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Submicron simultaneous IR+Raman spectroscopy for failure analysis of high technology components

We demonstrate a submicron simultaneous far-field IR+Raman microscope (the mIRage+R, Infrared and Raman microscope), based on Optical-Photothermal Infrared (O-PTIR) spectroscopy for failure analysis (FA) which has been applied to high technology components such as hard discs and displays. O-PTIR brings several advantages to IR microscopy, such as non-contact, sample preparation free, submicron analysis. The collected reflection mode spectra are of high quality and are library searchable against standard transmission or ATR mode spectral libraries. Furthermore, O-PTIR can be expanded to include simultaneous Raman microscopy for a more confident contamination identification. Raman can be used for traditional testing like strain/stress, dopant concentration, DLC and others. Contamination from submicron to microns was conclusively identified by IR+Raman microscopy using our transmission quality data while spectral searching of commercial databases. In many cases the sample thickness was <100nm which would be impossible with most FTIR measurements. This new method of FA testing is a game changer for identifying organic defects and contamination below the detection limits of traditional FTIR systems.

The mIRage microscope accomplishes what no other conventional vibrational spectroscopic technique can.

O-PTIR spectroscopy offers significant advantages:

1. **Submicron IR spatial resolution, 30x better than traditional FTIR/QCL microscopy**
2. **Non-contact, non-destructive, reflection (far-field) mode measurements with little to no sample preparation**
3. **High quality spectra (particle shape/size and surface roughness independent) with no dispersive/scatter artefacts that are directly searchable in commercial/custom FTIR transmission libraries**

Introduction

Confident contamination identification and control is critical to the ongoing successful development of the manufacturing processes needed to deliver new hi-tech products.. With ever stricter control standards and the shrinking size of high-tech products, confidently identifying smaller contaminants is becoming increasingly important and difficult. While high spatial resolution techniques exist for elemental and inorganic analysis such as SEM-EDX, identification of organic contamination at the micron and submicron scale has been challenging. The emerging technique of Optical Photothermal Infrared (O-PTIR) completely changes the infrared-based chemical analysis of small defects in microelectronics components[1]. In the past few decades, these specimens have posed significant difficulty to both conventional IR and Raman microscopy. Traditional FTIR/ QCL microscopy, though well-established and widely used, suffer from a number of key limitations, such as relatively poor spatial resolution (5-20 µm) and low sensitivity to domains smaller than 10 µm. Many times, these specimens have dimensions smaller than the wavelength of the mid-IR and are typically close to hard metallic interfaces, which could damage an ATR probe upon contacting the sample surface. Additionally, contamination can also be near a taller component or even within a narrow crevice, thus making ATR contact impossible. Non-contact modes of operation like reflection mode, are rarely used due to the dispersive scatter type artefacts, poor sensitivity exhibited in the spectra, and inability to search against commercial spectral databases.

The aforementioned issues are essentially eliminated with the advent and utilization of O-PTIR, which operates by exciting the sample with a broadly tunable and pulsed IR laser source that generates a modulated photothermal effect in the sample. The detection of this IR absorption is extracted and computed from this photothermal effect, by detecting the change in reflected probe beam intensity as a function of IR wavenumber tuning, thereby providing an IR absorbance spectrum, free of dispersive/scatter artefacts and fully searchable against existing IR (FTIR) spectral databases. Furthermore, it is

Figure 1: The mIRage, Infrared microscope, Photothermal Spectroscopy Corp., Santa Barbara, California

this short wavelength probe beam (typically 532 nm) that now defines the spatial resolution, rather than the long IR wavelengths as in traditional FTIR/QCL microscopy. Additionally, due to its unique system architecture and use of a Raman grade probe laser, this same laser can also serve as a Raman excitation source and thus when coupled with a Raman spectrometer, can provide for the first time, submicron and simultaneous IR+Raman microscopy at the same spot, same time, same spatial resolution.

Presented in this application are note are examples of difficult industrial, high tech failure analysis problems, from hard disk components (courtesy of Seagate Technology) and display components where defects of only a few microns are conclusively identified by submicron simultaneous IR+Raman. In the second example, a subsurface dark colored defect in a transistor display is identified.

Experimental

Samples were received and measured "as-is" with no further sample preparation. A mIRage+R, IR and Raman microscope was used, with a QCL source (1800-800 cm^{-1}) and a 532 nm probe beam/Raman excitation. Defects were located visually and targeted for reflection mode measurement. Typically, IR+Raman spectral measurement times were ~20 seconds per spectrum.

Spectra were searched using KnowItAll (Wiley) with access to their full IR and Raman databases (~ 300,000 spectra in total)

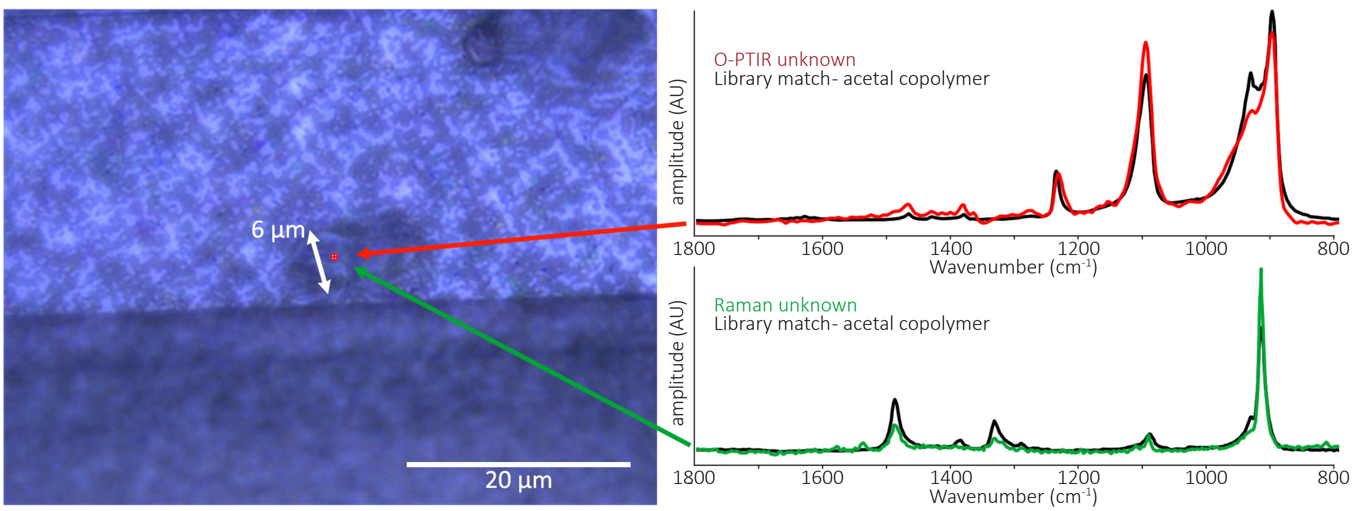

Figure 2: Left; visible image showing location of 6 µm defect, Upper Right; Comparison of unknown O-PTIR spectrum to nearest library match, Lower Right: Comparison of unknown Raman spectrum to nearest library match

Results and discussion

Figure 2 shows an example of the complementary and confirmatory O-PTIR and Raman spectral information that can be collected in reflection mode from a contamination particle that is ~6 µm wide. The O-PTIR and Raman spectra were simultaneously collected from the same spot and at the same spatial resolution. Although collected in reflection mode, the O-PTIR spectrum is fully comparable with library spectra from an FTIR transmission/ATR database. When the spectra were digitally searched against KnowItAll IR and Raman databases, it was determined that this particular contaminant is likely a polyether (acetal) material. The contaminates identified in these measurements include a small subset of the measurements Seagate Technology has measured using mIRage. The contamination includes a variety of materials plus foreign particles that can be introduced during research and development and include polymers, lubricants, etc. For this evaluation the mIRage was able to achieve better than 90% chemical identification.

There are many cases where contamination is not directly accessible with conventional IR instrumentation. Figure 3 shows a 20 µm dark contamination that is "underneath" the thin metal film, as can be seen from the white circular delamination of the metal film due

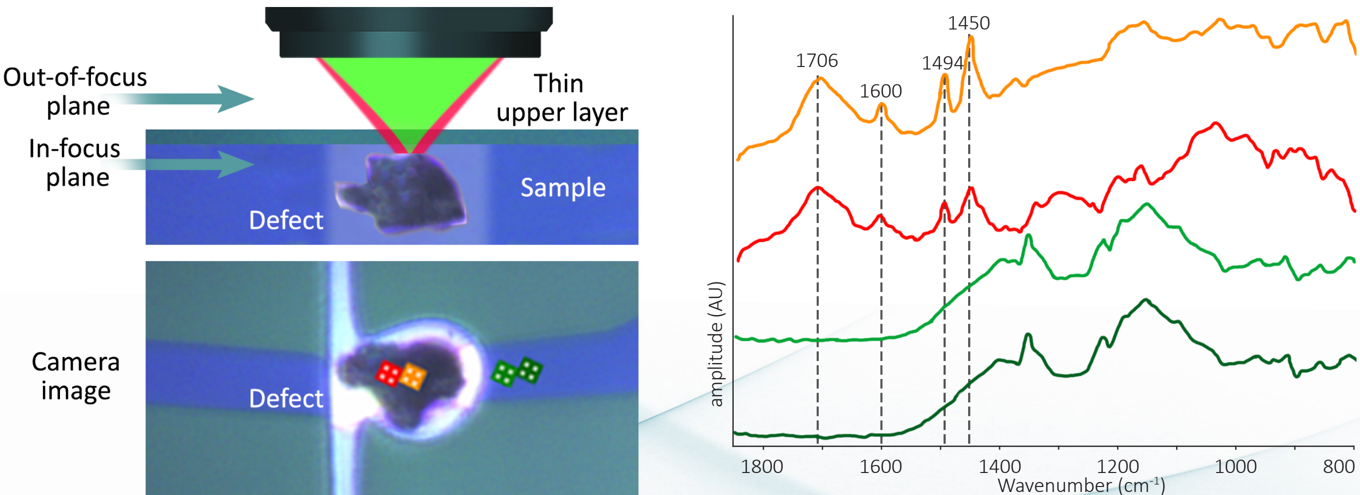

Figure 3: Upper Left; Schematic representation of sample and measurement, Lower Left; Visible camera image of defect, Right; O-PTIR spectra from on and off the defect. Colors correspond to markers on visible image

to the protrusion from this defective thin-film transistor display. Use of conventional ATR microscopy would have been limited by presence of the thin film, obstructing direct access to the contamination particle. The sample was able to be analyzed via O-PTIR without additional sample preparation or the need to perform any physical extraction of the particle. Examination of the spectra, particularly the presence of the strong broad IR absorbance band at the 1706 cm–1 band, suggests that this suspect contamination particle may have been a vulcanized styrene-butadiene rubber(SBR) that has oxidized to form a carboxylic acid.

Conclusion

We have introduced a revolutionary new vibrational microscope system, the mIRage IR microscope, that utilizes the proprietary technique of O-PTIR spectroscopy. The mIRage microscope delivers significant advances in IR microscopy, such as submicron measurements, non-contact (no ATR), non-destructive and sample preparation free analysis, point and shoot reflection mode analysis, free of any dispersive/scatter artefact that delivery FTIR transmission library searchable quality spectra. Seagate Technology chose the mIRage system to improve characterization of contamination earlier in the research and development of new manufacturing processes and have reported >90% accuracy in its chemical identificaiton . The fundamental principles and examples presented here display the advantages of O-PTIR in the identification of defects and contamination in hard drive and related electronic industries. O-PTIR spectroscopy has the potential to be disruptive in the important and growing field of IR microspectroscopy. In a further system enhancement, the addition of a Raman spectrometer expands its capabilities to now produce submicron simultaneous IR+Raman microscopy (same time, same spot, same spatial resolution), to give additional complementary and confirmatory information. Application areas are increasing and encompass all the traditional FT-IR fields such as polymers, defect analysis, and life science. Further adoption by the IR and Raman community will fuel exploration into new ways O-PTIR will break new limits.

Reference

1. Kansiz, M. et al., “Optical Photothermal Infrared Microspectroscopy with Simultaneous Raman – A New Non-Contact Failure Analysis Technique for Identification of <10 µm Organic Contamination in the Hard Drive and other Electronics Industries”, Microscopy Today, (2020), 28:3, p22-36

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