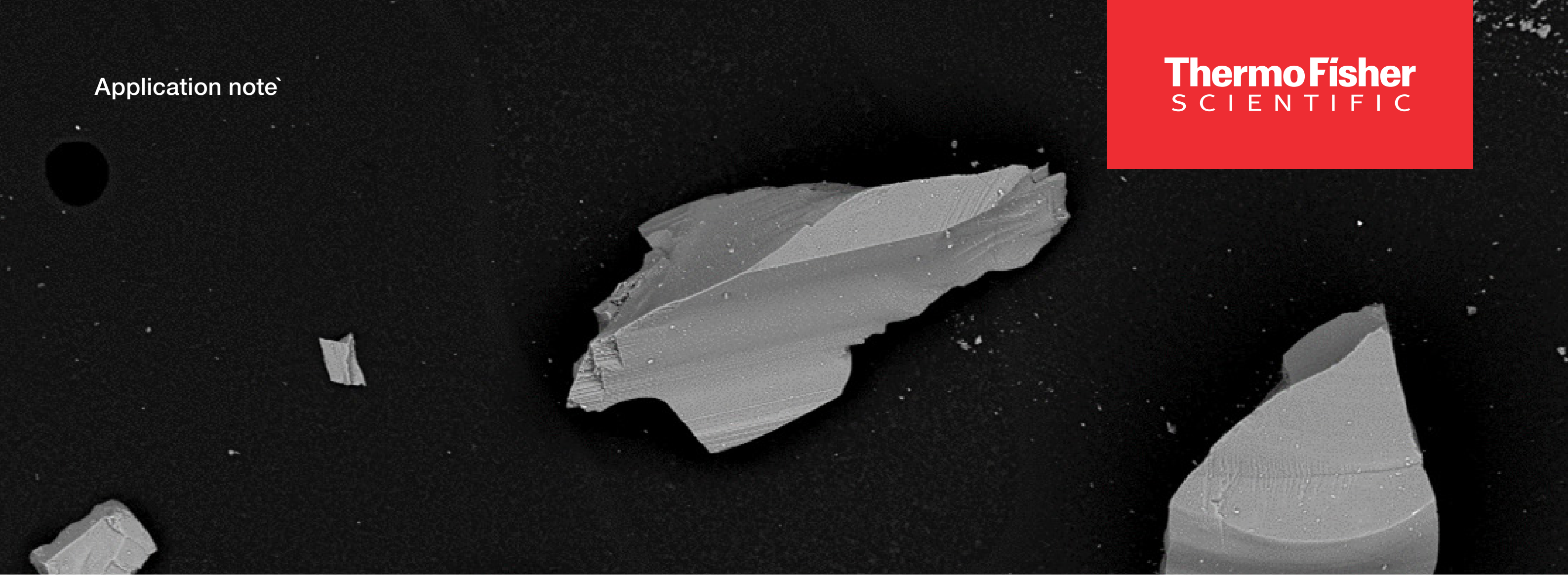
Application note`



Electron microscopy in technical cleanliness analysis

Introduction

The cleanliness of technical components plays a critical role in ensuring the longevity and performance of intricate systems across industries, from automotive and aerospace to electronics and semiconductors. Contaminants, even at minuscule levels, can have a profound impact on the functionality and durability of components. Technical cleanliness is therefore a fundamental factor influencing the quality and efficiency of products, preventing premature component wear and corrosion, as well as minimizing unexpected system failures or malfunctions.

Technical cleanliness is generally determined by analyzing particles obtained directly from components or their production environment. These particles can be collected by sampling the component surface with particle measurement cards (PMC), through the use of particles traps, or, most commonly, by washing the components. Particles removed by washing are collected from the cleaning medium with a filter and are generally micrometers to millimeters in size.

The process of collecting, measuring, and reporting on filtered particles is detailed in ISO 16232 and VDA 19.1. There are two types of microscopes described in these norms for particle analysis; optical microscopes and scanning electron microscopes (SEM). In this application note, the key differences in technical cleanliness analysis with optical microscopy and SEM will be discussed, including their methods of particle detection, their acquired data, and their relative speeds.

The process

Particles are initially collected on a flat medium or filter, which is ideal for microscopic analysis. The particles are then individually characterized, either manually or automatically, using either microscopy technique. The total number of particles can range from just a few hundred to several thousand, and can be spread across a large surface area relative to their size. It is therefore typical to use an automated process to perform the analysis; a brief summary is shown in Figure 1.

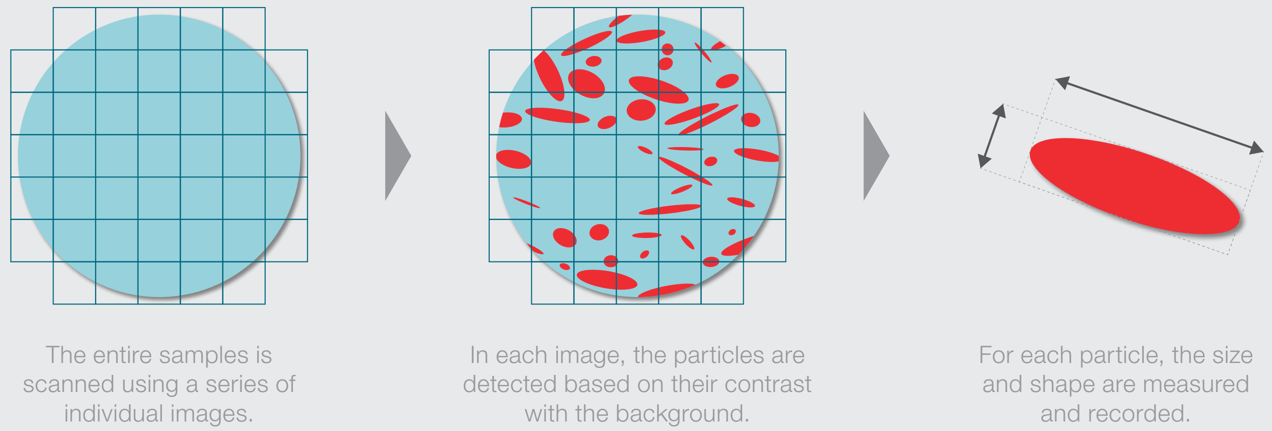


Figure 1. Summary of a typical particle analysis workflow.

Particle detection

Particle detection in digital images is typically performed using a contrast threshold, so particles must stand out from the background to be detected. For optical microscopes, this means that the particles need to have a different color then the background. For this reason, particles are often captured on a white filter, allowing materials such as carbon contaminants, biological matter, and metal particles to stand out. On the other hand, this makes light-colored (e.g., TiO_2) or transparent (e.g., glass) particles difficult to detect, as they lack contrast with the background.

Backscattered electrons (BSE) are used to detect particles in SEM. In a BSE image, contrast depends on the average atomic number of the material being imaged. The higher the atomic number, the more electrons are reflected, which translates into more signal and thus a brighter image.

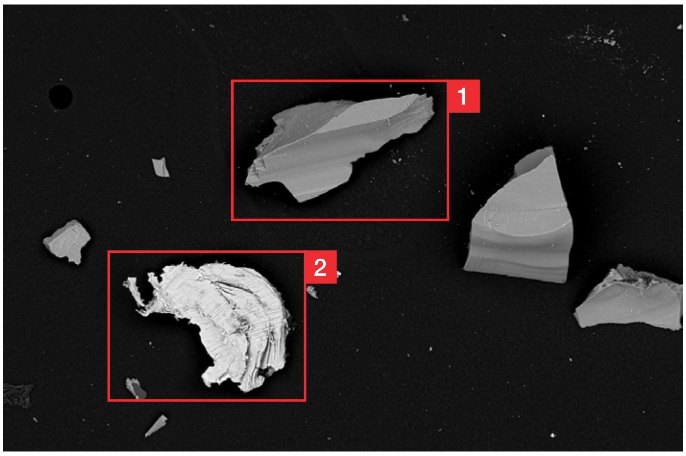


Figure 2. Al_2O_3 (1) and a pure aluminum particle (2) on a carbon background. The difference in contrast can clearly be seen.

Filters used for BSE imaging are commonly carbon-based, so particles made of materials heavier than carbon, such as TiO_2 or glass, can be easily visualized using the BSE detector. Carbon-based particles, meanwhile, are more difficult to detect and a different filter would have to be used, such as a silicon or gold-coated filter.

The data

Particle size is the key parameter for the ISO 16232 and VDA 19.1 norms; there can also be specifications regarding the number of particles that can appear in each size class.

The norms indicate that 5 µm is the smallest particle size that must be detectable. To be able to accurately measure a particle's size, it must be captured with enough pixels in the digital image to identify its shape. As a result, for small particles, the pixel size of an optical microscope is often insufficient for reliable size and shape measurement.

Electron microscopes can achieve much smaller pixel sizes, in the nanometer range, so sub-micron particles can easily be detected and analyzed.

The added benefit of SEM is that in can also quantify the elemental composition of the particles using an energy-dispersive X-ray spectroscopy (EDS) detector, which analyzes the X-rays emitted by the particle when impacted by the high-energy electron beam. This allows particles to be classified by both their size and composition. This can help to identify the source of contaminants, or more accurately classify detrimental materials like corundum (Al_2O_3) and silicon carbide (SiC). Whether a part passes or fails a cleanliness test is based on the amount, size, and class of the particles found on the filter. Being certain of the particle composition allows for more reliable product quality assessment and makes it more effective to fight the source of the contamination.

Speed

Both microscopes analyze the surface of a filter field by field, with a magnification and pixel size based on the size of the particles that need to be detected. The smaller the particles, the higher the magnification, the smaller the field size, and consequently the more images must be taken to scan the entire filter. The overall speed of the analysis is therefore heavily reliant on the minimum size of the particles that need to be detected.

Optical microscopes are generally faster than SEMs due to their larger field of view and faster image acquisition speed. An optical microscope can analyze an entire filter in roughly 5 to 10 minutes.

As stated previously, EDS offers valuable chemical characterization that optical microscopes cannot provide. For this reason, several manufacturers offer a correlative workflow that links optical and SEM analysis, combining the speed of optical microscopy with the chemical analysis of SEM-EDS. A lower magnification scan is first performed with the optical microscope and particles of interest are identified based on size. The sample is then transferred to an SEM, where the particles are re-located and analyzed with the EDS detector.

These additional steps of sample transfer, realignment, and data correlation greatly undercut the initial speed of optical microscope analysis. With sufficiently advanced SEM-EDS instrumentation, both scanning and EDS analysis can instead be performed in a single instrument. The Thermo Scientific™ Axia™ ChemiSEM with Perception Software, for instance, enables automated and fully integrated SEM-EDS particle analysis of up to 4 filters in one run. As the Axia ChemiSEM has a very large field of view, the resulting minimum magnification is 20x. Along with its high pixel resolution, this allows complete filters to be scanned, and the particles to be measured, within 20 minutes. The addition of EDS analysis adds about 0.1–1.0 seconds per particle to the analysis time.

With SEM, detected particles can be filtered based on their size or shape, just like with an optical microscope. Selected particles of interest (e.g., the 10 largest particles) can then be analyzed with EDS without any additional correlation steps. This saves time and increases accuracy, as no sample transfers or alignment is needed.

To determine whether a specific part meets the required specifications, Perception Software generates customized reports in compliance with ISO 16232 and VDA 19.1 standards. These reports facilitate easy interpretation of results, tailored to the specific needs of your industry, thereby ensuring informed decision-making and adherence to cleanliness standards.

Conclusions

Electron microscopes are highly valuable tools for particle analysis in technical cleanliness. They are capable of detecting a wide range of particles, including those smaller than 5 µm, surpassing the limitations of optical microscopy. Backscattered electron detection, based on atomic-number contrast, ensures efficient identification of various materials, including particles that are difficult to differentiate with optical microscopy like titanium dioxide or transparent/glass-based contaminants.

EDS analysis in the SEM adds elemental composition to particle size measurements, potentially identifying specific detrimental particle types that can be targeted in order to improve final product quality.

The choice between optical and electron microscopes ultimately depends on your specific analytical needs. If size is the only necessary parameter and throughput is the primary concern, optical microscopy can be sufficient. For more advanced characterization, electron microscopy provides high-resolution imaging with elemental identification, ensuring meticulous technical cleanliness and elevating the reliability and quality of manufactured parts.

Future

Specifications for technical cleanliness are continuously becoming more stringent, with increased demand for higher-resolution data on even smaller particles. For some areas, such as automotive and high-precision applications, tolerances can now be below the micrometer scale, meaning that sub-micrometer particles can have detrimental effects on the function and lifetime of parts. Scanning electron microscopy is already capable of routinely obtaining sub-micrometer details, which ensures its ability to support technical cleanliness workflows well into the future.

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