Overview

In the spring of 1985, when this symposium was first planned, the use of a microscope as an accessory in a Fourier transform infrared (FT-IR) spectrometer was still somewhat a new concept. The symposium was planned as a means of introducing many people to this new technique. The day long symposium, which was part of the 1985 Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) meeting, consisted of papers which covered design considerations, sample handling, and applications to such areas as fibers and polymers.

This volume consists of nine peer-reviewed papers that were presented at that symposium. As such they serve as an introduction to the field of infrared microscopy. The technique is growing very rapidly. ASTM E13.03 on Infrared Spectroscopy has a task group that is working on standards for IR microanalysis that will include sections on FT-IR microscopes. During the fall of 1986 there were two symposia organized on the topic; one at the 1986 FACSS meeting and one at the 1986 Eastern Analytical Symposium (EAS) meeting. The proceedings from both of these symposia are also being written. With the introduction of this STP and the proceedings from the two 1986 symposia, there will be valuable references for a spectroscopist who is getting involved in FT-IR microscope analysis.

The title of this Special Technical Publication, The Design, Sample Handling, and Applications of Infrared Microscopes, was chosen to be descriptive of the material found in this volume. Some of the design criteria for infrared microscopes are discussed by Messerschmidt. Both a custom-built microscope (Katon et al.) and a modification of a commercial instrument (Miseo and Guilmette) are discussed. All of the authors deal with sample handling to some extent in their papers. In particular, however, the papers by Humecki and Shearer et al. describe sample handling and the tools that are needed in great detail. These two papers would be the perfect place for the spectroscopist who is just getting an FT-IR microscope to start. They would provide a shopping list of other necessary tools his FT-IR microscopy laboratory would need. The entire purpose for the FT-IR microscope is to make it possible to analyze very small specimens or very small contaminants in specimens. Because of this, the entire volume deals with applications. There are several papers dealing with fiber analysis (Bartick, Chase, and Katon et al.) and two dealing with polymer analysis (Harthcock and Mirabella).

In "Photometric Considerations in the Design and Use of Infrared Microscope Accessories" Messerschmidt discusses some of the problems associated with FT-IR microscopy and how to prevent them. The major limitation for successful application of FT-IR microscopy is related to the diffraction theorem. This theorem is explained in detail as it applies to the infrared region and to the use of the FT-IR microscopes. By understanding the diffraction theorem, the spectroscopist is able to deal better with the limitations it imposes on the analysis. Two techniques are presented to deal with the problems caused by diffraction. These are ultramicroscopy and redundant aperturing. Both of these techniques were very new to FT-IR microscopy when this symposium was presented in the fall of 1985. Since that time a commercially available FT-IR microscope, the IR-Plan from Spectra-Tech, has been introduced which allows for redundant aperturing.

"FT-IR Microspectrophotometry as a Failure Analysis Tool" by Shearer and Peters mainly describes sample handling and the tools needed for FT-IR microscopy. This chapter is an excellent one for the analyst new to FT-IR microscopy. It describes some of the basic features of FT-IR microscopes as well as the basic sampling tools that the FT-IR microscopist would need.

The chapter by Humecki presents the spectroscopist with sampling information that optical microscopists have been using for many years. He also describes how to make what he calls a capillary brush. The capillary brush allows you to pyrolyze a specimen that is less than one microgram in size. A particle is pushed into the capillary brush and the end sealed in a microflame. The specimen is pyrolyzed by heating the capillary brush. The end of the brush is then broken off and a tiny amount of solvent is added. The solvent and specimen are dripped onto a salt plate, the solvent evaporated, and the pyrolyzate can be analyzed. This is just one example of the many useful techniques that are described. The IR spectroscopist who is dealing with FT-IR microscopes can learn a great deal about handling small samples from the optical microscopist.

Chase discusses some of the limitations of FT-IR microscopy and how the problems can be overcome. He describes some of the problems that Messerschmidt discussed in an earlier chapter. Once again the need for physical masking at the specimen, rather than a simple aperture at an image plane, is stressed. This appears to be essential for any quantitative work with the microscope accessory. One of the advantages of the FT-IR microscope that is pointed up in the paper is the ability to look at single fibers. By first characterizing the polarization characteristics of the FT-IR and the microscope, it is possible to measure a dichroic spectrum on a single fiber.

In "Considerations for Fiber Sampling With Infrared Microspectroscopy" Bartick deals with the specimen handling issues for fibers. Three different size categories are described, and the way each different diameter fiber is best prepared and handled is discussed. Techniques for flattening fibers to reduce the thickness of the fiber and therefore reduce the tendency for it to act as a lens are described. An important point about spectral subtractions is that all of the parameters must be the same. For FT-IR microspectroscopic analysis this means that the same specimen technique and aperture size must be used in order to obtain valid results.

Katon et al. also describe techniques for fiber analysis. The problem of the beam being defocused due to the cylindrical shape of the fiber is discussed. Several ways to prevent this include flattening the fiber prior to analysis or measuring the fiber in a diamond anvil cell. At the time of the symposium not much work was being done with diamond anvil cells in FT-IR microscopes. Most likely this was due to the fact that the diamond anvil cell did not fit into many of the commercial microscopes. Katon et al. describe a custom-built microscope and the results they obtained with the diamond anvil cell. Since the symposium more commercial FT-IR microscopes can accommodate diamond anvil cells, and this will certainly be a field of further growth.

The paper by Miseo and Guilmette describes using the FT-IR microscope for several very difficult kinds of industrial problems. One of these dealt with identifying an additive in ink. They cast a spot of ink onto a silver chloride plate and in effect performed a chromatographic separation of the ink using methylene chloride. After separating the original spot into several smaller spots, those spots were further separated with methylene chloride. The various spots on the silver chloride plate were analyzed using the FT-IR microscope. This paper may well describe the first use of the FT-IR microscope for TLC type analysis. Since this symposium was held, the use of FT-IR microscopes for analyzing chromatographic fractions has become very popular and will most likely continue to be.

Harthcock describes some of the advantages of FT-IR microscopy over traditional infrared microsampling techniques. The applications in this chapter and the next chapter by Mirabella deal with polymer analysis. The use of FT-IR microscopes in the analysis of contaminants in polymers is a growing technique. One of the applications that Harthcock describes is the study of multilayer polymer films. A cross section of the polymer laminate is first obtained by microtoming it to the appropriate thickness. The various layers of the laminate can be then analyzed using FT-IR microscopy. By masking the specimen, layers as thin as 10 to 15 μm can be measured. One problem is that spectra cannot be readily obtained from layers that are thinner than 10 μm by using the FT-IR microscope. However, attenuated total reflectance (ATR) can be used as a complementary technique to study these specimens. By using both techniques Harthcock was able to analyze the laminate more completely.

Mirabella's paper describes two applications using the FT-IR microscope to analyze polymers. One of these involves the analysis of a multilayer film by using a combination of FT-IR microscopy and ATR. Being able to identify successfully the individual layers of a multilayer specimen is of critical importance to the polymer industry. This application may well become one of the most popular industrial uses of the FT-IR microscope. The other application that Mirabella describes is a novel technique involving the simultaneous measurement of thermal property response and infrared spectra using a differential scanning calorimeter (DSC) and an FT-IR microscope. The DSC microscopy cell was used with sodium chloride windows, rather than the standard sapphire windows used for optical microscopy. The DSC microscopy cell was then placed in the FT-IR microscope. This allowed for simultaneous measurement of FT-IR spectra before, during, and after the melting of the polymer specimen. Using this combined technique it may be possible to correlate the thermally induced changes in the infrared spectra with the effects on particular chemical bonds.

In my overview of this STP I have tried to cover briefly some of the highlights of the individual papers. One paper that is sadly absent from the volume is the one presented by Tomas Hirschfeld at the symposium in 1985. His untimely death in April 1986 occurred before his paper was completed. We will all certainly miss Tomas, his friendship, and his enormous contributions to so many areas of spectroscopy. Because of this, I would like to dedicate this STP to the memory of Tomas B. Hirschfeld.

Patricia B. Roush

Perkin-Elmer Corporation, Norwalk, CT 06859-0903; symposium chairman and editor.