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CASE STUDY

Kunststoff Institut Lüdenscheid

On the track of failures

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If a process is to be improved, it is essential to be familiar with every aspect. For successful optimization, the focus of attention should, above all, be on the weaknesses of a production process, a product or a system.

Every year, the Materials and Failure Analysis department of the Kunststoff-Institut Lüdenscheid deals with over 1,000 cases of failure and the identification of material characteristics, making use of all customary methods of plastics analysis for this purpose. While the equipment for the most important procedures is directly available in-house at its laboratory, requests for specialized analysis are outsourced to an extensive network of competent and experienced partner laboratories. For customers, this means service from a single source, which centers on the coordination and interpretation of all acquired data to ensure fast and reliable identification of the cause of failures. From the first telephone call to the accredited test report, customers are offered a complete service package. This can subsequently be extended to include the definition, implementation and monitoring of quality assurance measures if required.

Why use a scanning electron microscope (SEM)/EDX?

Every day, we receive a range of enquiries about the problems of plastic components, each with a different background. Every component has its own special combination of materials and geometry, a process-dependent past and, where it originates from the field, an individual history of the loads imposed by the end customer. This case study outlines some of the examples that the Thermo Scientific™ Phenom ProX Desktop SEM allowed us to investigate without extensive preparations while also achieving savings in terms of time.

In one case, delamination on a galvanized plastic component made of the terpolymer acrylonitrile butadiene styrene (ABS) brought belts to a standstill. It was essential to identify the cause without delay. Another customer complained about the rapid failure of parts made of polyamide 6 reinforced with 30% glass fibers. A third client suspected that his non-woven synthetic fiber contained an inorganic foreign substance. One thing that all these enquiries had in common was the manner in which the solution to these problems could be found.

A stereo microscope, and also an optical microscope, allows us to push the boundaries of the feasible to clarify aspects such as pickling structures, fiber connections or the analysis of foreign substances. However, a combined system consisting of high-resolution imaging methods and spatially resolved analysis, i.e. using a scanning electron microscope and energy dispersive X-ray spectroscopy (EDX for short), can help us solve such cases.

Sample preparation

The preparation of samples is relatively simple when analysis is carried out using the Phenom ProX Desktop SEM. The sample holder is generally equipped with an aluminium plate, and the sample is fixed to its surface using conductive, double-sided adhesive tape. Alternatively, there is a practical fixing device for the polished sample holder, which features a clip. The sample itself should not exceed 25 mm in diameter and 30 mm in height. Ideally, the contact surface with the pad should be as large and as smooth as possible, and dry, too. Prior sputtering, which

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is expected with non-electrically conductive surfaces such as plastics, is only necessary for highly magnified images. Non-sputtered samples can be observed directly using a charge reduction holder.

After opening the sample chamber, the sample holder is introduced via a slide rail, and the software then automatically detects the holder type. This allows you to make preparations for measurements and to perform them quickly. The software is designed to be intuitive so that laboratory technicians are easily trained in its use. The entire process from loading the sample to the first image generally takes no more than 60 seconds.

Application examples:

Scanning electron microscope (SEM) images

The Kunststoff-Institut mainly performs examinations on plastic components, although it also deals with granulates and annealing residues. These investigations are carried out mainly for the purpose of failure analysis or to monitor the quality of components or granulates. The following images, which were produced using a backscattered electron detector in material contrast mode, show an example of the observation of filler dispersion.

Figure 1 and **Figure 2** show a plastic material reinforced with glass beads. It was not just the distribution of the glass beads that was under examination here, but also the bonding of the silicone and aluminium-based beads to the surrounding plastic matrix after several recycling processes. Good bonding is indicated by adhesion of the glass surface to the surrounding plastic. Porous structures resulting from the progressive degradation of material were also observed in the course of examination.

Another example of routine laboratory usage involves process-related component defects. **Figure 3** shows an example of cold spots, including the characteristic wrinkled surface. During injection molding, the polymer melt, which had already become semi-solid, was pressed further into shape through the application of holding pressure. As a consequence, a reduction in the load capacity of the article should be expected here. In **Figure 4**, a component shows a large number of fractures, despite being reinforced with glass fibers, due to strong glass fiber orientation with a detrimental effect. This orientation of the fibers is parallel to the stress direction and thus contributes little to the mechanical load capacity of the component. In addition, the empty, smooth-structured fiber beds demonstrate that optimum fiber-matrix bonding has not been achieved here.

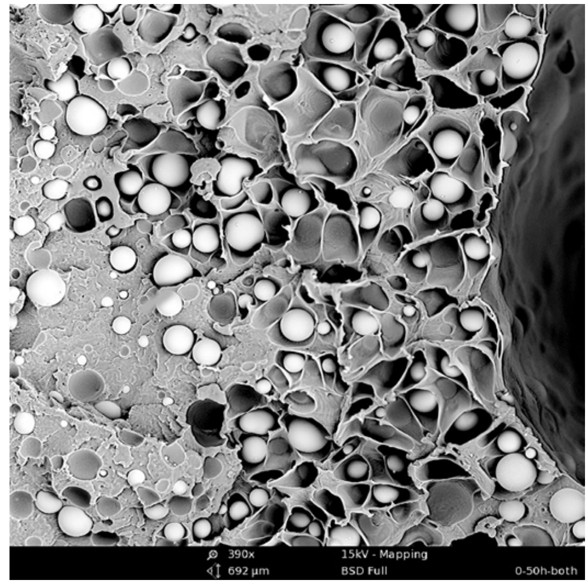

Figure 1. Distribution/size of glass beads.

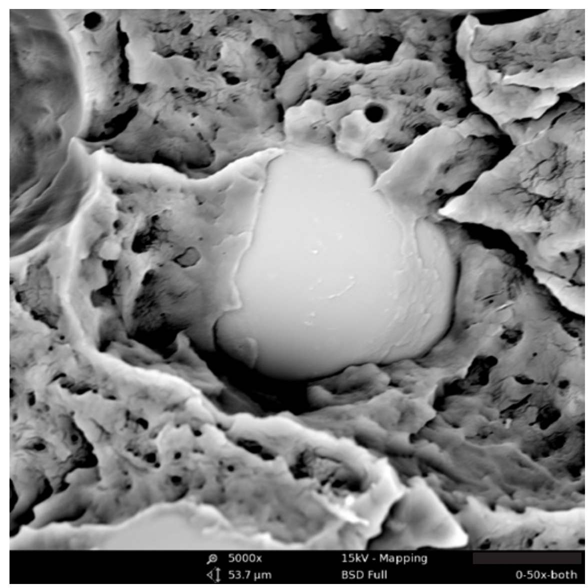

Figure 2. Bonding of glass beads.

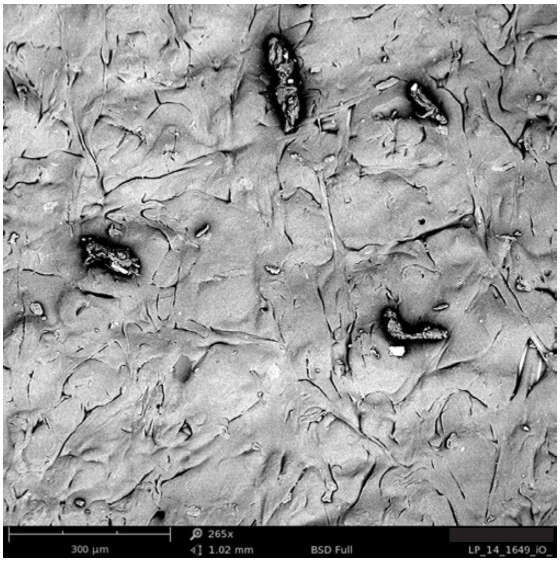

Figure 3. Cold spots in component.

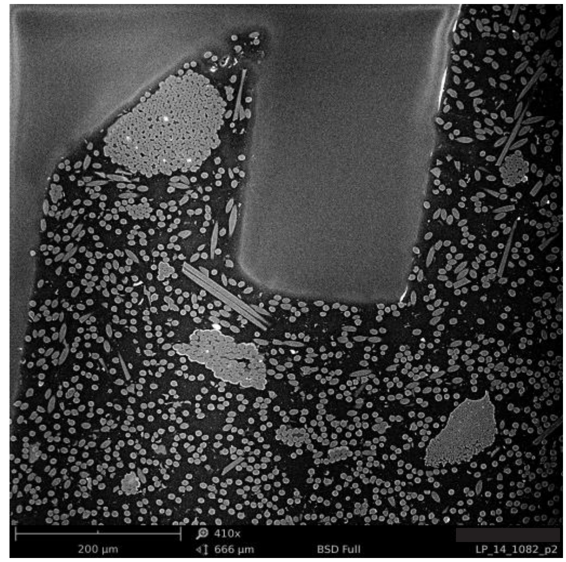

Figure 5. Fiber strands in component.

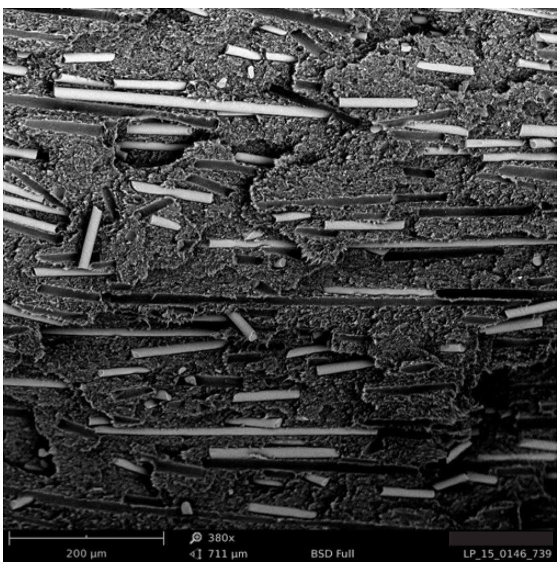

Figure 4. Strong fiber orientation in component with detrimental effect.

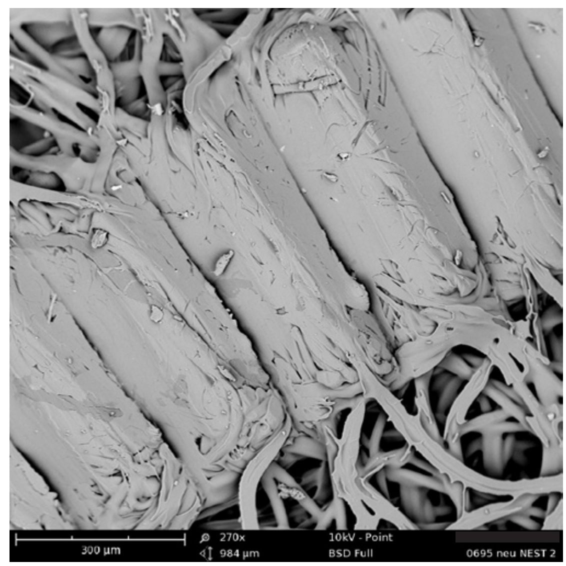

Figure 6, Evaluation of a weld seam.

Figure 5 documents an example of inhomogeneous fiber distribution. The finished component contains several fiber strands that may encourage cracking. The fiber heads can mainly be seen, so the orientation of the fibers in relation to the anticipated load can be specified in this case. **Figure 6** displays a successful weld seam in the peripheral area of a filter membrane, while gaps can be observed between the cylindrical weld packets in the reject part (not shown).

Another important application is the evaluation of pickled surfaces, which are relevant to galvanized plastic parts made of ABS or PC/ABS. An etching process is used here to extract the butadiene of the terpolymer near the surface. Homogeneously distributed, round cavities of comparable size with undercuts offer an ideal prerequisite for chemical deposition of the subsequent layers of metal in the process and their bonding to the surface. Oval and distorted cavities that differ in size, smooth zones or delamination, as shown in **Figure 8** and **Figure 9**, not only indicate that impaired adhesion of the galvanic layer can be expected, they also signify that the injection molding process could be optimized. This process stretches the molding material and subjects it to an excessive degree of shear, which may ultimately lead to various other failure patterns.

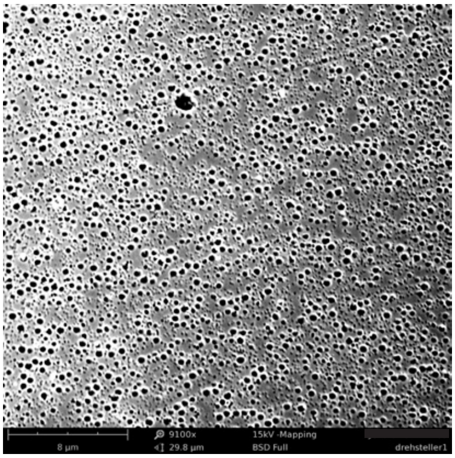

Figure 7. Good pickled finish to an ABS surface.

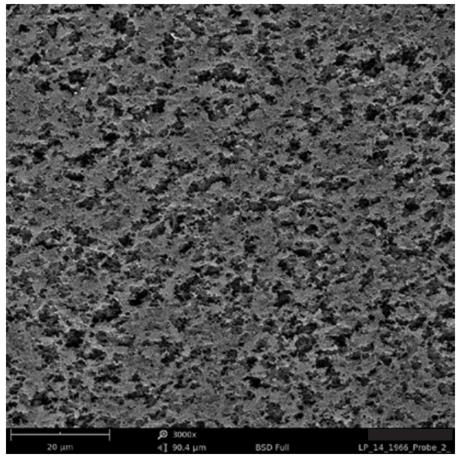

Figure 8. Suboptimal pickling of an ABS surface.

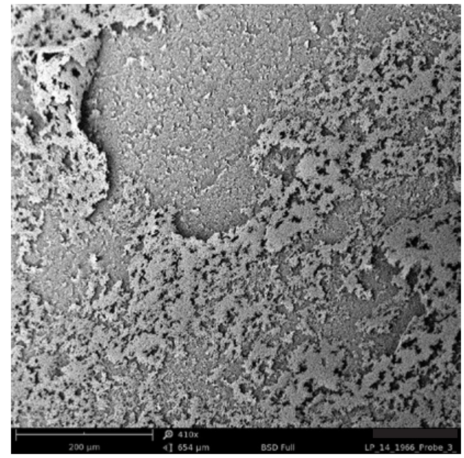

Figure 9. Delamination.

Figure 10 shows another example of a galvanized plastic part. In this cross ground micrograph, a foreign substance can be observed below the galvanic layer, which is coated with a fine layer of plastic and thus originates from the injection molding process. **Figure 11** shows a fracture surface in which it is possible to deduce the direction and origin of fracture due to the V-shaped fracture lines. In **Figure 12**, we can see a change in the type of fracture in the bottom half of the image, where the residual material bond has become brittle and spontaneously failed once cracking was initiated.

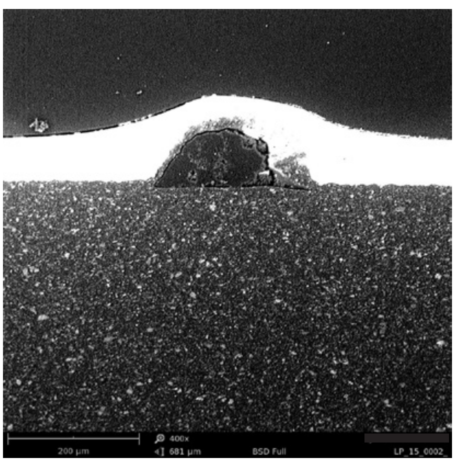

Figure 10. Foreign substance below galvanic layer.

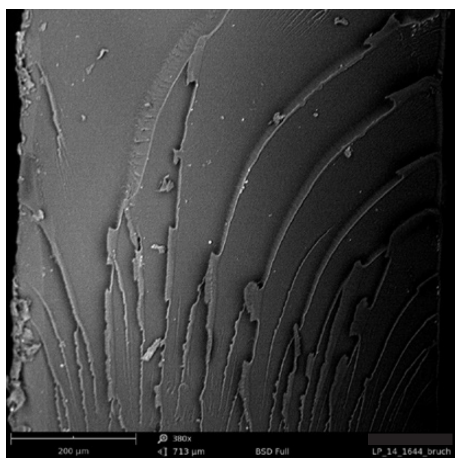

Figure 11. Fracture surface with crack lines.

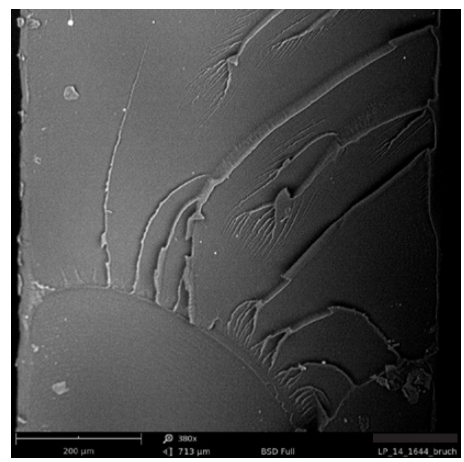

Figure 12. Diverse fracturing.

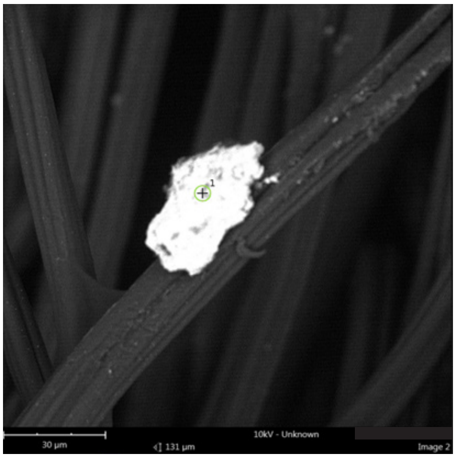

Figure 13. Foreign substance in a non-woven material.

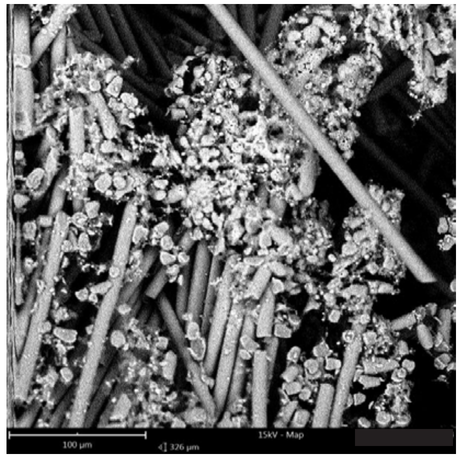

Figure 14. Annealing residue.

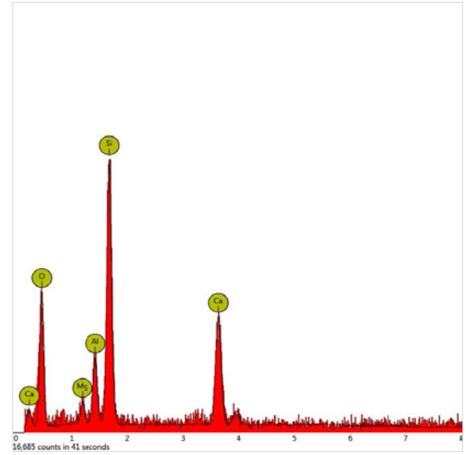

Figure 15. Elements of fillers (glass fibers and talcum).

Element identification using energy dispersive X-ray spectroscopy (EDX)

Certain problems do not just call for the high resolution offered by the scanning electron microscope, but also the precise analysis of individual elements. The Phenom ProX Desktop SEM can be used to select specific elements and identify them systematically by means of energy dispersive X-ray spectroscopy. The main purpose here is the identification of (predominantly inorganic) foreign substances, in addition to the observation of coatings and fillers and their dispersion. In **Figure 13**, a metallic foreign substance can be seen in a synthetic non-woven material. After 30 seconds, analysis revealed that this substance was rust. The results of the examination can then be saved in the form of a report with a very simple structure. In the future, it would be desirable for customers to be able to adapt the form and layout of the report so that documentation of the analyses could cater more closely to the problem at issue or correspond to the customer's CI. The report includes SEM images, indicating the areas under analysis, as well as the element spectrum and a table listing the results. An example of such a spectrum for a sample of annealing residues can be found in **Figure 14** and **Figure 15**.

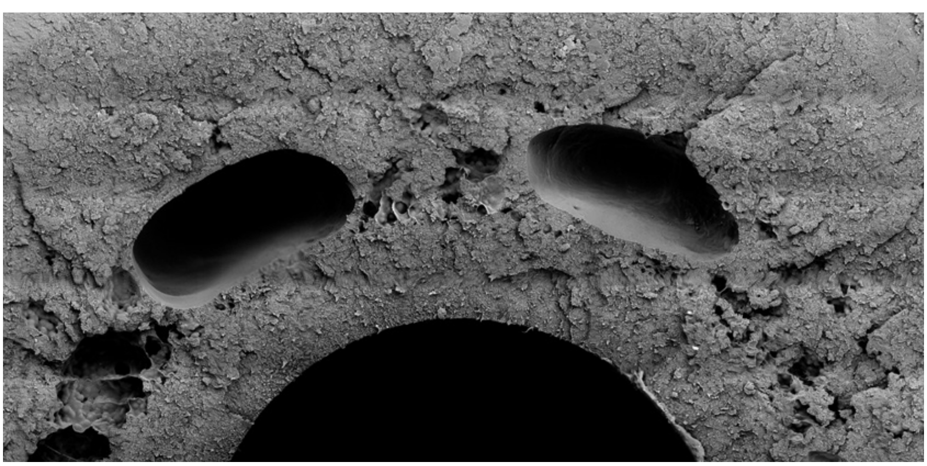
Figure 16. Cavities in the fracture surface of a cylindrical hollow part.

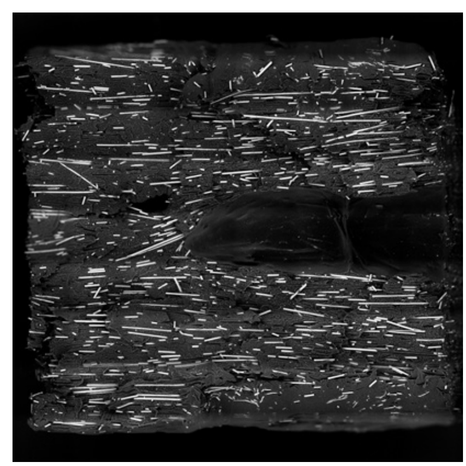
Figure 17. Image of a granule, which has been cut in half to examine distribution of the glass fibers and matrix bonding.

Automated image stitching (AIM)

A stitching function can be used to map sizeable areas of a sample by means of SEM images. This function scans a marked area and produces an overall image from a collection of individual images, albeit without any scaling.

Figure 16 shows different-sized cavities in a fracture surface that are a factor contributing to fracturing. **Figure 17** shows the overall image of a granule, which has been cut in half in a longitudinal direction. In this case, it was evaluation of the fiber distribution and fiber/matrix bond that was of interest to the customer.

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Conclusion

The Phenom ProX Desktop SEM can be used for the intuitive processing of a wide range of different problems that are conditional on the benefits offered by electron microscopy, such as its high resolution and depth of focus. Staff can be trained in its use in a very short time and avoid almost all major errors during application. The device particularly impresses with its sample introduction system. Although sample size is limited in the entry-level version, introduction is greatly accelerated. The overview and camera functions are helpful when it comes to comprehensible selection of the measuring points and allow optical images to be recorded in addition to the SEM images. You can navigate either with the mouse or via the integrated touch screen display. It is possible to adjust image quality according to the task at hand and the attributes of the samples.

The system delivers outstanding images even at high resolutions, providing that the sample is properly fixed. Another benefit is the charge reduction holder, with sputtering not being necessary per se for plastic surfaces, which do not require strong magnification. If the option of sputtering is available, even better image quality levels are possible. One accessory that can be recommended is the holder with a clamping mechanism, as alternative usage of the conductive adhesive surfaces requires the contact area to be as smooth as possible. When using conductive fixing compound, occasional movement of the field of view should be expected. Extension of the application with EDX and AIM is also simple and will deliver good results in a very short time. Minor optimization of the software in terms of reporting and the scaling of stitching images would be desirable for future usage.

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At the Kunststoff-Institut Lüdenscheid, work revolves around improving levels of quality and economic efficiency, specifically in the case of injection-moulded parts made of thermoplastic and thermoset materials and their manufacture. The company assistance with the selection, development and optimisation and realisation of products, tools and process sequences for all applications involving plastics engineering.

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