



Enhancing Material Inspection and Characterization Information and Data Integrity

By Combining Light and Scanning Electron Microscopy in
a Correlative Workflow

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Both Light Microscopy (LM) and Scanning Electron Microscopy (SEM) are well established imaging techniques for work piece (sample) inspection and characterization in applications such as industrial Quality Assurance (QA) and materials characterization. Both techniques exhibit distinctly different, yet complementary imaging and analytical attributes; however, these microscopes are rarely found sitting side-by-side in materials (QA) or research laboratories.

On our mission to match microscopy solutions to customer challenges by leveraging the three basic types of microscopy technologies (Light, Electron and X-ray), we at ZEISS, are able to focus on developing multi-modal workflows that make it faster and easier for researchers and engineers to get a complete picture of their samples. Our core competencies in both LM and SEM allow us to recognize the synergies among these two different microscopy applications, and our DNA compels us to explore and further develop correlative microscopy solutions to improve information gathering and productivity for our customers in both research and industry.

LM and SEM: More Powerful Together

The attributes of SEM for high resolution material inspection and characterization are well known and have long been established in a wide range of business sectors and research facilities. Likewise, light microscopy is widely used to quickly observe and record sample features at lower magnifications.

Inherent to SEM are several methods of data acquisition that deliver different kinds of information about the sample being studied. Secondary electron imaging provides sub-micrometer scale resolution to reveal surface details beyond the resolution limit of light microscopes. Backscattered electron imaging on the other hand, produces a distinctly different contrast mechanism to reveal material density differences as shades of grey. When equipped with an Energy-Dispersive X-ray Spectrometer (EDS), the SEM is able to produce analytical information via the interaction between the electron beam and the sample surface to yield high spatial resolution, elemental chemistry data that can be quantified to determine material composition.

The ZEISS EVO MA10, the base model in ZEISS' extensive range of SEMs, is a versatile instrument capable of accommodating the typical samples studied in the industrial QA or materials research lab. The EVO MA10 is designed from the start to support an upgrade path, with a multi-port specimen chamber that accommodates multiple accessories, variable pressure vacuum technology to enable imaging of non-conductive samples without additional preparation, and a specimen stage that also accommodates larger and heavier samples.

The field of light microscopy has greatly benefitted from a recent innovation that is not always well known to SEM users: the *digital* light microscope. Digital light microscopes have overcome many of the challenges with traditional light microscopy, namely surface reflections and depth of focus. A combination of co-axial and segmented LED ring lighting delivers unprecedented illumination flexibility to mitigate the reflections from the sample surface that were previously

suppressed with polarization filters. Additionally, digital light microscopy enables fast, automated acquisition and processing of a large number of images at different focus settings (image Z-stack). This reveals light microscopy images with unrestricted depth of focus, or even 3D images of sample features that can be viewed from different angles. The data presented in this paper comes from the ZEISS Smartzoom 5 wide field zoom digital light microscope.

The Subject Samples

We will demonstrate the range of complementary data that can be acquired from digital light microscopy and SEM using three classical materials samples from the QA or engineering environment.

The first sample is an industrial surface coating (Figure 1a). The sample was the subject of inspection, characterization and documentation with both LM and SEM to support an assessment of the coating's attributes.

The second sample is a printed circuit board (Figure 1b) with soldering structures (solder bumps) that require high resolution inspection.

The third sample is a fractured metal rod, with a fracture surface revealing micro-cracks and other anomalies requiring inspection and characterization to determine the root cause of failure (Figure 1c).

The most obvious attribute of the light microscope is its ability to bridge the magnification range required for basic inspection, imaging and documentation, from low magnifications, to microscopic magnifications up to values of 1000x or more. SEMs are generally limited to lowest magnifications around 20x, and therefore are not ideally suited for basic sample inspection and documentation.

In the correlative process, we therefore begin with overview images from the Smartzoom 5 such as presented in Figure 1.

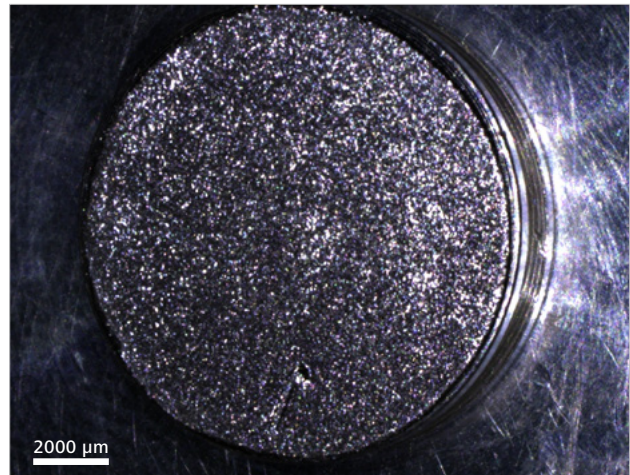


Figure 1a Overview image of an industrial (car paint) coating applied to an aluminum stub.

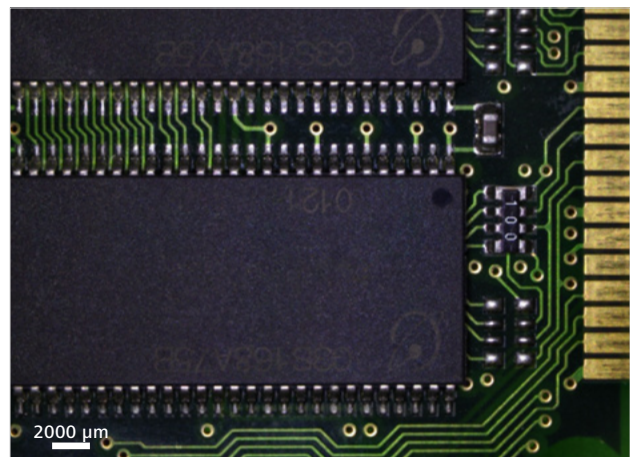


Figure 1b Overview image of structures on a Printed Circuit Board (PCB).



Figure 1c Overview image acquired with the digital light microscope of a metal rod fracture surface.

Innovative Imaging Attributes of the Digital Light Microscope

The next step in the correlative process is to zoom in and inspect samples with the digital microscope at elevated magnifications.

Two particular attributes of the digital microscope mitigate historical challenges to do with surface reflections, and limited depth of focus.

As mentioned earlier, digital light microscopes employ a segmented LED ring lighting system integrated in the objectives that can be controlled manually or automatically to yield illumination settings that remove reflections, as illustrated in Figures 2a and b. Prior to the availability of this functionality, inspection tasks on surfaces prone to reflections were typically handled with SEM. Having these glare removal capabilities on the digital light microscope frees capacity in the SEM for the material characterization tasks.



Figure 2a Features on a PCB revealing clear reflections from the segmented LED ring light (all switched on).

Automating acquisition of a number of images within a predefined focus range (Z-stack), then following with image reconstruction means samples can be documented either in 2D with enhanced depth of focus (Figure 3a), or in 3D for elements with a definite form factor, such as the solder bump shown in Figure 3b. A further attribute of light microscopy imaging is its ability to measure contour, form or surface dimensions as 3D topographical and cross sectional image data, also shown in Figure 3b.

Imaging and Analysis Attributes of the Scanning Electron Microscope

The next step in the correlative process is to transfer samples to the SEM and collect the data not attainable from the (digital) light microscope.

This step is facilitated by ZEISS' "Shuttle & Find" combined hardware/software solution. Shuttle & Find ensures fast and



Figure 2b Same PCB now imaged with "glare removal" illumination settings that suppress the reflections visible in Fig. 2a.

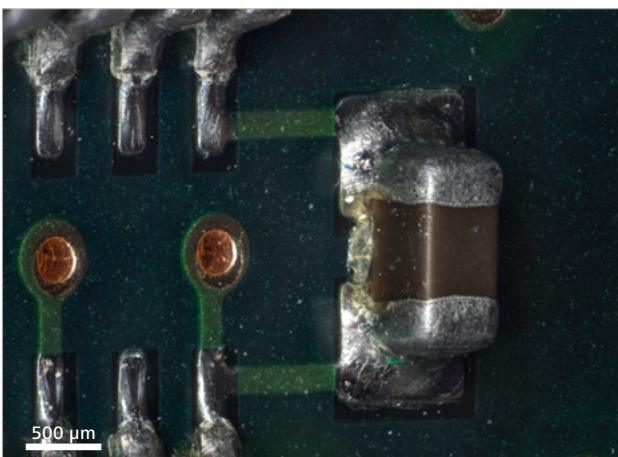


Figure 3a PCB structure imaged at an angle with enhanced depth of focus from left to right on the image.

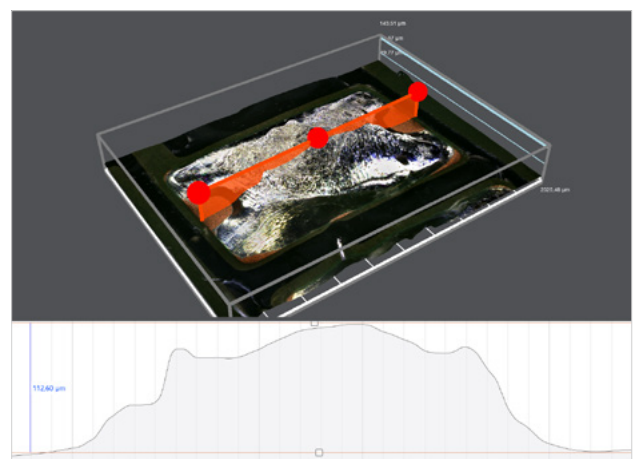


Figure 3b Solder bump image reconstructed in 3D, with a height / depth measurement profile.

secure transfer of the sample between microscopes, relocation of the predefined regions of interest, and access to the data already collected on the digital microscope.

The imaging and analysis attributes of the Scanning Electron Microscope relate directly to the interaction of the electron beam with the sample surface. As a result of this interaction, the sample emits secondary electrons, backscattered electrons and X-rays.

Secondary electron imaging highlights edges to reveal sub-micrometer detail of the sample surface. This effect can be seen clearly in the secondary electron image of the metal fracture (Figure 4a).

Backscattered electron intensity, on the other hand, scales with material density, whereby dense materials appear bright, and lower density materials appear dark. This qualitative aspect of backscattered imaging already hints at compositional differences between the coating particles, which appear with two distinct shades of grey in the backscattered image (Figure 4b).

For a definitive picture of sample composition, the SEM utilizes an Energy Dispersive Spectrometer (EDS) detector to yield the X-ray emission energy spectrum. The position of the intensity peak on the energy scale indicates which element accounts for the X-ray emission at that particular energy. The darker coating particles reveal carbon and oxygen peaks in the X-ray spectrum, indicating the presence of a paint-like coating on aluminum particles (Figure 4c).

Data Reporting and Archiving

The final step in the correlative process is the reporting and archiving of combined data from the (digital) light microscope and SEM.

The combined light and electron microscopy data from typical industrial samples, such as the three presented in this paper, demonstrate a more compelling and convincing story than either of these two microscopies would have been able to reveal if used in isolation (Figure 5). Light microscopy



Figure 4a Secondary electron image of metal fracture, revealing sub-micrometer detail of the fracture surface morphology.

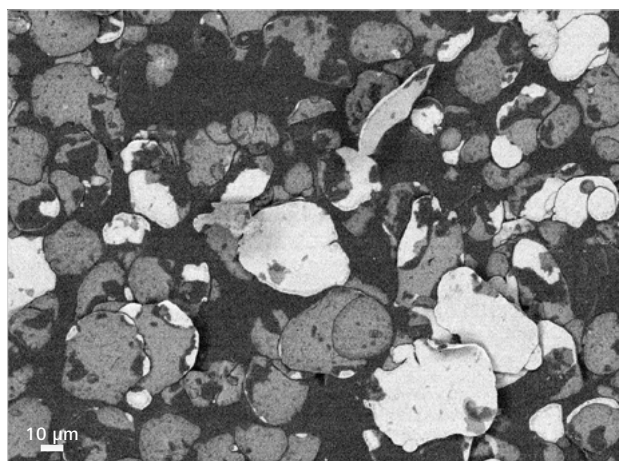


Figure 4b Backscattered electron image of the coating, revealing both bright and dark coating particles, indicating a compositional difference between the two particle types.

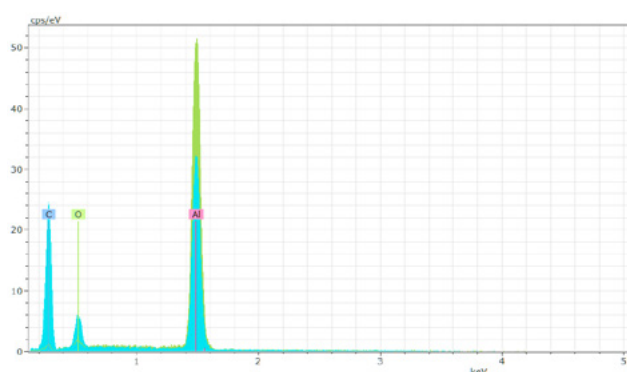


Figure 4c EDS spectra of bright coating particles (in green, revealing only aluminum) and dark coating particles (in blue, revealing carbon and oxygen in addition to Al).

yields data to which engineers outside the materials characterization or industrial QA laboratory can easily relate, because of familiar attributes such as superior field of view, color, depth of focus and 3D visualization.

The scanning electron microscope then delivers the data not attainable by light microscopy to complement the sample's story with superior sub-micrometer detail and qualitative and quantitative material compositional information.

Equally important to data acquisition is the ability to now report and archive the data such that engineers outside the laboratory can respond to the findings. It is critical that the data is retained in compliance with ISO and other quality management standards and best practices.

To prevent the LM and SEM data from being separated on the respective PCs or network data folders, the ZEISS correlative workflow implements our ZEN 2 core imaging and image

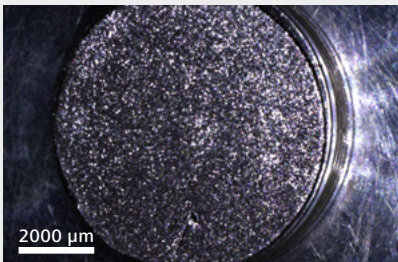
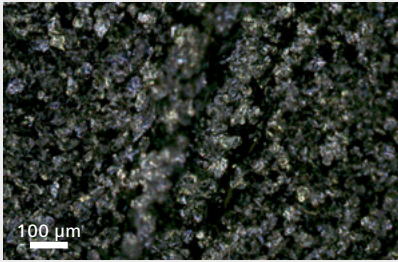
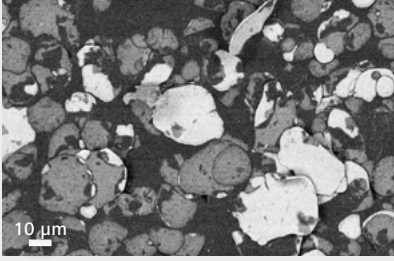
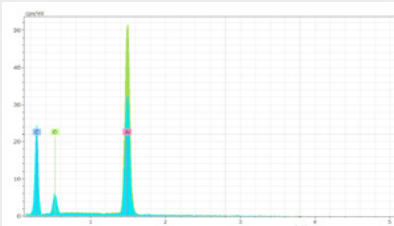
Information	Digital Microscope Data	SEM Data
Sample overview documenting size, shape and other attributes.	 <p data-bbox="596 1048 995 1097"><i>Overview image of an industrial (car paint) coating applied to an aluminum stub</i></p>	<p data-bbox="1027 1048 1422 1146">SEM's typically are more limited in attainable field of view. SEM also does not capture the quality attributes of the coating, e.g. reflectivity, color, etc...</p>
Surface coating inspection, revealing particles with different reflectivity, as well as a coating defect.	 <p data-bbox="596 1384 995 1411"><i>Illumination optimized LM image</i></p>	
Characterization of the compositional heterogeneity of coating particles, revealing two particle types appearing as distinctly different gray values in the BSE image.	<p data-bbox="596 1688 995 1738">Compositional information not available from the light microscope.</p>	 <p data-bbox="1027 1688 1422 1715"><i>Backscattered electron image</i></p>
Determination of the chemical composition from the two types of coating particles.		 <p data-bbox="1027 1966 1422 1993"><i>EDS spectrum</i></p>

Figure 5 Summary of both light and scanning electron microscopy data acquired from the industrial coating, revealing a complete assessment of the coating attributes from optical inspection to detailed characterization and analysis of the coating particles.

analysis software. The Shuttle & Find interface handles the correlation of both the LM and SEM data. ZEN 2 core software enables LM and SEM image overlay and stores the data from the LM and SEM in the same (network) folder to assure that both datasets are instantly available for reporting, and to guarantee data integrity for subsequent archiving.

Summary

The digital innovations in light microscopy have mitigated some of the classical limitations of this technique, making the digital light microscope a perfect complement to the scanning electron microscope, with its high resolution imaging and analytical capabilities. Both microscopies have their specific merits, so pairing them seamlessly in a workflow enables acquisition of a much broader data set than could be collected using these techniques independently. The software facilitating the correlative microscopy workflow aids reporting of the combined LM and SEM data, and ensures data integrity to meet ISO and other quality management requirements. Together, this solution suite delivers a powerful combination of data for industrial QA and materials characterization or research laboratories.



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