Combined light-microscope – FIB/SEM failure analysis on automotive body parts

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Seeing beyond

Combined light-microscope – FIB/SEM failure analysis on automotive body parts

In quality inspection of painted car body parts, a variety of surface defects with different root causes may be found. To sustain a high-quality surface finish and to prevent such defects from occurring the root cause has to be investigated with the fitting method. Increasing quality in manufacturing and latest technologies in surface finishing thereby lead to defects getting smaller and occurring more rarely and thus necessitate microscopic methods to find, locate, prepare and investigate surface defects. In the present work, a correlative microscopy approach combining light-microscopy and a focused ion-beam SEM for efficient investigation in failure analysis is outlined.

By the example of in development painted bumper parts made from recycled material which shows sparsely distributed bulges in the painted surface after extensive aging tests the process of finding and locating the defects and the preparation of a cross-section to investigate to underlying structures is shown. The light-microscopic part is covered by the ZEISS Smartzoom 5 digital microscope and preparation and investigation is carried out with the ZEISS Crossbeam Laser. The correlation of both systems to precisely relocate the defects in the FIB/SEM is done with the help of the ZEISS ZEN connect software solution.

Finding the root cause of sparsely distributed and small defects on large samples for an efficient failure analysis requires a convenient workflow of locating, documenting, re-locating, preparing and investigating regions of interest. Focused ion-beam equipped scanning electron microscopes (FIB/SEM) have shown to be a versatile tool to precisely prepare cross-sections on small regions and reveal buried features that are not accessible with standard materialographic target preparation methods. In addition to the narrow field of view electron microscopes usually provide, sometimes specific regions and points of interest cannot be visualized with electron optics and are therefore easier to locate using a light microscope. To overcome this a method to locate regions of interest in a light-microscopic environment and easily and precisely re-locate these regions in the FIB/SEM is needed. The ZEN connect software solution in combination with ZEISS ZEN data storage provides exactly this (Fig. 1).

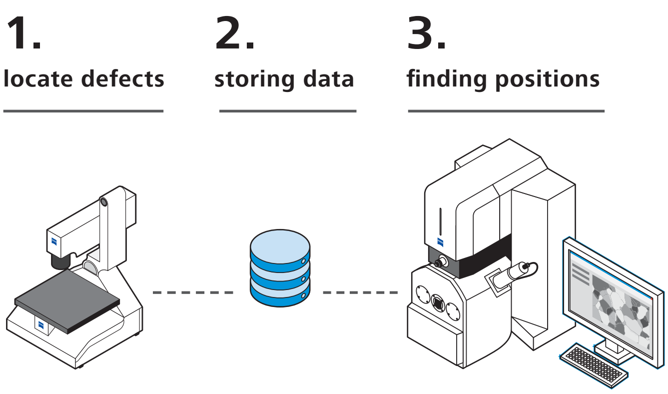

Figure 1: ZEISS connected lab, using Smartzoom 5 to locate defects, storing the data in ZEN data storage and finding the exact positions in the Crossbeam Laser.

To showcase the correlative workflow using ZEISS ZEN connect the present work shows a failure analysis on an in development painted car bumper made from recycled parts.

In the development process these parts undergo a specific aging test with varying temperature and humidity to guarantee high quality requirements or to prove the technical applicability. After such tests some parts showed several bulges in the optical inspection and the task was to find out if these peculiarities were caused by the underlying recycled material or if surface treatment and/or the painting itself was faulty.

In a first step, a cut-out section of ca. 40 x 40 mm was examined with the ZEISS Smartzoom 5 digital microscope and several regions with peculiarities were located.

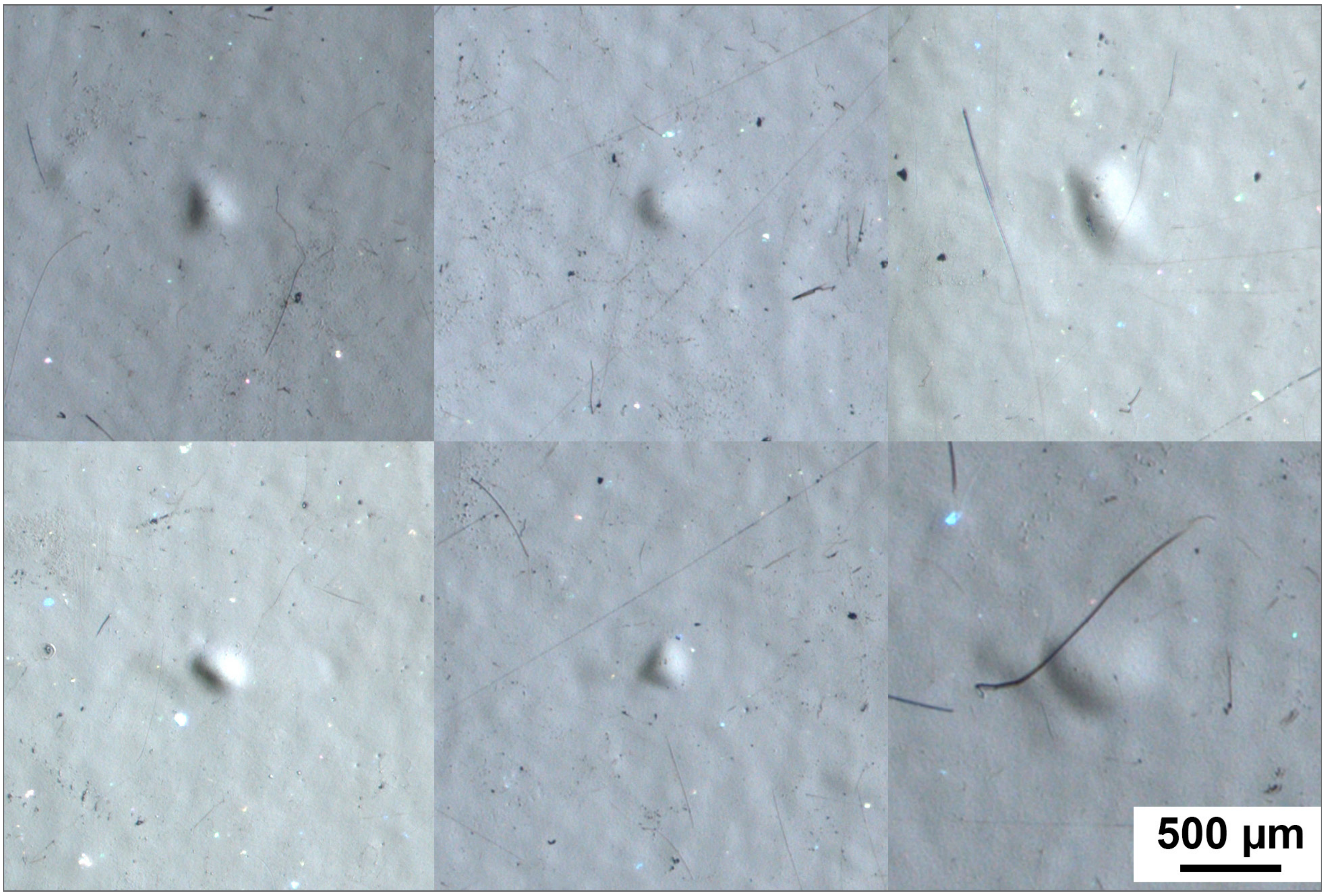

Figure 2: Set of different surface bulges found on the investigated part imaged with ZEISS Smartzoom 5.

Figure 2 shows a set of the found bulges on the painted surface after aging test with a diameter of approximate 100 µm. As ZEISS Smartzoom 5 is a fully integrated microscope, the precise location of every image on the sample is known to the software at any time. The whole set of acquired images was afterwards stored in the network accessible ZEN data storage to be re-loaded at the FIB/SEM.

After light microscopic investigation, the sample was prepared for FIB/SEM preparation and analysis via sputter coating with a conductive gold-layer and transferred to the Crossbeam Laser. The light-microscope images were then re-loaded from the ZEISS ZEN data storage onto the Crossbeam Laser and correlated via image overlay to the actual sample position in the FIB/SEM.

With a simple click the SEM-stage is then positioned on the exact position of the marked ROI and sample is ready for further preparation and/or investigation steps. As the cause of the defect was expected to be located in the recycled bumper base material, a cross-section preparation of the buried feature was necessary. Typical FIB cross-sections are prepared in the range of 10-30 µm in width and depth. Widening and deepening the cross-section is possible but will drastically increase preparation time. Therefore, to fully prepare the whole ROI of approx. 100 µm a femtosecond (fs) laser pre-preparation step was implemented.

The use of a fs-laser facilitates extremely fast material removal and surface preparation of deeply buried features as well as large areas and, due to the pseudo athermal ablation process it provides, the surface quality often reveals the true microstructure directly on the laser cut cross-section or only requires just some short FIB post-polishing.

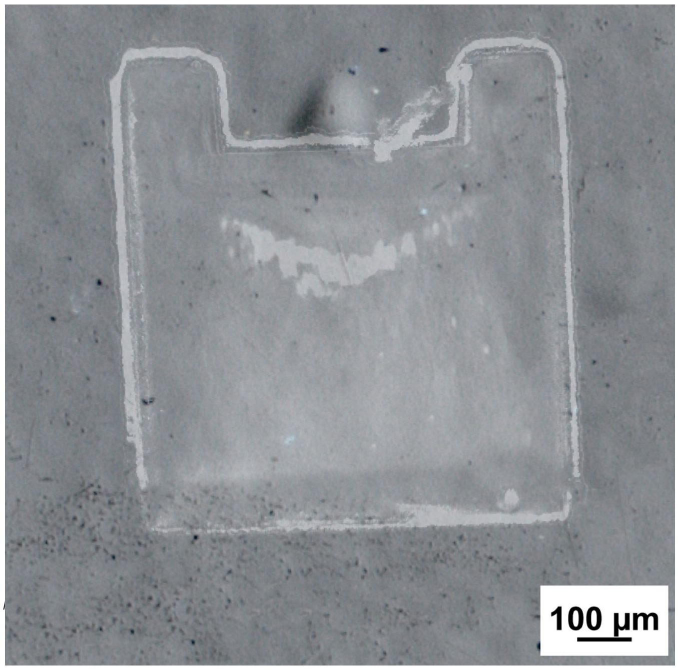

Figure 3: Overlay of laser-milled trench on lightmicroscope image of ROI.

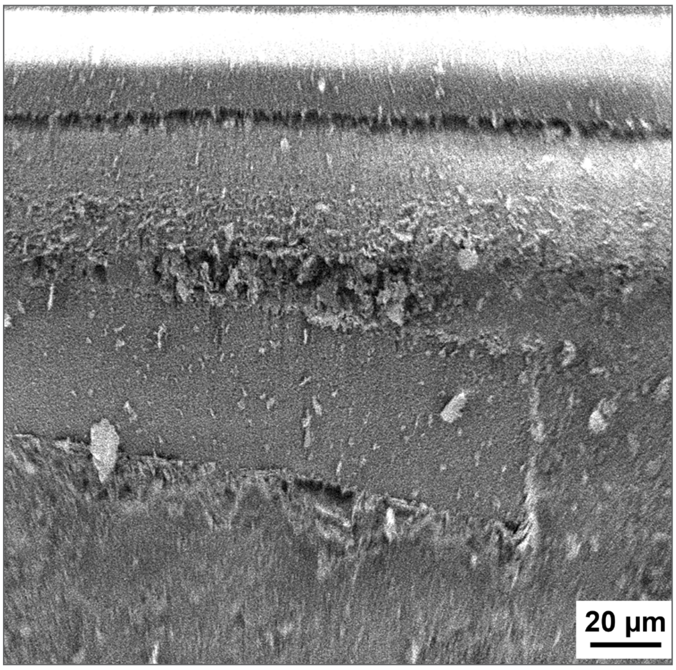

Figure 5: Suspicious feature in baser material underneath paint, laser-milled surface; SEM, SESI, 450x.

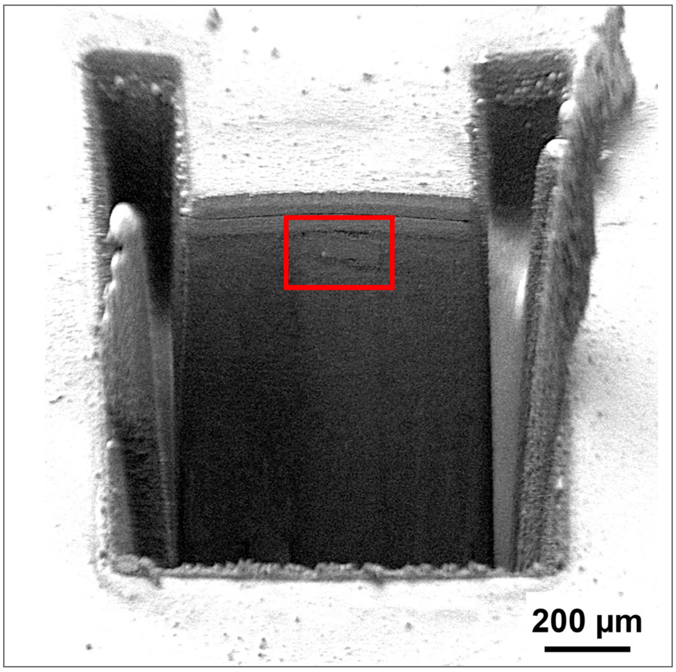

Figure 4: Laser-milled cross-section through surface defect, suspicious feature beneath paint layers visible; SEM, SESI, 50x.

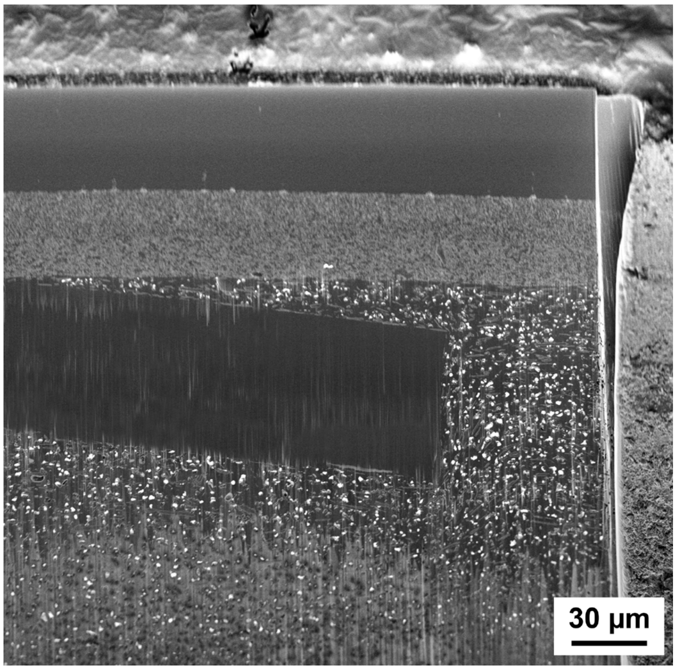

Figure 6: Suspicious feature after FIB post-polishing, good surface finish with clearly distinguishable features; SEM, InLens, 450x.

With the help of the fs-laser a large trench of ca. 500 µm width and with a depth of > 500 µm was prepared. Figure 3 shows the overlay of the fs-laser trench on the exact position of the ROI marked in the light-microscopic investigation. In Figure 4 the cross-section of the failure area in the laser-milled state is shown. Next to the good surface quality of the cross-section and the steep side walls a suspicious area can be observed right underneath the several paint layers. To find out what the suspicious area is and if it might be the cause of the surface peculiarities, a closer inspection was carried out. Figure 5 shows the suspicious area in the as-laser-milled state. Even on the roughly milled surface a rectangular particle with a different appearance right underneath the first paint layer can be seen. Figure 5 further shows some redeposited material and other debris on the cross-section surface which make further analyses and interpretation of the suspicious findings difficult. However, as the laser-milling reveals a large sample area and allows for the suspicious area to be located, a necessary FIB post-polishing

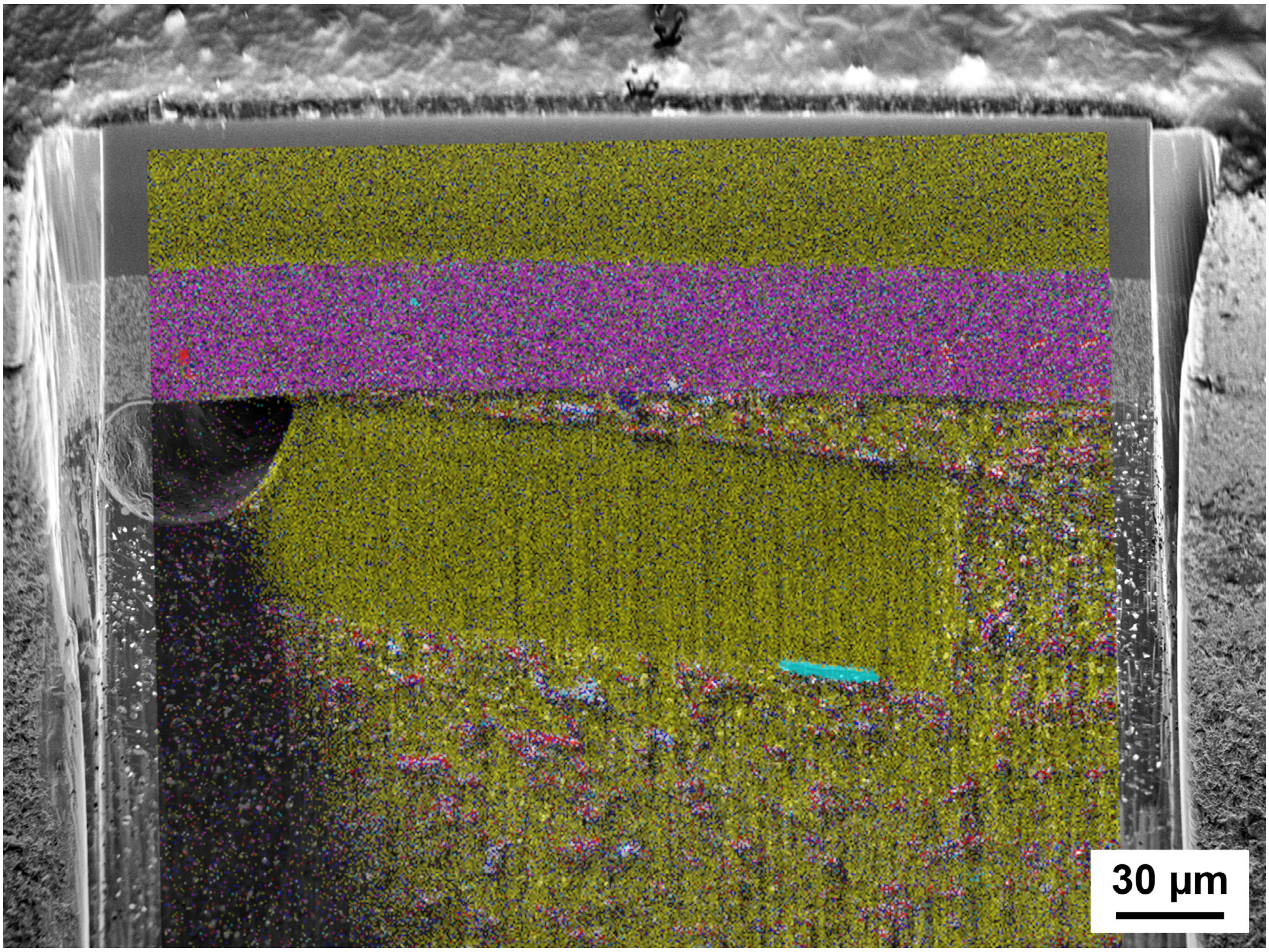

Figure 7: EDS element-mapping of FIB polished area, yellow: C intensity, blue: Al intensity, pink: Ti intensity, red: Si intensity.

can be done much more precisely and with minimal effort. In case of the suspicious feature in figure 5 a relatively wide and deep FIB cut had to be done to fully reveal the feature but as a lot of material was already removed with the laser, the FIB post-polishing time did not exceed 2 hours. The results of the FIB post-polishing are presented in figure 6 and reveal an unstructured rectangular particle in the bumper base-material underneath the first paint layer with a large pore on the interface particle/substrate. Also, the different paint layers with their inner structure as well as the filler particles in the base material can be clearly distinguished in the FIB cross-section.

To find the origin of the unstructured particle in the bumper material, an EDS elemental map was acquired on the FIB cross-section.

Figure 7 shows the intensity map of C, Ti and Si over the whole cross-section. Further an Al signal can be detected on the lower end of the foreign particle. A denser coloration here means a higher intensity of one element and less intensity of the other. The structural similarity of the foreign particle with the top-coat of the sample and the high Al-content particle on the lower end indicate that the particle is residual clear lacquer with some small residual metallic lacquer additives which was not fully removed prior to the recycling step.

Based on these findings it can be assumed that the combination of humidity and temperature might result in a formation of pores at the interface between plastic and residual lacquer particle due to weak adhesion. The resulting volume change leads to the observed local buckling of the overlying layers.

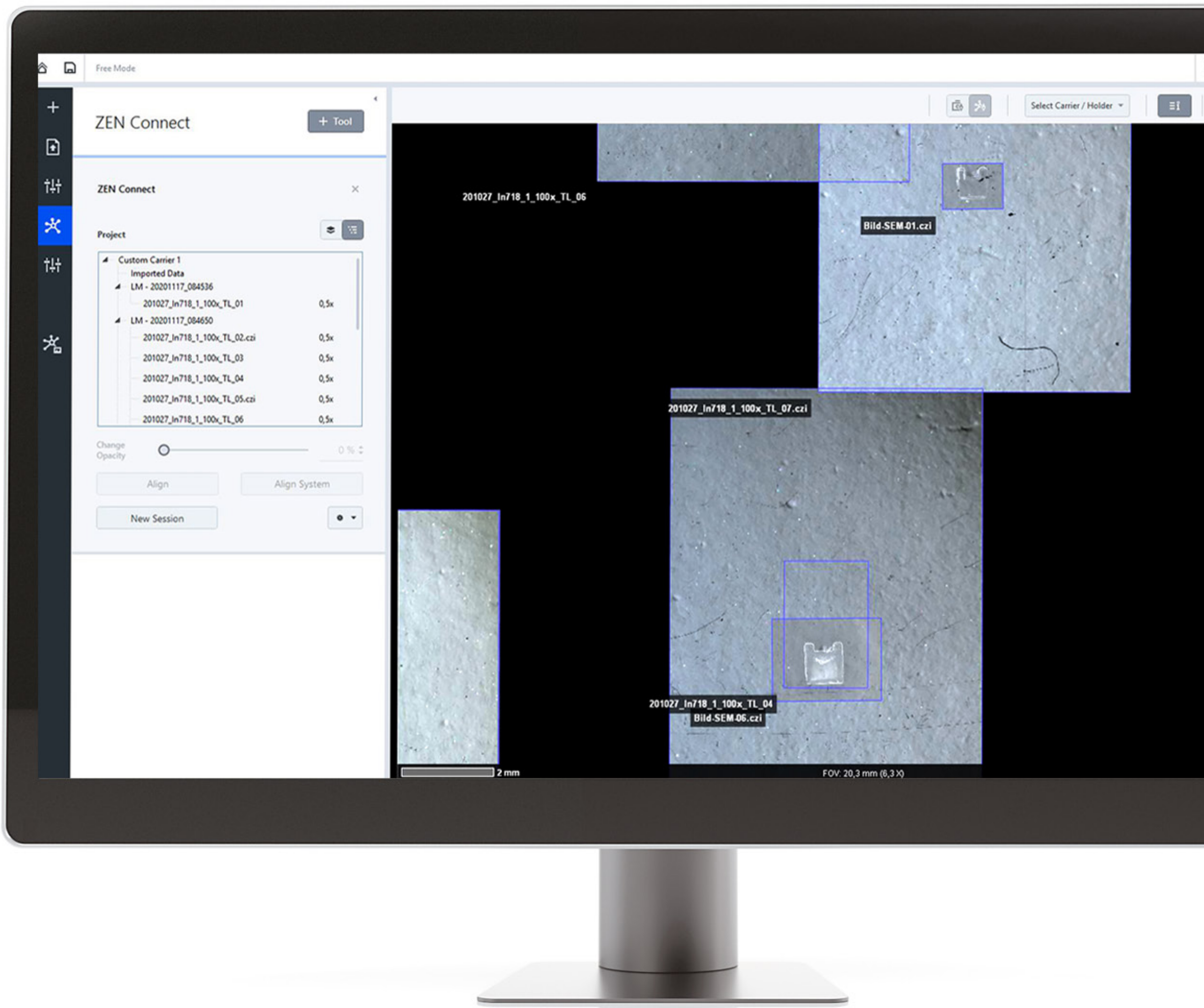

Figure 9: Overlay of laser-milled trench on lightmicroscope image of ROI; SEM, SESI, 450x.

Conclusion: The presented work shows that a correlative microscopy approach combining digital microscopes and a FIB-SEM make a convenient tool-box for failure analysis when defects are rare and sparsely distributed on large sample areas. By the example of bulges on painted car body parts it could be shown that regions of interest can be determined in the light-microscope and easily be re-located in the FIB-SEM for further preparation and investigation.

The new femtosecond laser addition for the ZEISS Crossbeam family also proved to be a convenient tool for large area location specific preparation.

The found surface defects extended the range of economic FIB-preparation but could be pre-prepared with the fs-laser in a quick and easy way.

With the help of fs-laser and FIB cross-section polishing and EDS analysis, the cause of the surface could then be determined as carbon fiber scraps. The correlative microscopy approach also makes it possible to time efficiently investigated more than one area of interest. All results are subsequently saved in a coherent project and are, thanks to the ZEISS ZEN data storage option, accessible for everyone in need for further investigation or reporting (Figure 9).

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