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Applying dosage to improve image resolution in fast multielement mapping with LIBS

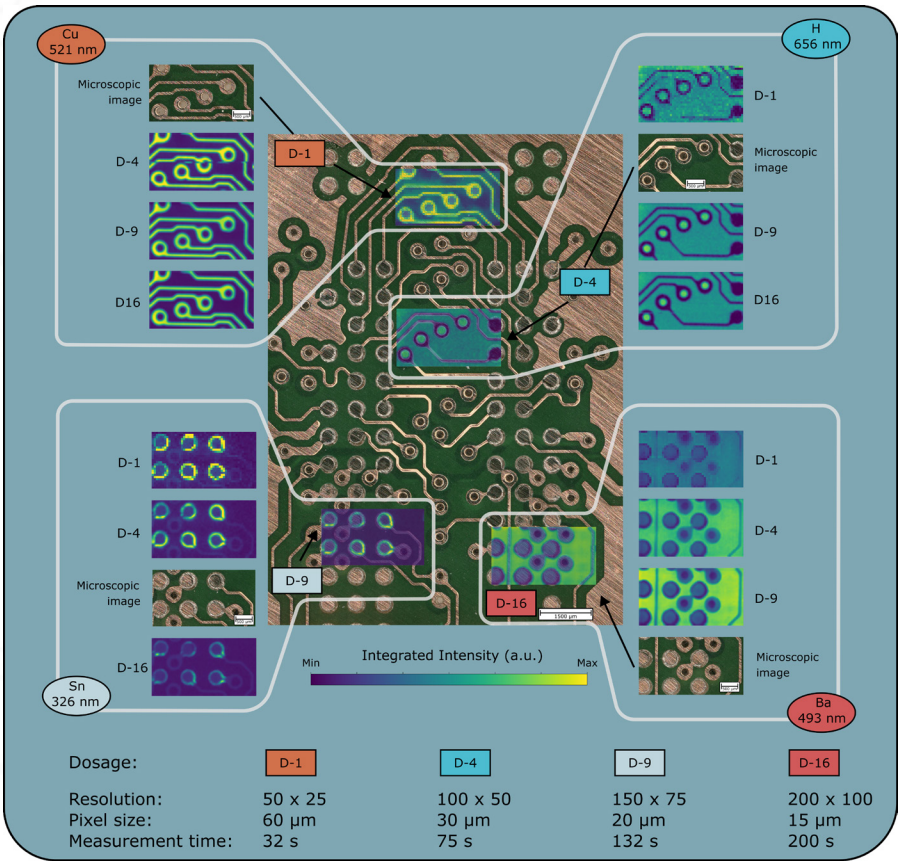

Figure 1. LIBS images of different locations on a PCB recorded using different levels of laser dosage.

Brief

LIBS in combination with 193 nm laser ablation is quite new since this laser type was conventionally preferred for LA-ICPMS analysis, whereas LIBS most commonly used longer wavelengths in the IR regime. However, the high energy stability, beam homogeneity, repetition rate and stage precision of the new generation of excimer LA instruments are also very attractive for LIBS analysis, especially since the same LA instrument can be used for both techniques (also simultaneously). Furthermore, LIBS can additionally measure molecular fragments (e.g., C_2, CN, CaF), and non-metals (H, N, O, Cl, F), which can be extremely beneficial for various analyses. To reach satisfying sensitivity, LIBS requires higher pulse energies and larger spot sizes than LA-ICPMS. However, to keep the lateral resolution of the resulting elemental images high, an oversampling approach can be applied, which is demonstrated in the work presented here.

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Electronic components analysis

Process and quality control in the electronics industry often demand spatially resolved elemental information on their raw materials, their products between specific process steps or on the final products. This enables the identification of inhomogeneities or contaminations within used materials or of processing anomalies, thereby facilitating an adequate response to keep production on track and within certifications. Additionally, for failure analysis of electronic components, spatially resolved chemical analysis methods are indispensable. Laser induced breakdown spectroscopy (LIBS) is a method perfectly suited for the aforementioned requirements, providing fast elemental imaging analysis with the capability to resolve micro-structured samples. In order to maximize the spatial resolution without compromising sensitivity, which is typically associated with using smaller laser spot sizes, an attractive solution is to implement overlapping shots resulting in an increased laser dosage.

Dosage

In this context, the term dosage describes the number of laser shots that are fired onto a specific sample location, from which data is evaluated to one pixel of the resulting image. Depending on the point of view it follows two purposes: (I) Improving the image resolution without significant losses in the signal-to-noise ratio (S/N) of the analysis – this is done using a fixed spot size and creating a pattern of overlapping shots, i.e. applying a denser laser shot position raster. (II) Improving the S/N without significant losses of image resolution, which is performed by increasing the spot size while the laser shot position raster remains identical. Identically, both result in oversampling, only viewed from the opposite perspective. Performing such an oversampling measurement approach can significantly improve the image quality and determine if the performed analysis is fit for purpose in terms of required sensitivity and resolution.

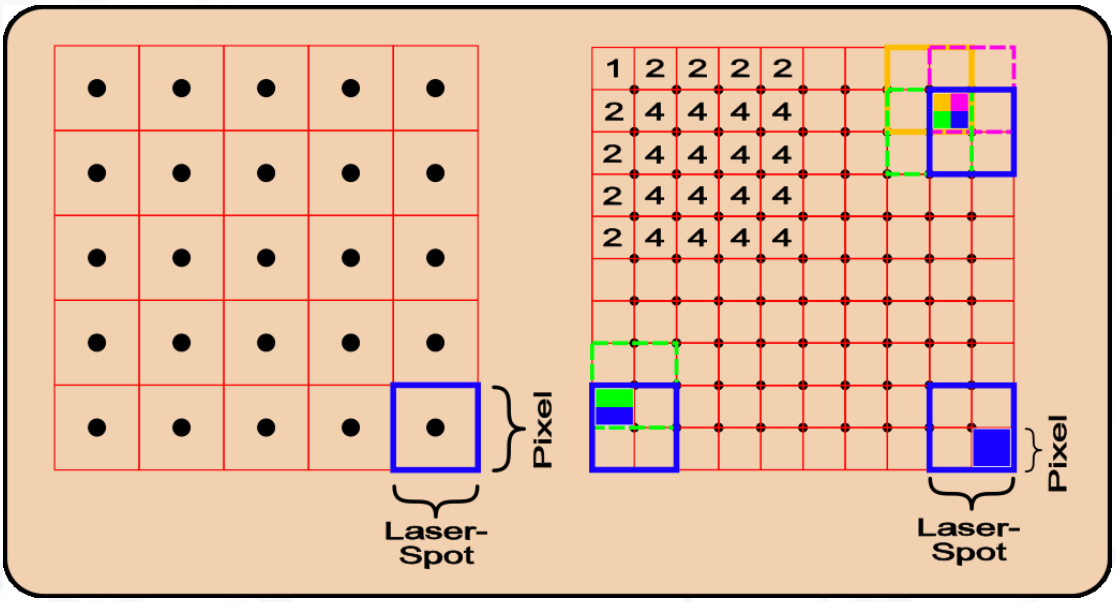

Figure 2. Schematic depiction of the laser spot and pixel raster with dosage 1 (left) resulting from non-overlapping shots and dosage 4 (right) resulting from 50% overlapping shots in x and y direction.

Dosage *(continued)*

In Figure 2 (left side) the image illustrates the case with a laser dosage of one, with the pixel raster being identical to the laser shot raster and the size of each pixel corresponds to the laser spot size. Figure 2 (right side) demonstrates the case where the laser spot size (blue square) remains identical, while introducing 50% overlapping shots in x and y direction, the laser shot raster is four times denser introducing a dosage of four to each pixel. Each pixel intensity is calculated from the intensities of all laser shots that include the corresponding sample location, as demonstrated using different colors in the illustration.

Instrumentation – imageGEO193^{LIBS} and ESLumen

The imageGEO193^{LIBS} laser ablation system, provides ports for attachable fiber optics at its analytical sampling cup, enabling collection of element specific emission from the laser induced plasma at the sample surface. The laser provides circular and rectangular beam apertures for spot sizes from 1 to 200 µm, and high energy stability and energy homogeneity throughout the spot size, ensuring even ablation craters. The recently released ESLumen LIBS spectrometer is capable of broadband acquisition using a 5-channel CMOS detection (also available in 6 channels), covering a wavelength range from ~190-1100 nm with up to 1000 Hz. It is directly connected to the imageGEO193^{LIBS} via trigger cable, data transfer, and fiber optical cable. The acquisition parameters and data recording for both instruments are controlled from the ActiveView2 software, providing a smooth workflow during initial testing, method optimization and final measurements.

Table 1. Measurement parameters for the imageGEO193^{LIBS} LA instrument and the ESLumen LIBS spectrometer.

imageGEO193^{LIBS} Parameters	Value
Fluence	5 J/cm^2
Spot size	60 x 60 µm^2 (rectangular aperture)
Overlap / Dosage	0 µm / 1 | 30 µm / 4 | 40 µm / 9 | 45 µm / 16
Repetition rate	200 Hz
Image dimensions	3000 x 1500 µm^2
Measurement time / Total pixels (px)	32 s / 50x25 px (1250) | 75 s / 100x50 px (5000) | 132 s / 150x75 px (11250) | 200 s / 200x100 px (20000)
Chamber Helium flow	800 mL/min
ESLumen Parameters	**Value**
Spectrometer delay	0.1 µs
Integration width	200 µs

[a]Overlap was carried out in x and y dimensions. Dosage D = Dx * Dy

Sample

A printed circuit board (PCB) is a very suitable sample to demonstrate the capability of LIBS for fast multielement imaging and the influence of different dosage settings on the resulting image resolution and measurement time. A variety of different elements, such as Cu in conducting tracks, Ni / Sn on soldering pads and solders, Sr / Ba as well as C / H / O from the fabric, polymeric resin and binder. Additionally, impurities such as Na can be present and locally different. For this study, a DRAM unit was cut into pieces of approximately 20 x 30 mm^2. The DRAM chips were removed from the board, and the underlying PCB pieces were embedded in epoxy and then grinded (SiC paper 300-1200 mesh) until the copper conducting traces and soldering pads were partially uncovered of the conformal coating. A photograph of the embedded sample after the measurements is shown in Figure 3 and a schematic cross section of the layout of a PCB is demonstrated in Figure 4.

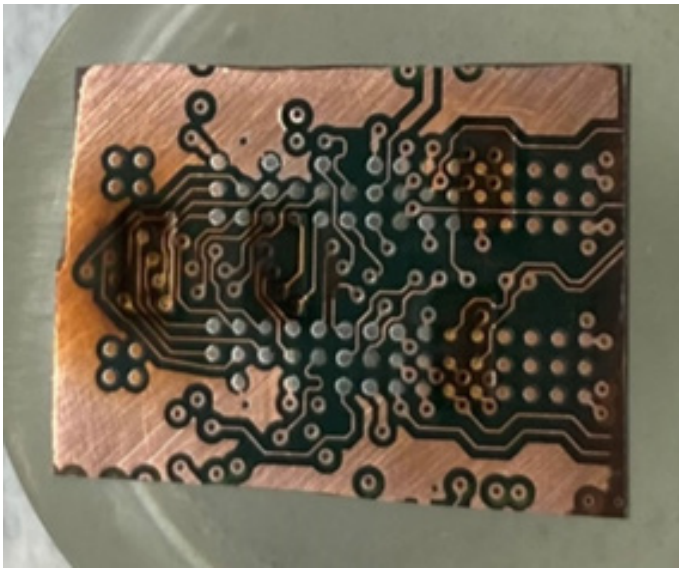

Figure 3. Epoxy embedded PCB piece from DRAM unit after the LIBS measurements. At each position the experiments using dosages of 1, 4, 9, and 16 were performed directly in sequence resulting in total ablation of 30 layers at each position, which corresponds to a depth of approximately 10-20 µm.

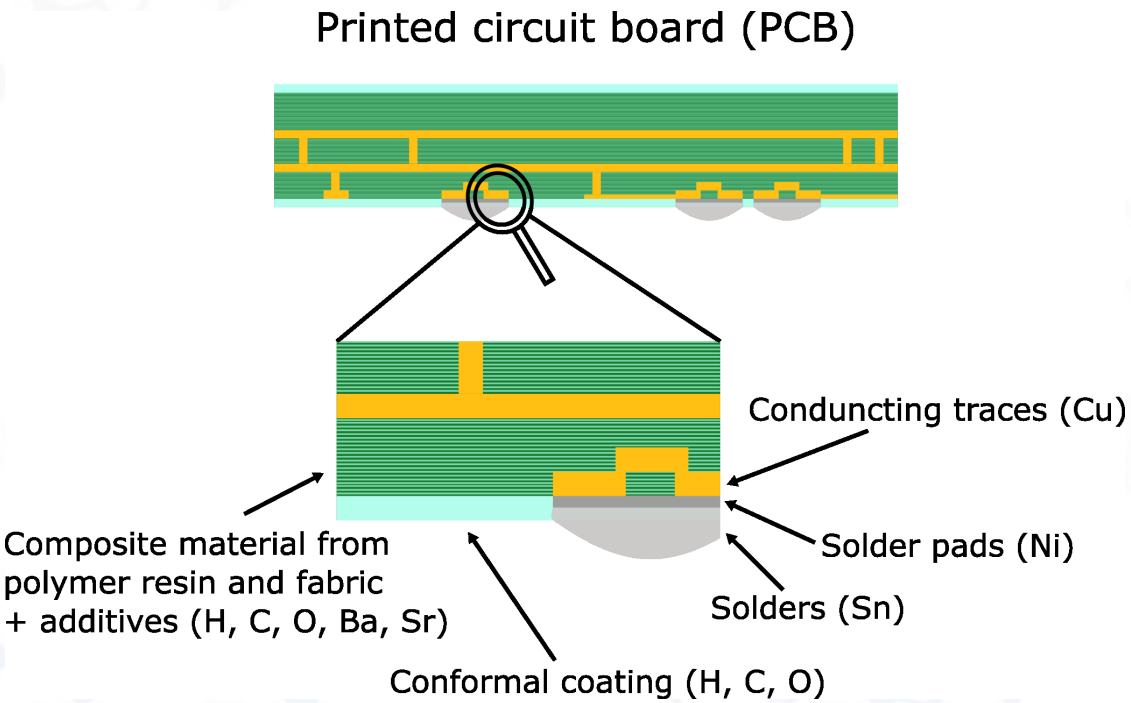

Figure 4. Schematic cross section of a typical PCB structure's composition.

Measurement and evaluation

As shown in Figure 5, ActiveView2 is used to simultaneously control the imageGEO193^{LIBS} excimer laser ablation system and the ESLumen LIBS spectrometer via the Lumen Add-in. Initial testing and method optimization by, e.g., varying laser energy, spot size, etc., can be performed with live viewing of the resulting LIBS spectra. With optimized parameters, a queue is created, and the remaining measurements are performed automated and each pattern from AV2 results in a separate LIBS data file.

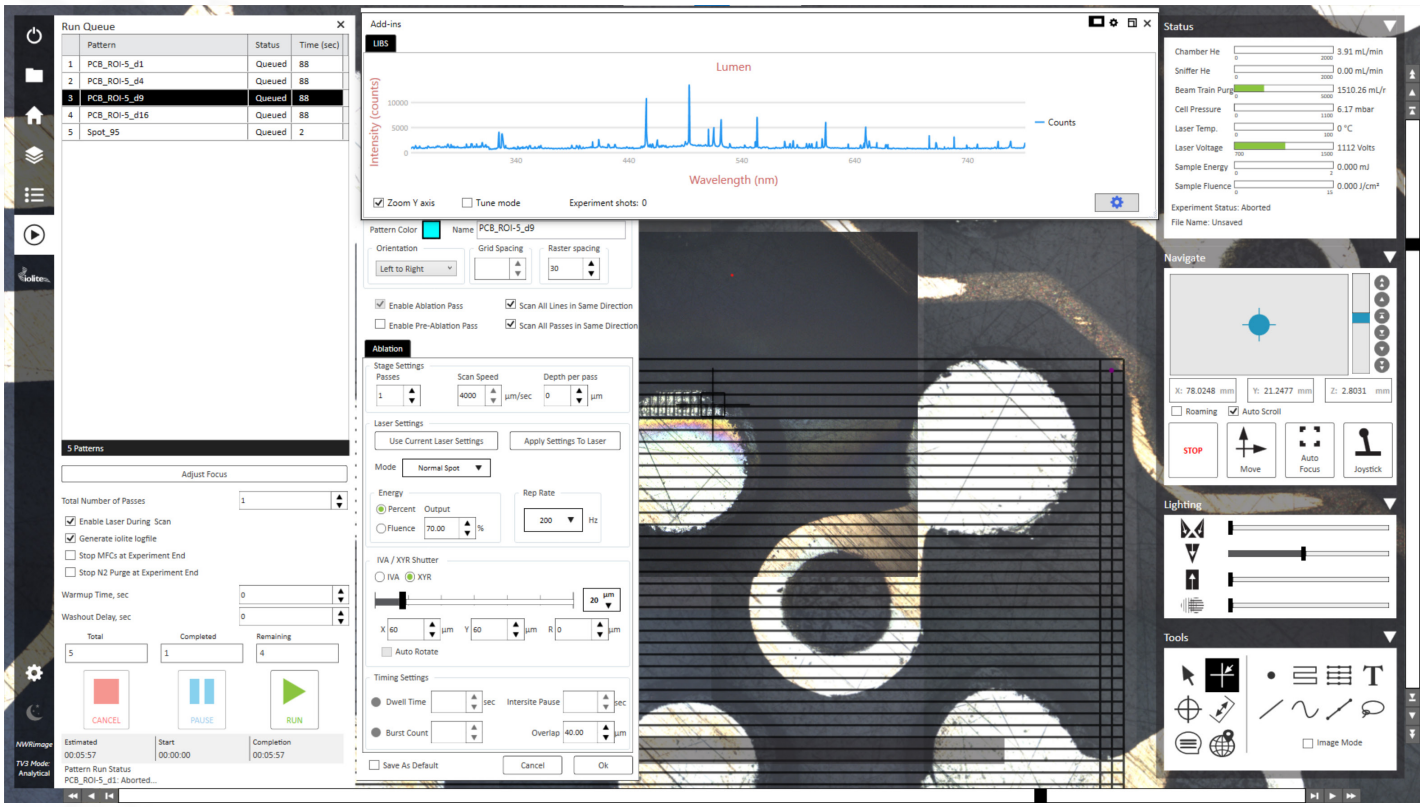

Figure 5. Screenshot of the ActiveView2 software showing the queue window, pattern settings window and LIBS Add-in to control the laser parameters for the imageGEO193^{LIBS} and the LIBS acquisition parameters for ESLumen.

The recorded LIBS spectra are imported into the Iolite software for data evaluation using the LIBS Importer (Figure 6, upper section). Spectra can be accumulated to facilitate easier recognition of emission lines. Suggestions of prominent emission lines from a database can be displayed in the broadband spectra for each element of the periodic table. Next, emission lines are selected and can be either automatically or manually annotated and added into the peak selection. Finally, for each selected line, the integration parameters (ranges, background subtraction) are set, and the dataset is imported into iolite for further image evaluation. The deconvolution of the pixel matrices for different dosage levels and the displaying is then performed in iolite's Imaging window (Figure 6, bottom section). Displaying properties and parameters, such as scale bar, color scale, lower and upper scaling values, various image filters and further details can be adjusted here. Then, the images can be exported as pixel matrices as well as in different common image file types.

Measurement and evaluation *(continued)*

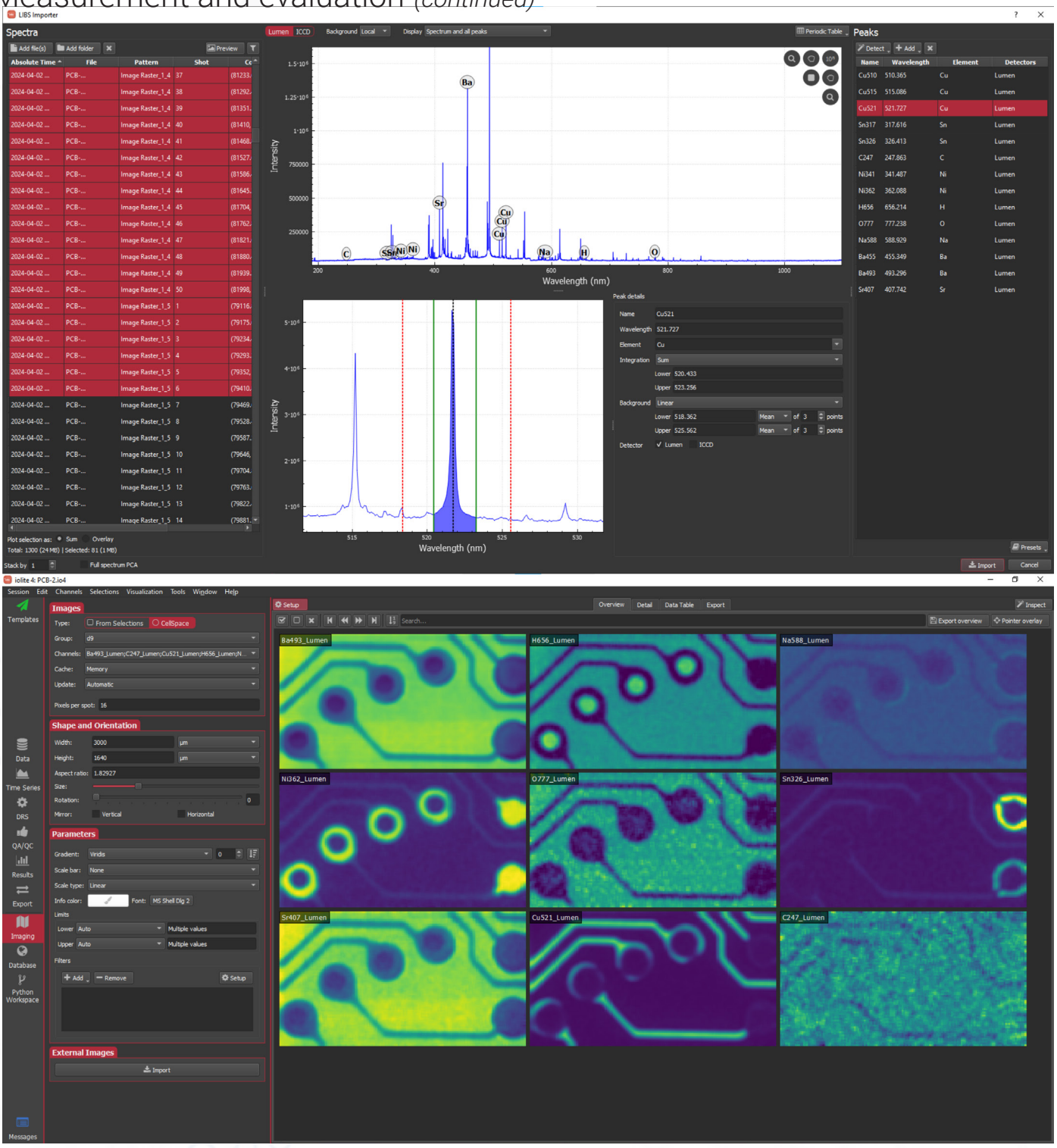

Figure 6. Upper section: Screenshot of the iolite LIBS Importer: Selected spectra files to the left, the broadband spectra at the top, selected elemental emissions to the right, and the evaluation parameters for each single integration range for the elemental emissions at the bottom. Lower section: Screenshot of the iolite Imaging window: Image evaluation and displaying properties to the left and selected elemental mappings to the right.

Results

For each laser pulse, the laser induced plasma recorded and yields a broadband spectrum with element specific emission lines. This corresponds to the simultaneous detection of the elements present in the sample. A special advantage of LIBS compared to other elemental imaging techniques (e.g., LA-ICPMS) is the capability to measure all elements, including non-metals such as hydrogen, carbon, nitrogen, oxygen, fluorine, chlorine, and more. An exemplary spectrum including the emissions that were evaluated for this study is shown in Figure 7.

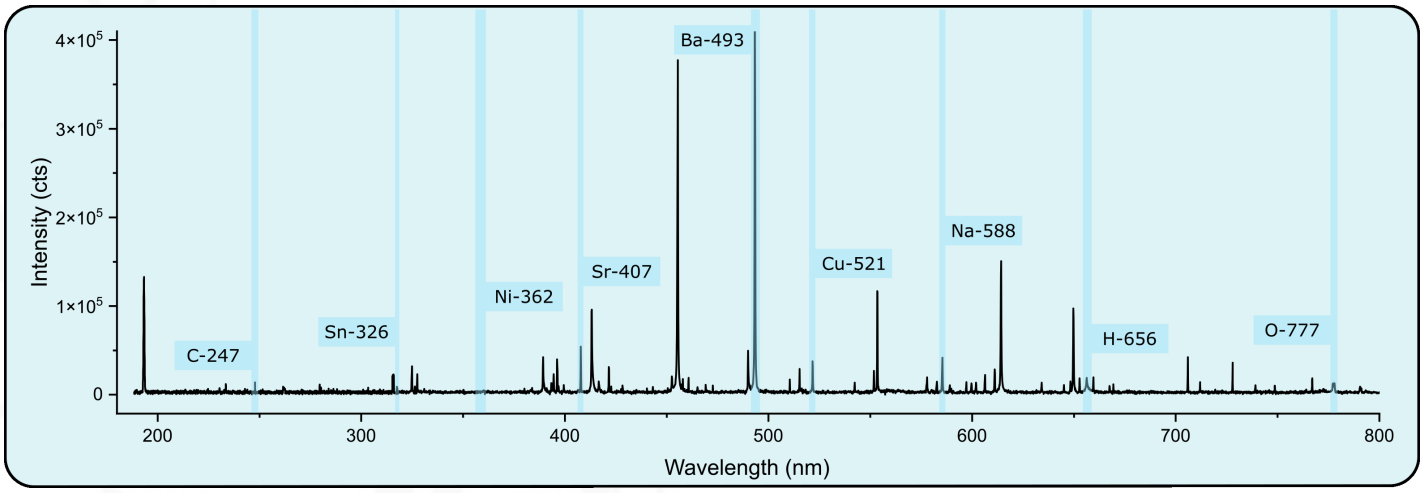

Figure 7. Accumulation of 50 broadband LIBS spectra (corresponding to 1 pixel row of a D-4 image).

A selection of the images taken in the different regions of the PCB is shown in Figure 8. Each row corresponds to a microscopic image of the sample region and the elemental mapping for one element with dosage 1 to 16 (left to right). The measurements with increasing dosages were taken on the same sample locations in sequence to another. Thus, the information in the images with different dosage levels corresponds to a different sample depth, sometimes showing different structures due to the layered structured nature of the PCB sample. As shown in Figure 1 and Table 1, the measurement of the images with an area of 4.5 mm^2 took only 32 seconds for dosage 1 resulting in 60x60 μm^2 pixel resolution, and 200 seconds for dosage 16, resulting in a pixel resolution of 15x15 μm^2.

In ROI 1 of Figure 8, the sodium elemental map shows higher intensities in the D-1 image, which was measured first. This corresponding to the uppermost layer and the higher sodium concentration is explained by the surface contamination from the sample preparation and handling. Below the surface, the Na concentration is significantly lower. With increasing dosage, the measured sample depth also increases linearly, revealing spot-wise sodium contaminations at the interfaces between the copper conducting traces and the resin composite material. ROI 2 shows nickel from the soldering pads and tin from the solders. In ROI 3, the elemental maps of copper demonstrate well the influence of different dosage levels on the image resolution, based on the appearance of horizontal, vertical lines, or diagonal progressing structures as well as circular structures. Depending on the size and shape of the structures to resolve, different dosage levels will be required. In the presented case D-4 brings significant

Results *(continued)*

improvements to D-1. However, from D-4 to D-9 and further to D-16 only marginal changes can be seen with the bare eye based on the appearance of the structures. Thus, based on the sample, choosing an adequate dosage level can bring the required improvements in image quality while saving measurement time and limiting sample ablation depth. Lastly, mappings for the non-metals hydrogen and oxygen are shown in ROI-4, displaying the locations of organic materials (resin or conformal coating) in contrast to the metal-based structures on the PCB.

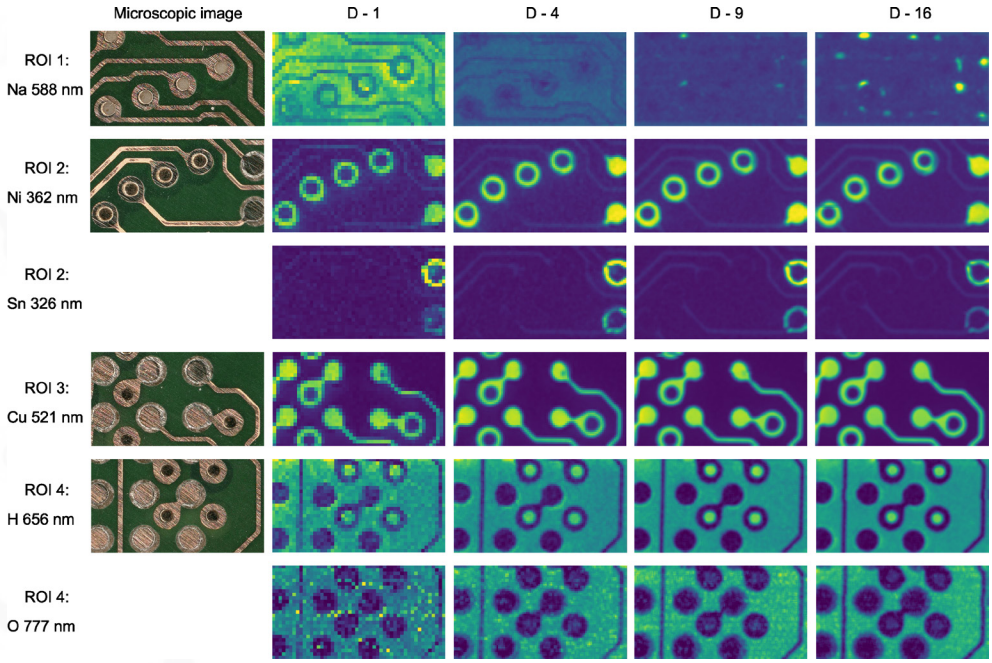

Figure 8. Selected elemental mappings on different regions (ROI 1-4) of the PCB, each measured consecutively with dosages of 1, 4, 9, and 16.

Conclusion

The imageGEO193^{LIBS} was used in combination with the ESLumen to measure elemental images of 4.5 mm^2 sized areas of a PCB, harvested from a DRAM unit. Simultaneous multielement acquisition of metals and non-metals with 200 Hz revealed the lateral and layered microstructure of the sample. The concept of dosage was demonstrated to significantly improve the image resolution from 60x60 µm^2 to 15x15 µm^2 without compromising the signal-to-noise ratio. Dosage levels from 1 to 16 were shown, resulting in measurement times from 32-200 seconds for images containing 50x25 (1250) to 200x100 (20000) pixels

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