

INTRODUCTION

Purity is essential in semiconductor manufacturing – even trace amounts of specific contaminants can have a significant negative effect on semiconductor yield. Controlling contaminants in process streams using enabling technologies, like liquid filtration and purification, is a vital way to improve operating efficiency.

Contamination in liquid processes can be divided into three categories: metal ionic contamination, organics, and particles. Only when the contaminant origins are identified can the impurities be prevented from reaching the wafer surface. In this study, we leverage multi-modal imaging and analysis techniques to identify sources of microcontamination.

This method allows for the creation of a single sample that is then analyzed with confocal laser 3D microscopy, atomic force microscopy (AFM), scanning electron microscopy (SEM), and electron dispersive spectroscopy (EDS), Micro-Raman, Micro-Fourier-transform infrared spectroscopy (FTIR), and Nano-infrared spectroscopy (IR). The combined data collected from the different methods provides image, volume, and possible composition or organic functional groups of the contamination of interest. This “one sample, multiple results” analysis is an efficient and powerful method to drive the evolution of enabling technologies that control contamination in semiconductor processes.

EXPERIMENTAL

Instruments:

- 3D Laser Confocal Microscope (VK-X, Keyence)
- SEM (FEI Quanta200), DES (Oxford Instrument, Inca Energy 300)
- FastScan AFM (Bruker)
- FTIR Hyperion 1000 with a Tensor 27 spectrometer (Bruker)
- Raman microspectrometer (LabRam, Horiba-Jobin-Yvon)
- AFM IR (NanoIR2-FS, Anasys Instrument/Bruker)

Samples: De-ionized water (DIW) or other liquid samples from semiconductor application processes

RESULTS AND DISCUSSION

Imaging and EDS analysis

When a liquid sample is dried onto a surface, there are always residues left behind that contain particles and non-volatile impurities. The amount of residue reflects the cleanliness of the sample. Different imaging techniques each provide unique information that can help identify the contaminants.

- **3D confocal microscopy** provides topographic images that can be used to estimate the residue amount.
- **SEM** provides a further-detailed residue image
- **EDS** provides elemental compositions on different portions of the residue
- **AFM** provides detailed topography information in the nanometer range.

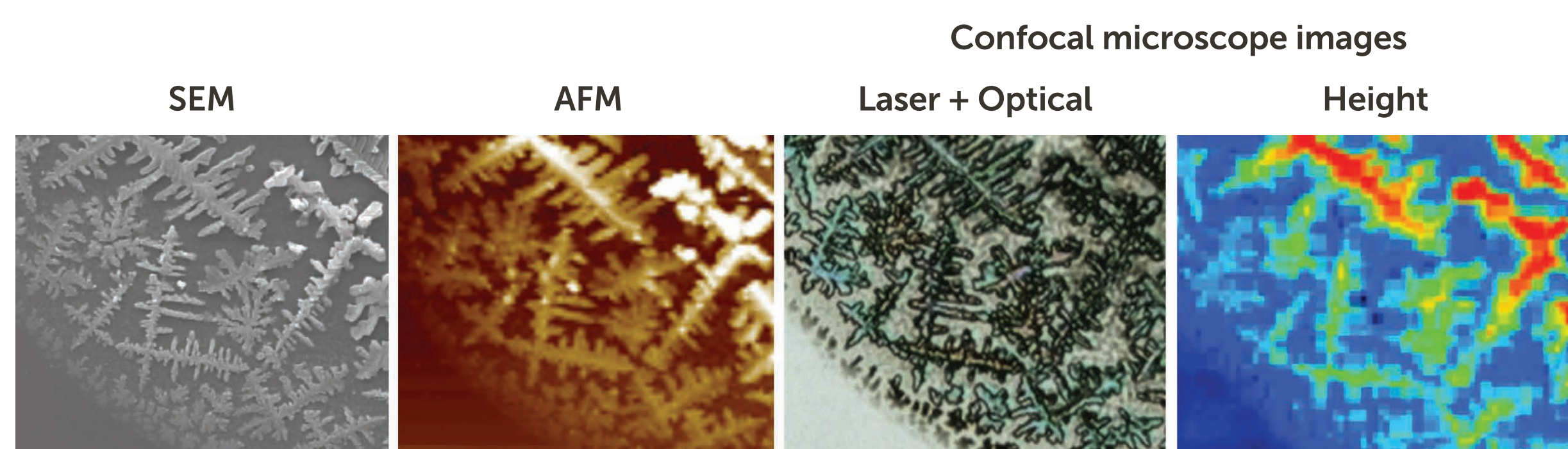


Figure 1. A comparison of different imaging techniques. The sample is collected from a wet-etch process, SEM/EDS result shows the residue contains C, O, and Al.

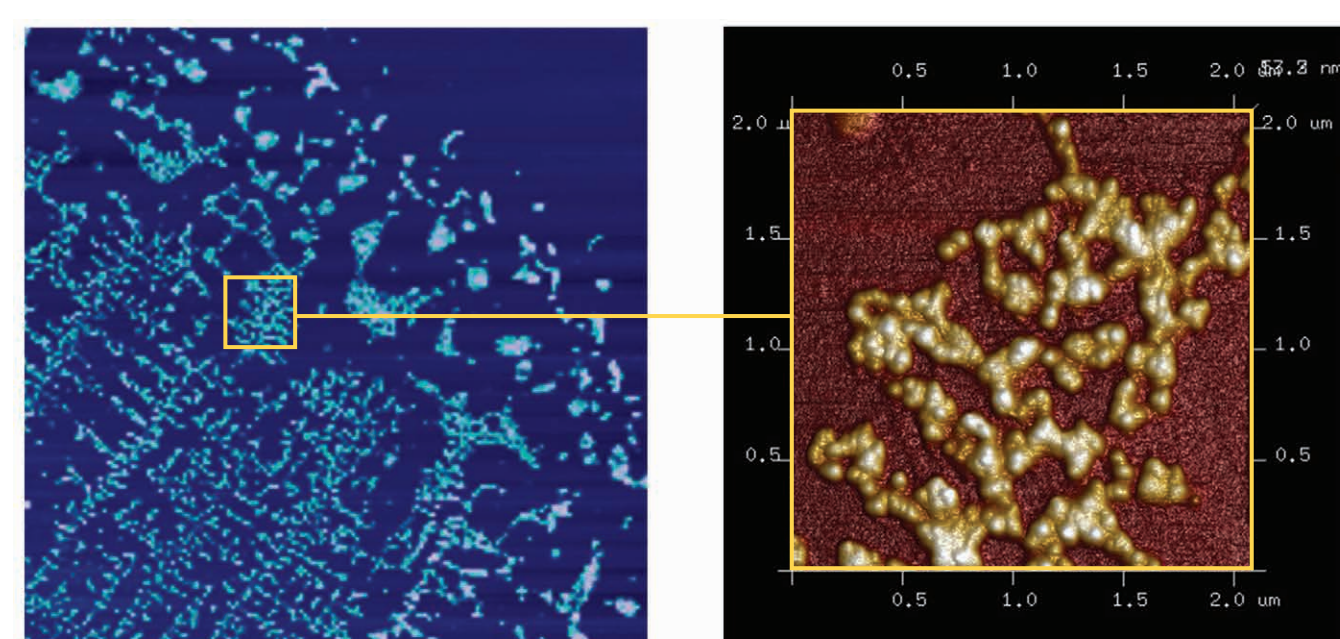


Figure 2. The residue collected from a DIW sample. The image on the right is a close-up of AFM which shows aggregated particles.

Micro-spectra techniques for analyzing unknowns

Most sample residues contain organic impurities. For the quickest and easiest method to detect the organics, micro-IR, or micro-Raman are used because no further sample preparation is needed after imaging is complete. Although these methods may not be able to identify the sample unknowns, they can provide organic functional groups and fingerprint information. A FTIR library can also help to identify possible chemical structures. Figure 3 shows two FTIR spectra from DIW samples. The two examples show entirely different spectra, indicating that the contamination from each sample is different.

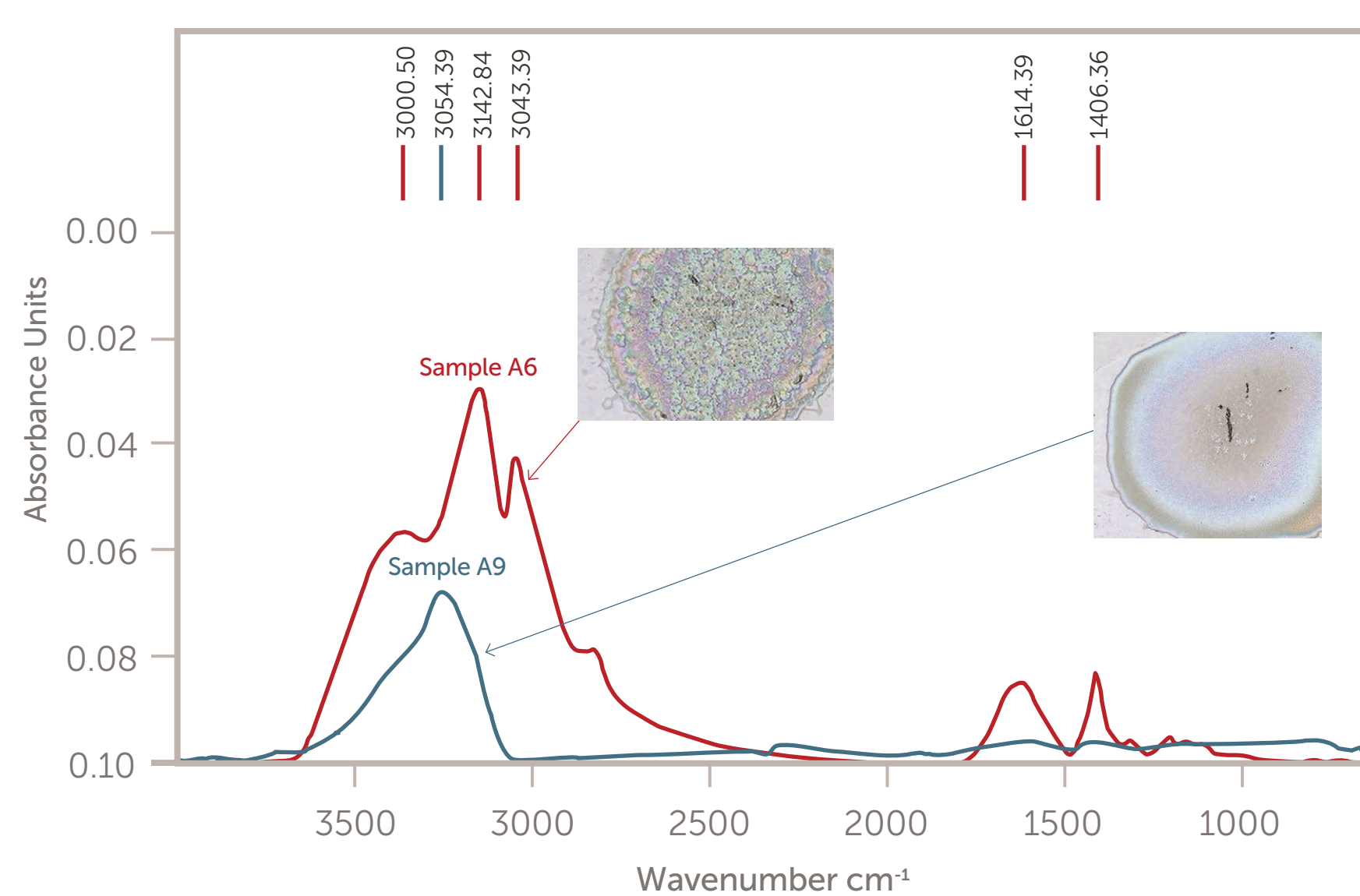


Figure 3. Micro-IR spectra (scan spots ~30 μm). Two residues collected from different sources of DI water show entirely different IR adsorption peaks.

Huth, et al¹ used PMMA as a sample and confirmed that Nano-FTIR could reach a 20 nm spatial resolution. Their results also show the Nano-FTIR absorption spectra correlate well with conventional FTIR absorption spectra. Figure 4 is Nano-IR test results for a DIW sample. The spectra can be searched and compared with library data system of FTIR for further analysis.

Contact AFM-IR Spectra

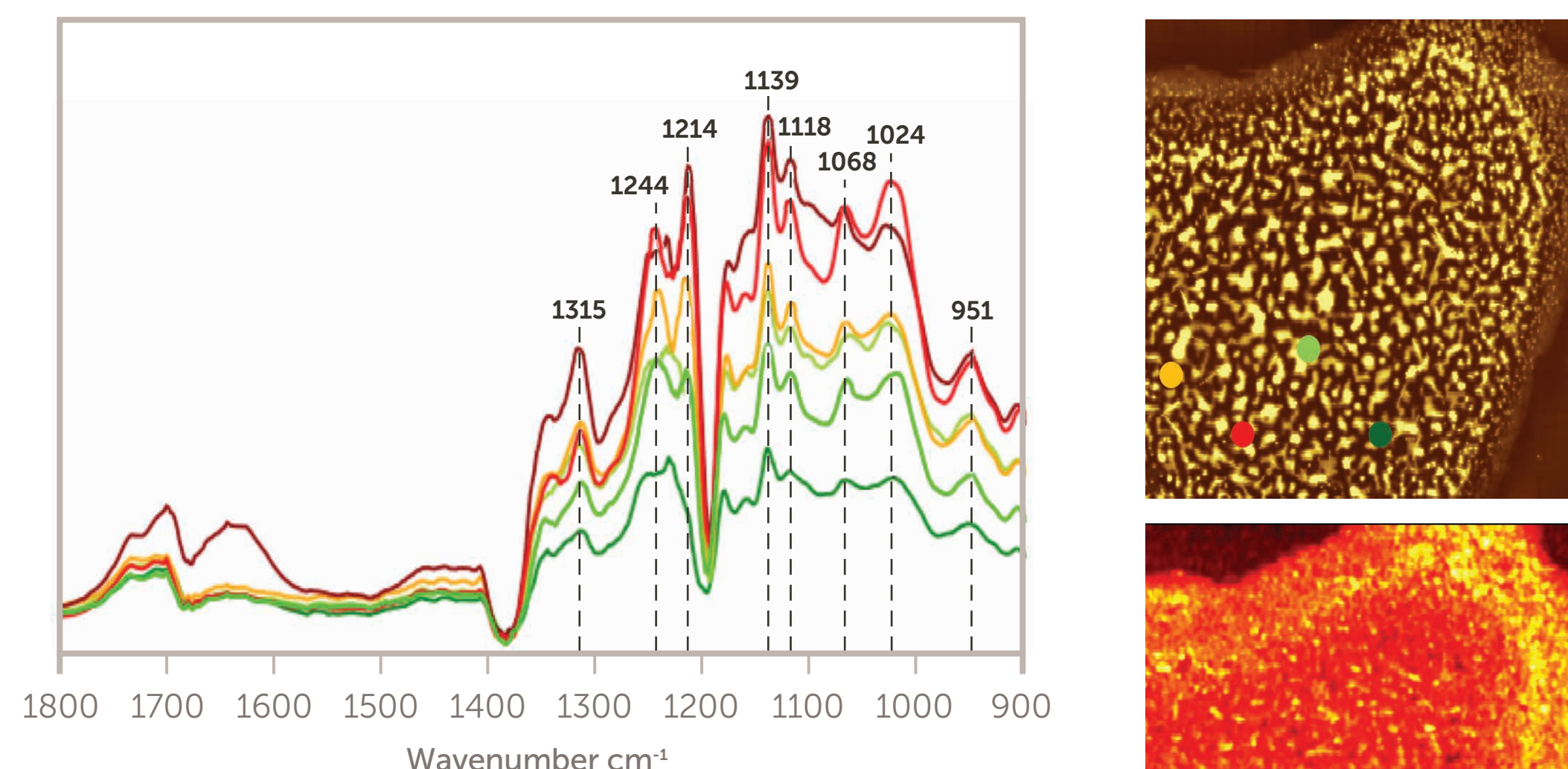


Figure 4. Chart: Selected spots of Nano-IR spectra; right top image: AFM Height image of the sample; right bottom image: IR absorption map showing the compounds with the IR peak at 1140 cm⁻¹ (yellow means containing more such a compound).

CONCLUSIONS

Multi-modal imaging and analysis is an efficient method to analyze micro-contaminants in liquid samples. EDS, FTIR, Raman, or Nano-IR can be used to further identify the potential sources of contamination. The information collected with 3D Confocal Microscope and AFM provide topographic images which can be used for evaluating filtration effectiveness. SEM can be used for analysis on particles and image details. “One sample, multiple results” analyses are very efficient in collecting a vast amount of information from one sample. This methodology reduces analysis time, avoids possible variation from the process of sampling, and provided a rich dataset from which to identify contaminants that are of utmost importance in liquid semiconductor processes.

REFERENCE

1. F. Huth, et al., “Nano-FTIR Absorption Spectroscopy of Molecular Fingerprints at 20 nm Spatial Resolution.” Nano Lett. 12, 3973–3978 (2012).

ACKNOWLEDGEMENT

The authors are very grateful to Jennifer Braggin and Thomas Phely-Bobin for their support and discussions

*Corresponding author: suwen.liu@entegris.com