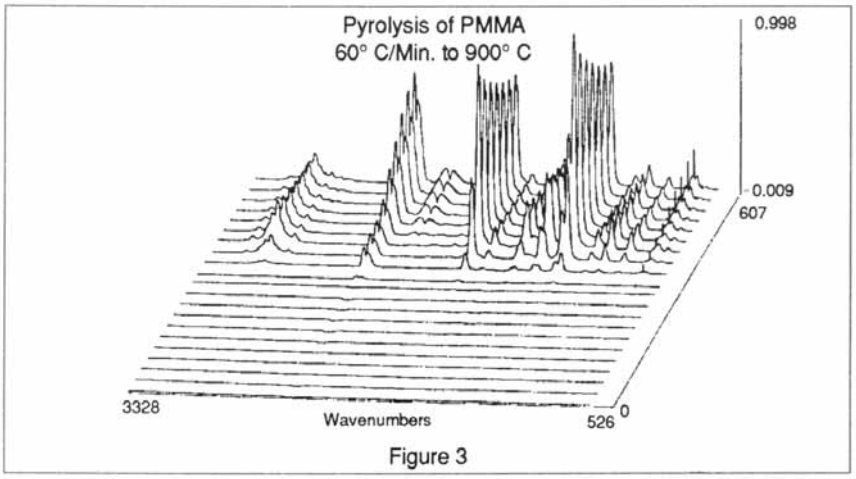

Figure 3

Bands in the fingerprint region are helpful in identifying various compounds. Much like pyrolysis/GC provides a fingerprint for qualitative identification of polymers; direct pyrolysis/FT-IR can be used to give a quick determination of polymer identity.

Pulse pyrolysis is not the only way to obtain direct pyrolysis/FT-IR spectra. Samples can be heated at slower rates in order to monitor the appearance of various species as they are evolved from the sample. Figure 3 shows a time-evolved study of PMMA which was heated at 60° C/minute to a final temperature of 900° C. By looking at the appearance of the acrylate bands early in the temperature program, it is verified that the initial degradation step is depolymerization.

This technique has many applications in the polymer industry, but may also be seen as a possible solution for evaluating complex composite materials.

EQUIPMENT PYROLYSIS

Pyroprobe model 1000 filament pyrolyzer, with temperatures continuously variable to 1400° C.

INTERFACE

Brill Cell for Direct Pyrolysis/FT-IR, containing ZnSe windows and sweep gas inlet ports.

DATA ACQUISITION

IBM PS2-70 with a Hewlett-Packard plotter.

For more information on this and related applications, we recommend the following readings:

J. T. Cronin and T. B. Brill, "Thermal Decomposition of Energetic Materials 26. Simultaneous Temperature Measurements of the Condensed Phase and Rapid-Scan FT-IR Spectroscopy of the Gas Phase at High Heating Rates." Appl. Spectr. 41, 1147, (1987).

J. W. Washall and T. P. Wampler, "Analytical Pyrolysis of Complex Multicomponent Samples." J. Chromatogr. Sci. 27, 144, (1989).

T. P. Wampler, "Thermometric Behavior of Polyolefins." J.A.A.P., 15, 187, (1989).

Additional literature may be obtained by contacting your CDS Instruments Representative, or by writing to the CDS Applications Lab.

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