Application Note

FT-IR: JI-Ap-FT0507-002

Applications for the IMV-4000 Multi-channel Infrared Microscope

Over the last two decades, IR microscopy has been widely utilized to identify increasingly smaller samples. Incorporation of a linear array detector and rapid scanning in a FT-IR microscope permits high-speed FT-IR spectroscopic imaging, with applications in semiconductors, polymers, the pharmaceutical industry, biomaterials research and other areas. JASCO's IMV-4000 offers the highest performance in the industry in terms of measurement speed and signal-to-noise ratio. This report describes the system configuration and several applications of the IMV-4000

1. Introduction

Over twenty years have passed since infrared micro-spectrometers (micro-FTIR) were first used in microscopic characterization. As well as measuring the spectra of microscopic sites, such as impurity analysis in manufactured products, early micro-FTIR was used for much the same purposes as normal FTIR. Recently, however, it has increasingly been used for analyzing the two-dimensional distribution of samples such as natural products and polymeric materials, resulting in what is termed infrared imaging. Infrared imaging is recognized as a technique that measures the infrared spectrum at each in-plane point and then uses peak heights, peak areas and other criteria to visualize the molecular structure distribution. However, with conventional systems, it takes an extremely long time to complete the image since the method involves moving the sample on the stage with each measurement to obtain data for discrete points (Figure 1). While the utility of infrared imaging is well known, this problem is probably the reason why the technique has not become more widespread.

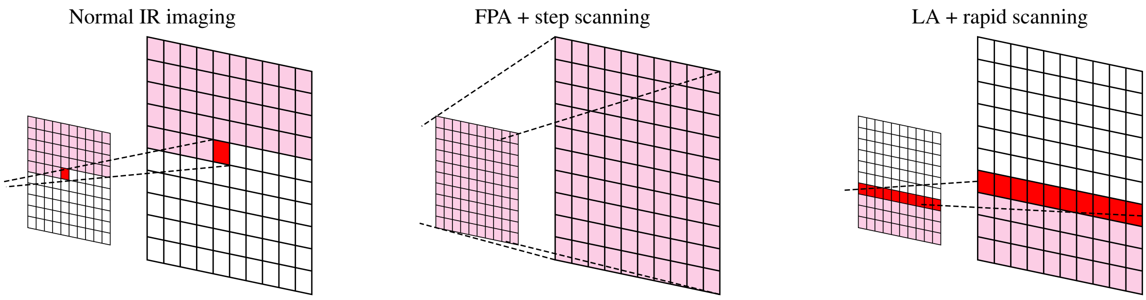
In the mid 1990's, an infrared microscope was proposed that would perform infrared imaging in a shorter time. The proposed system featured a focal plane array (FPA) detector capable of nearly instantaneous wide area measurements. However, these detectors with many detector elements arranged on the focal plane (for example, 16 x 16 elements) are extremely expensive (Figure 1). Furthermore, high-speed data processing systems that could perform Fourier transforms on large arrays on interferograms are complicated, so the systems had to be combined with step scanning instruments. Because of these two cost factors, systems using FPA detectors are extremely expensive and thus have never become widely used.

JASCO has developed a multi-channel infrared microscope (IMV-4000) that uses an inexpensive linear array detector, with fewer elements than an FPA detector. The IMV-4000 is an infrared microscope that incorporates a 16 x 1 element linear array MCT detector. By combining the system with a fast-scan FTIR, a high-precision auto-stage, a linear array MCT detector and a high-speed parallel data processing circuit, the IMV-4000 imaging system is capable of imaging up to 9,600 points per minute (Figure 1). The system demonstrates that it is possible to perform infrared imaging faster by a figure of two or more than imaging using a conventional single element detector. It has also made it possible to provide a fast infrared imaging system at less than half the price of systems featuring FPA detectors. In the following section, we discuss various measurement examples using the IMV-4000.

2. Measurement Window

Figure 2 shows the computer software window during measurement. Since the IMV-4000 transfers data using a high-speed parallel data processor, it can display spectra and peak intensities in real time. The top portion of the window displays the infrared absorption spectra from the individual detector elements and the bottom left portion shows a visible image of the measurement area captured by the CCD camera. The bottom right portion displays a color-coded infrared image of the peak intensity selected prior to the measurement. The sample displayed is a trademark seal for the circuit board of an electronic device. The measurement was done in reflection mode on a 6.2 x 1.75 mm area. Since the spatial resolution was 12.5 x 12.5 µm, 490 x 140 (60,440) points were measured. Spatial resolution is determined by the magnification of the cassegrain objective.

Figure 1. Principles of Multi-Channel Imaging



Each point is measured and the stage is then moved.

Measurements are performed simultaneously using a two-dimensional detector (FPA). Normally, step-scanning measurement is performed due to restrictions on data processing rates.

Line analysis (LA) is performed using detector elements arranged in a row and the stage or mirror is moved to perform imaging.

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When the 16X objective of the IMV-4000 is used, a spatial resolution of 12.5 x 12.5 µm results while the 32X objective yields a spatial resolution of 6.25 x 6.25 µm. The measurement only required about 7 minutes. Since conventional single point imaging requires at least 5 seconds per point, conventional techniques would have required about 80 hours (including the time for moving the stage) for the same measurement. The IMV-4000 can therefore complete measurements in 1/700 the time of conventional instruments. The color coded image in the bottom-right portion of the window displays the peak intensity of the overtone of the C=O stretch near 3500 cm^{-1}. Since all data points are being measured as spectra, it is possible to reconstruct a color-coded image at any wave number using the data processing functions after measurement.

3. Analysis Example Demonstrating the Advantages of Fast Imaging

In this section, an example of a fast imaging experiment is illustrated. To maximize the utility of the imaging system, an imaging analysis program was developed in conjunction with the IMV-4000 imaging system. The imaging analysis program features a variety of computational and data processing functions for infrared spectra, based on the molecular absorption to be visualized, including color-coded images and "birds-eye" views. The measurement examples discussed below were analyzed using this program. We will also illustrate a program that images molecular structure using a multivariate analysis technique and a tool that was developed for preprocessing samples.

This section discusses the results of measuring patterns on a silicon wafer in order to evaluate whether the visible image and the infrared image of the IMV-4000 match and to demonstrate the speed of the new instrument compared to the conventional technique. Figures 3 and 4 illustrate the measurement examples for patterns and impurities on silicon wafers.

The measurement size of one point was 12.5 x 12.5 µm, since the measured area was 600 x 600 µm and a 16X cassegrain objective was used. Consequently, 48 x 48 (2,304) points were measured. A resolution of 16 cm^{-1} and 16 coadded scans were used to collect the data. Since visible light cannot penetrate the silicon wafer but infrared light can, the visible light image was captured in reflection mode and infrared imaging was performed in transmission mode.

Using the SiO peak near 1100 cm^{-1} as a probe, a color-coded image based on the peak intensity was created (Figure 3). The visible image and infrared images closely match. In addition, an impurity appears when a color-coded image is created using the peak near 2900 cm^{-1}, attributed to the stretching vibration of -CH. After extracting and analyzing the spectrum of the impurity area, it was determined that the contaminant was a type of fatty acid. With conventional infrared imaging using a single element, it would have taken several hours to complete the measurement but we obtained data for 2,304 points in approximately four minutes.
Based on the above result, we confirmed that fast infrared imaging is possible. The infrared images and visible images correspond and the analysis of wafer patterns and impurities was facilitated. We measured impurities that could be verified using visible observation but there are also cases where transparent impurities affect product quality. In such cases, it is possible to identify the site of the impurity from an infrared image. When there is an impurity comprising multiple components, conventional techniques measure the heterogenous area and yield a spectrum in which the various components overlap, which may interfere with interpretation. Infrared imaging enables easy identification of contaminants by spatially resolving them and measuring the contaminant directly. This technique is expected to be a powerful tool in the analysis of a wide variety of samples.

Figure 2. Measurement Window of the IMV-4000

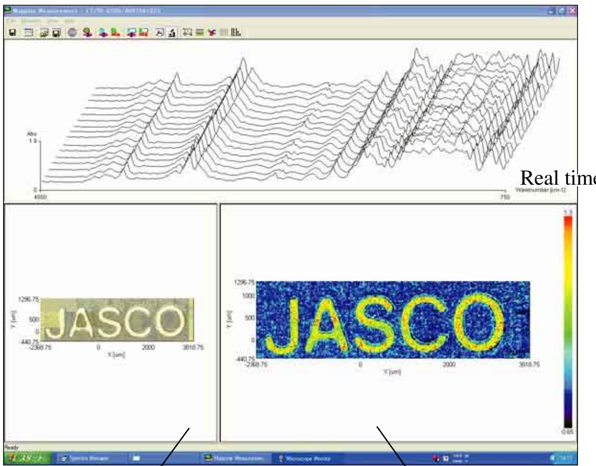

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Figure 3. Pattern Analysis of a Silicon Wafer

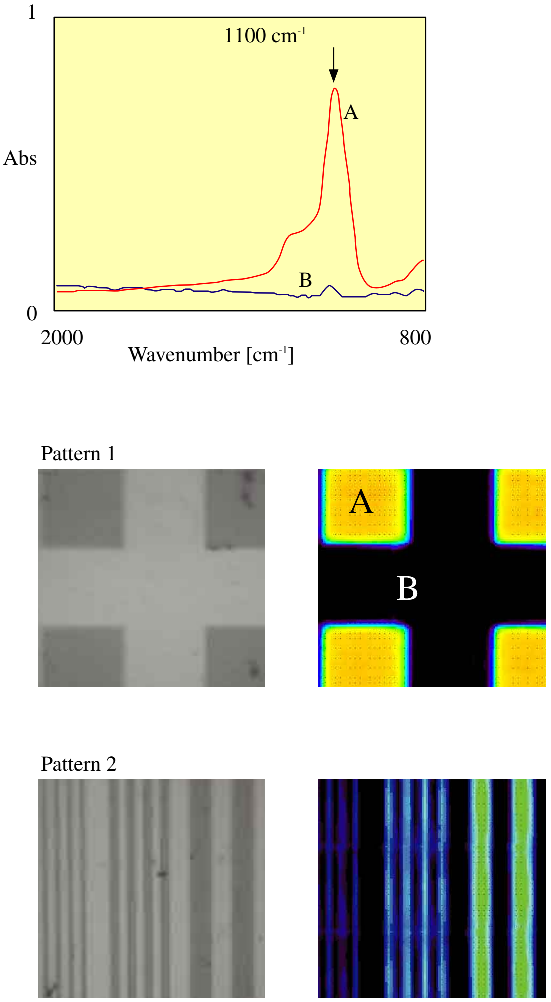
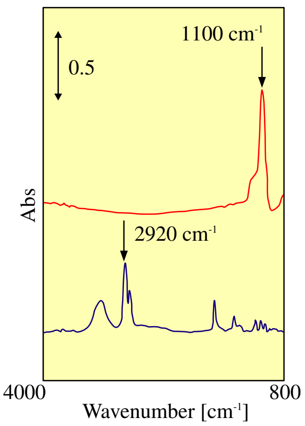
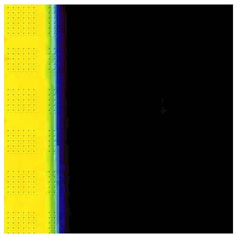


Figure 4. Impurity on a silicon wafer

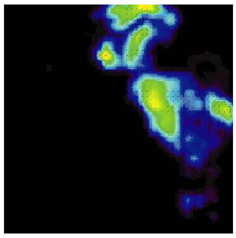


Measurement area	: 600 x 600 µm
Number of measurement points	: 48 x 48
Spatial resolution	: 12.5 x 12.5 µm
Resolution	: 16 cm^{-1}
Accumulation	: 16
Collection time	: Approx. 4 minutes

Sample image (600 x 600 µm)



Peak intensity at 1100 cm^{-1}



Peak intensity at 2920 cm^{-1}

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