

UTILIZING FOURIER TRANSFORM INFRARED SPECTROSCOPY FOR PLASTICS FAILURE ANALYSIS

This article explores one of the most commonly used analytical techniques in plastic component failure analysis—Fourier transform infrared spectroscopy—along with a case study to demonstrate its use.

Jeffrey A. Jansen

The Madison Group, Madison, Wisconsin

Failure within plastic components can occur in several different ways, including aesthetic alteration, deformation/distortion, degradation, wear, and fracture. In the case of failure involving fracture, a number of common plastic failure mechanisms exist such as ductile overload, impact, creep rupture, environmental stress cracking, and fatigue. Determination of the fracture failure mode involves identifying the crack initiation mechanism and the propagation mode. This is usually ascertained using visual-based techniques such as stereomicroscopy, scanning electron microscopy, and the preparation of mounted cross sections.

Assessing the mode of the failure is often not as difficult as establishing why the part failed. Evaluating why the part failed usually requires analytical testing beyond the visual-based techniques. In many cases, a single cause cannot be identified because multiple factors may have contributed to the failure. All the factors that affect the performance of a plastic component can be classified into four categories: material, design, processing/manufacturing, and service conditions/environment^[1]. These factors do not act independently on the component but

instead act in concert to determine its performance properties.

The main differences between how failure analyses are performed on metal and plastic materials are based primarily on the techniques used to evaluate the material's composition and structure. Unlike metals, polymers have a molecular structure that includes characteristics such as polymer functional groups, molecular weight, molecular weight distribution, crystallinity, tacticity, molecular orientation, and fusion. These characteristics have a significant impact on the properties of the molded article. In addition, plastic resins are formulated with additives such as reinforcing fillers, plasticizers, colorants, antidegradants, and process aids. It is this combination of molecular structure and complex formulation that requires specialized testing^[2]. This article explores one of the most commonly used analytical techniques in plastic component failure analysis—Fourier transform infrared spectroscopy—along with a case study to demonstrate its use.

FTIR SPECTROSCOPY

Fourier transform infrared spectroscopy (FTIR) is a nondestructive micro-

analytical spectroscopic technique that involves the study of molecular vibrations^[2]. The analysis results provide principally qualitative, but also some quantitative, information regarding the composition and state of the evaluated material. FTIR uses infrared energy to produce vibrations within the molecular bonds that constitute the material being evaluated. Vibrational states of varying energy levels exist within molecules. Transition from one vibrational state to another is related to absorption or emission of electromagnetic radiation^[3]. These vibrations occur at characteristic frequencies, which correspond to functional groups within the molecular structure of the sample. FTIR produces a unique spectrum, which is comparable to the fingerprint of the material. It is the principal analytical technique used to qualitatively identify polymeric materials—and is commonly the first analytical technique used in a plastics failure analysis.

Several different sampling techniques, all involving either transmission or reflection of the infrared energy, can be used to analyze the sample material. This allows evaluation of materials in all forms, including hard solids, powders, liquids, and gases. In the analysis of

polymeric materials, the most common sampling techniques are transmittance, reflectance, and attenuated total reflectance. Additionally, a microscope can be interfaced with the spectrometer to focus the infrared beam and allow the analysis of samples down to 10 μm . Regardless of the sampling technique, the beam of infrared energy is passed through or reflected off the sample and directed to a detector. The obtained spectrum shows frequencies that the material has absorbed and frequencies that have been transmitted.

The results generated through FTIR analysis are referred to as an infrared spectrum. The spectrum graphically illustrates the relative intensity of the energy absorbed on the y-axis versus the frequency of the energy on the x-axis. The frequency of the energy can be represented directly in microns (μm) or as reciprocal centimeters (cm^{-1}) referred to as wave numbers. The spectrum can be interpreted manually or, more commonly, compared to voluminous library references with the aid of a computer. The discrete spectral features present in an FTIR spectrum are known as absorption bands, which correlate to functional groups within the molecular structure of the sample.

FTIR IN FAILURE ANALYSIS

Material identification: Possibly the most important use of FTIR in failure analysis is the identification of the base polymer that comprises the sample. Determining the composition of the failed component is an essential part of the investigation. Because different polymers have a wide variation in their physical, mechanical, chemical resistance, and aging properties, the use of the wrong resin can yield detrimental results in many applications. Fourier transform infrared spectroscopy is well suited for the identification of polymers that have different molecular structures (Fig. 1).

Confirming that the failed article was produced from the specified material is a primary consideration of the failure analyst in assessing the cause of the failure. The use of FTIR in characterizing the composition of the plastic resin base polymer is illustrated

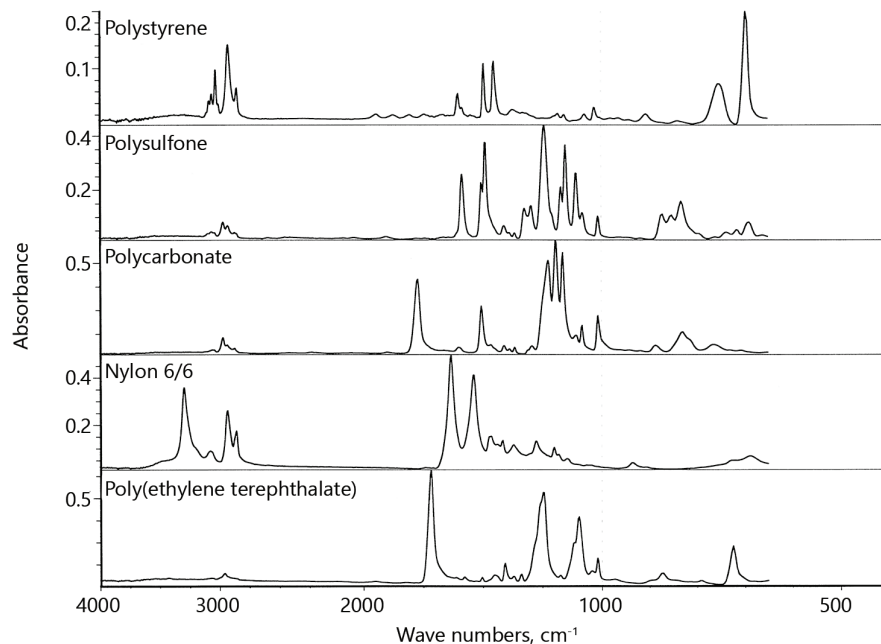


Fig. 1 – FTIR spectral comparison showing distinct differences between the results obtained on various plastic materials.

in the case study included in this article. However, one area in which FTIR is inadequate is differentiating polymers that have similar molecular structures, such as the members of the nylon family, between poly(ethylene terephthalate) and poly(butylene terephthalate), and between polyacetal homopolymers and copolymers.

Aside from determination of the base polymer, FTIR can be helpful in characterizing other formulation constituents. FTIR analysis can provide information regarding the presence of additives and filler materials. Due to the nonlinearity of infrared absorptivity of different molecular bonds, it is not possible to accurately state minimum concentration detection limits. However, it is generally considered that materials present within a compounded plastic resin at concentrations below 1% may be below the detection limits of the spectrometer. Given that FTIR is principally used for the analysis of organic materials, its use in the evaluation of inorganic filler materials is somewhat limited. However, some commonly used fillers—such as calcium carbonate, barium sulfate, and talc—produce unique and identifiable absorption spectra.

Degradation: FTIR is a valuable

tool in assessing a failed component material for degradation such as oxidation and hydrolysis. Molecular degradation, often involving molecular weight reduction, has a significant detrimental impact on the mechanical and physical properties of a plastic material. This degradation can result from several stages in the material's life, including resin compounding, molding, and service. As a polymeric material is degraded on a molecular level, the bonds comprising the material are altered. Fourier transform infrared spectroscopy detects these changes in the molecular structure.

While FTIR cannot readily quantify the level of degradation, it is useful in assessing whether the material has been degraded and in determining the mechanism of degradation. Specifically, several spectral bands and their corresponding molecular structures can be ascertained. These include: the carbonyl band, particularly carboxylic acid, with formation representing oxidation; vinylidene group formation as an indication of thermal oxidation; the abstraction of bands associated with unsaturation resulting from thermal degradation; vinylene functionality for photooxidation; and hydroxyl group formation indicating hydrolysis^[4].

The following case study illustrates the effectiveness of FTIR in assessing molecular degradation.

CASE STUDY: BRITTLE PERFORMANCE OF POLYSTYRENE HOUSINGS

Housings from two different production lots exhibited significant variation in performance during quality testing. Specifically, the difference was

detected during impact testing performed on the molded parts. One of the lots exhibited superior impact properties and ductile behavior, similar to that of a previous production run. The other lot demonstrated a marked reduction in impact properties, as evidenced by lower impact strength along with relatively brittle behavior (Figs. 2 and 3). The housings were specified to be produced by injection

molding using a high-impact polystyrene (HIPS) resin.

Testing and results: Visual examination of the impacted housing specimens confirmed the reported property variation. Specimens representing the retained reference parts from an earlier production lot showed significant ductility in the form of stress whitening and deformation. Conversely, the new production lot samples exhibited characteristics of brittle fracture. The two lot samples were analyzed using FTIR in the attenuated total reflectance mode. A comparison of the results showed a general match. Both sets of spectra exhibited absorption bands characteristic of styrene and butadiene functionalities (Fig. 2). However, variation in the relative intensities of the styrene and butadiene bands was observed (Fig. 3). Band intensity measurements were taken for the butadiene and styrene functionalities at 964 and 751 cm^{-1} , respectively, for the two lot samples. The results, as shown in Table 1, clearly illustrate that the samples that performed in a brittle manner had a relatively lower level of butadiene content.

Conclusions: At the conclusion of this testing, the variation in housing performance was attributed to the use of two different resins to mold the parts. Although both production lots were molded from HIPS, the relative level of butadiene functionality was significantly lower in the brittle parts. Butadiene is added as an impact modifier. As such, it enhances the ductility of the resin as increasing amounts are added to the resin mix. ~AM&P

For more information: Jeffrey A. Jansen, engineering manager, The Madison Group, 2615 Research Park Dr., Madison, WI 53711, 608.231.1907, info@madisongroup.com.

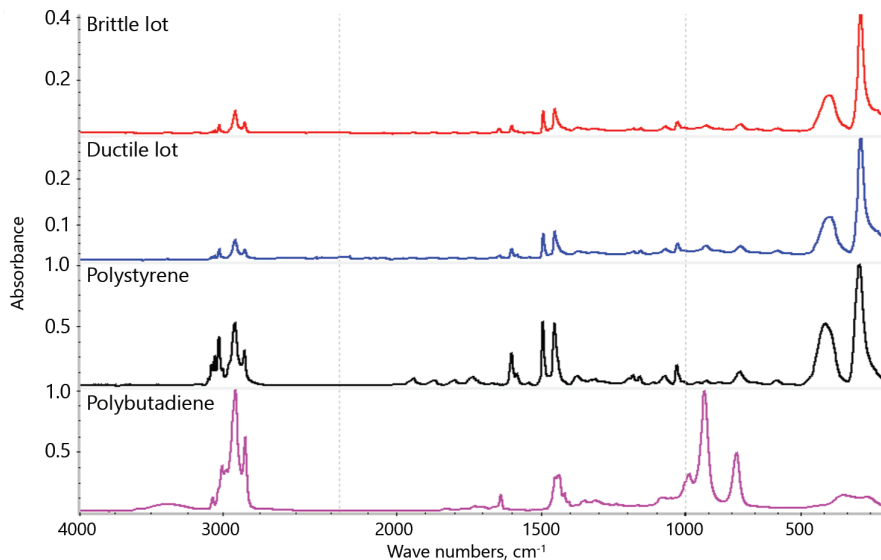


Fig. 2 – FTIR spectral comparison showing generally good agreement between brittle lot and ductile lot materials. Both spectra contained absorbances that are associated with styrene and butadiene functionalities.

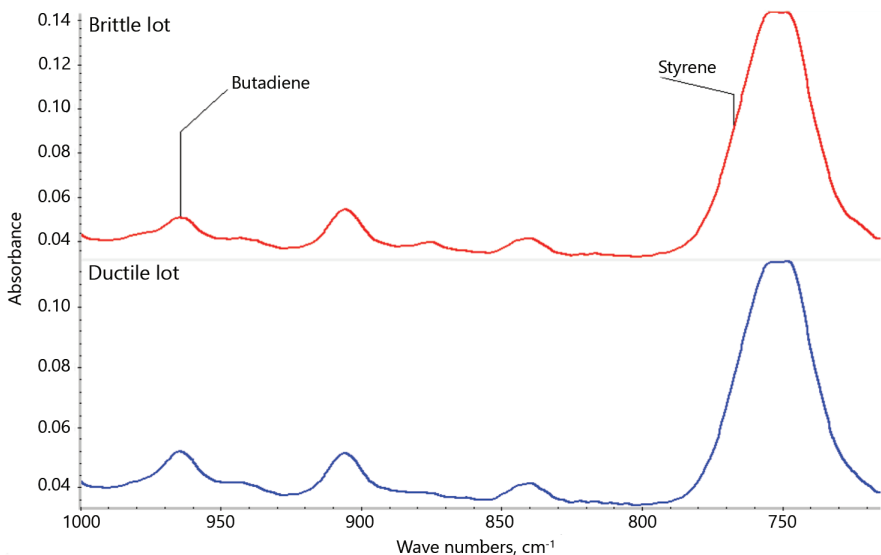


Fig. 3 – FTIR spectral comparison illustrating a relatively lower level of absorption associated with butadiene functionality in the brittle lot material compared with the ductile lot material.

TABLE 1 – SPECTRAL BAND INTENSITIES

Sample	Butadiene 964 cm ⁻¹	Styrene 751 cm ⁻¹	Butadiene/styrene ratio
Ductile lot			
1	0.0130	0.0780	0.167
2	0.0110	0.0660	0.167
3	0.0099	0.0570	0.174
Brittle lot			
1	0.0068	0.0685	0.099
2	0.0130	0.1320	0.098
3	0.0110	0.1040	0.106

Spectral band intensities for a ductile sample and a brittle sample, illustrating variation in the ratio in content between butadiene and styrene.

References

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