Automated SEM-EDS enhances surface cleanliness management in cleanroom manufacturing

Introduction

Cleanrooms and associated controlled environments are critical across various industries, including pharmaceuticals, food, semiconductors, and microelectronics. In order to meet the high standards for contamination-sensitive production, cleanrooms are designed to minimize the introduction, generation, and retention of particulate contamination. Identifying sources of particle contamination is therefore an essential part of maintaining cleanliness. Common sources include human activities, equipment operation, and material handling, all of which have the potential to further disperse particles as well.

These potential sources of contamination significantly impact cleanroom manufacturing performance and must be monitored and controlled within certain limits. The level of airborne particles is a particularly critical metric, and is widely used as a cleanroom classification standard and to establish grading level. Additionally, the deposition rate of airborne particles impacts the surface cleanliness of vulnerable parts, and the effectiveness of cleaning processes for these parts needs to be monitored and validated.

Monitoring and validation of surface cleanliness

The International Organization for Standards offers a practical approach for cleanliness monitoring in cleanroom manufacturing through its updated ISO 14644 standards. Beyond just general particle concentrations, recent updates, such as ISO 14644-8:2022 and ISO 14644-10:2022, have highlighted a growing emphasis on chemical contamination, specifically addressing air chemical cleanliness and surface contamination by chemical compounds. Understanding the composition of contaminants is therefore becoming increasingly important. Additionally, there is an increased focus on nanoparticles, particularly those below 100 nm, as indicated in ISO 14644-8:2022 and ISO 14644-12:2018. Future revisions of ISO 14644-14:2016 could expand this size range below 100 nm as standards align with the evolving requirements of modern industries (i.e., nanotechnology) and the need for more precise contamination control measures.

A thorough understanding of contamination is essential to meet these stringent surface cleanliness protocols for cleanroom manufacturing. Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS) is a powerful solution that, compared to traditional methods, can detect smaller particles with high precision while also providing detailed chemical information. This advanced technique offers a comprehensive approach for the identification and analysis of surface contaminants, ensuring that cleanliness standards are met with greater accuracy and reliability.

Automated SEM-EDS monitoring of surface cleanliness

1. **Sampling approaches** Traditional sampling methods, such as adhesive stamps, risk secondary contamination or damage to sensitive parts, which is why alternative sampling methods are often necessary. Novel samplers, such as particle measurement cards (PMCs), avoid leaving residue and provide direct, reliable feedback on surface cleanliness, but may come with higher costs.

   Air samplers, placed near the production line, offer a more cost-effective alternative for the collection of airborne particles, and are available in several different types including impaction, filtration, and gravitational. Of these, gravitational samplers, such as particle traps, are particularly useful because they mimic the conditions under which the parts are being processed. They consist of a substrate coated with a sticky substance that improves capture efficiency.

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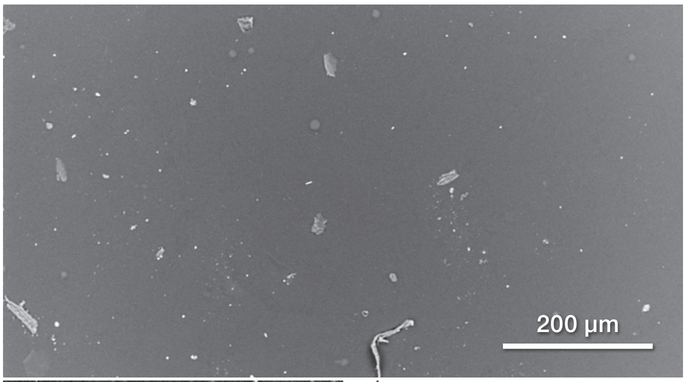
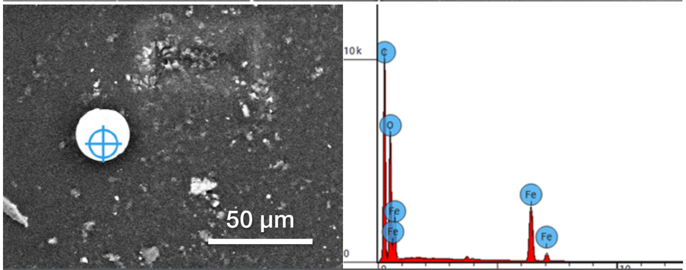
Figure 1. Sampling for SEM-EDS analysis, conducted with a PMC (left) and a particle trap (right).

These two sampling approaches can provide a complete and complementary set of inspection and monitoring procedures for cleanrooms. PMCs can be used to detect the current surface cleanliness of the product, which helps to verify the contaminants that a specific process may generate, or validate if a cleaning method meets standards. Particle traps are usually placed at strategic points for long-term contaminant monitoring at different locations throughout the cleanroom, and they are used to confirm whether the contaminants come directly from the cleanroom environment. For example, if large quantities of a specific element are detected with PMC sampling, but the particle trap indicates that concentrations of this element are low in the cleanroom, it can be deduced that the contaminants may come from a closed process (e.g., equipment friction or reaction by-products).

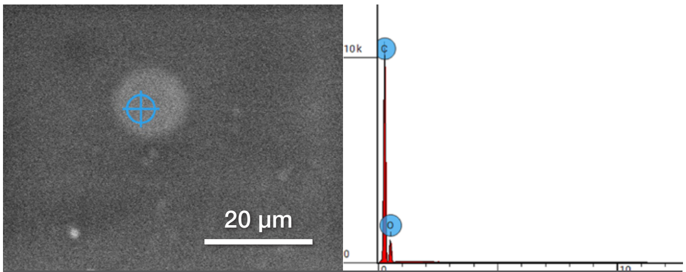
2. **SEM-EDS analysis** SEM-EDS provides the structural and chemical information needed to accurately characterize a wide range of contaminant particles. To demonstrate this, a particle trap was placed in a manufacturing cleanroom of Thermo Fisher Scientific (De Schakel 2, 5651 GG Eindhoven, Netherlands) for 192 hours under operational conditions. Collected particles were identified and characterized with a Thermo Scientific™ Phenom™ ParticleX Desktop SEM (Figure 2). The shape, size, and roughness of the particles can be directly visualized and measured from the SEM images. The corresponding compositional information (i.e., element concentration), shown in the EDS spectrum, is crucial for understanding the nature of the particles. (It is worth noting that a significant contribution of carbon and oxygen can be expected from the substrate itself.) Based on the SEM-EDS data, contaminants can be categorized as metallic or organic particles, minerals, microplastics, etc.





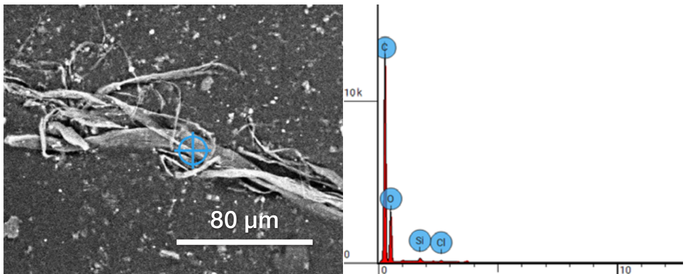
Metallic (Iron-rich)

C	O	Fe
56.6%	30.5%	12.9%



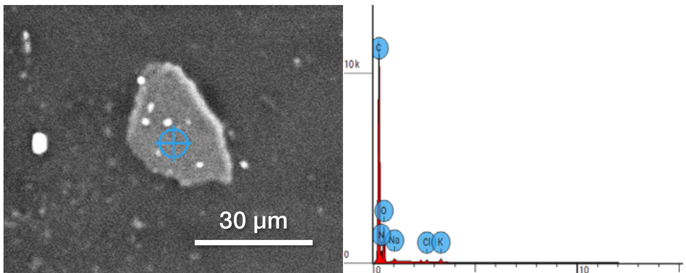
Organics

C	O
77.1%	22.9%



Microplastics

C	O	Si	Cl
66.8%	32.7%	0.3%	0.2%



Minerals

C	O	N	Cl	K	Na
60.8%	26.4%	11.6%	0.7%	0.4%	0.2%

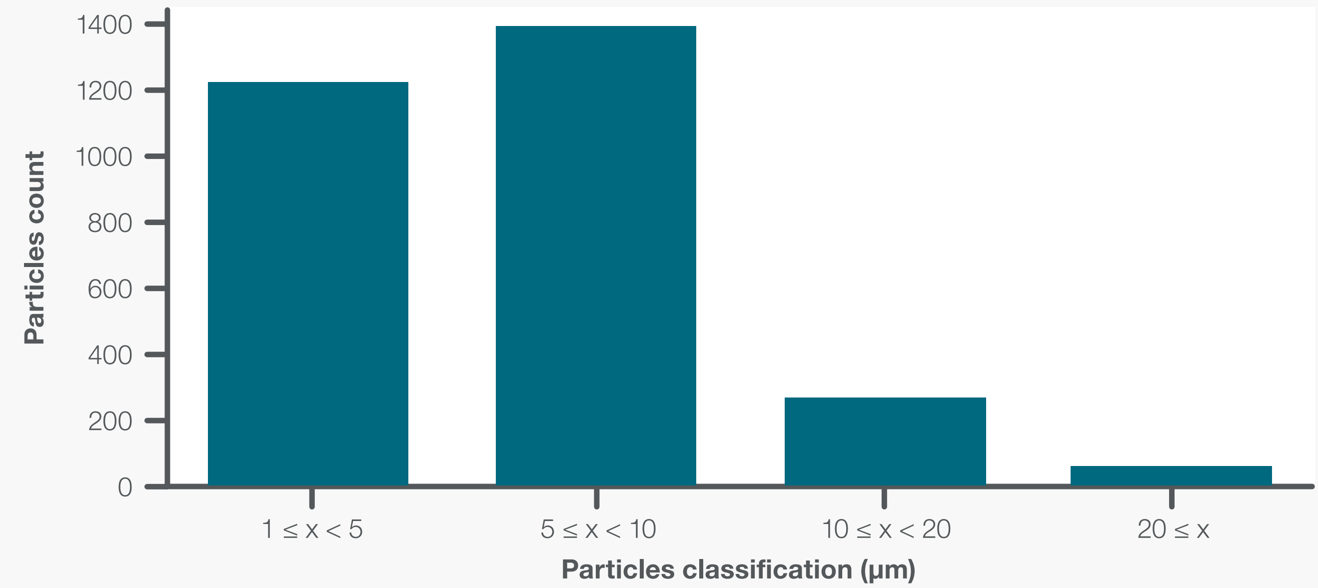
Figure 2. Overview of several particles found on a particle trap that was left in a Thermo Fisher Scientific cleanroom for 192 hours. Based on their shape and compositional information, these particles were classified as metallic particles, microplastics, organics, or minerals.

Customized automated workflow with Perception Software

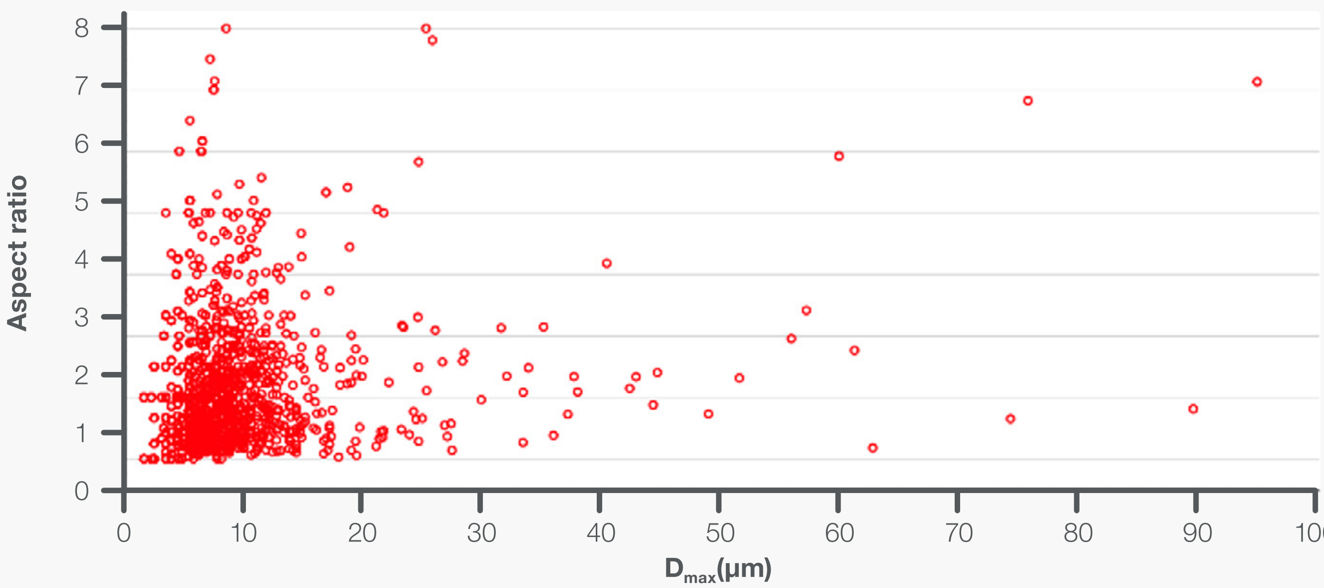
Continuous monitoring of cleanroom conditions is greatly facilitated by the automation of repetitive SEM-EDS data acquisition and analysis tasks. Thermo Scientific Perception Particle Analysis Software provides such automated image acquisition and processing with customizable statistical analysis. In Figure 3, Perception Software was used to quantify the particle size, aspect ratio, and chemical information for a PMC that was used to check the surface cleanliness of a manufactured part, produced in the same Thermo Fisher Scientific cleanroom.

While these measurements were performed on a Phenom ParticleX Desktop SEM, Perception Software can also be used with Thermo Scientific™ Axia™ ChemiSEM™ and Apreo ChemiSEM Systems.

The results in Figure 3 indicate that most particulate contaminants are smaller than 10 micrometers. The higher copper content suggests the presence of contamination sources early in the manufacturing process. When combined with the relatively high levels of other metals (such as iron and aluminum), it can be assumed that these contaminants likely originate from equipment wear, or as by-products during manufacturing.

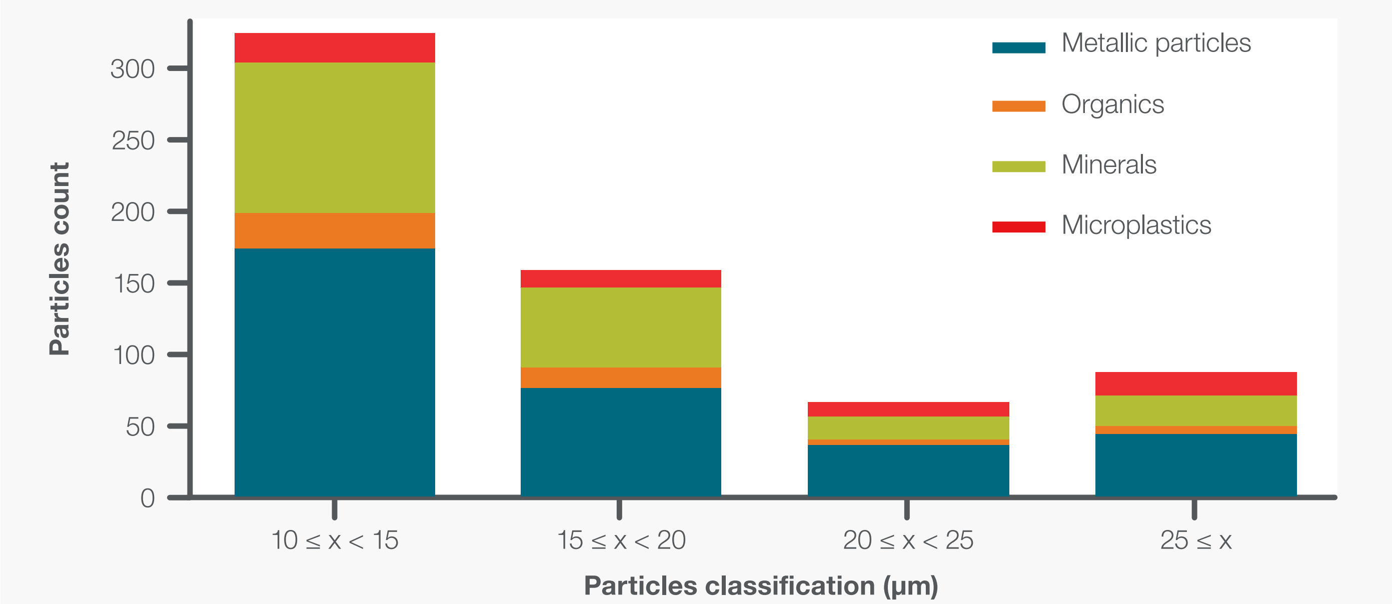


Size distribution (x=D_{max})					
Size range (µm)	Total	1 ≤ x < 5	5 ≤ x < 10	10 ≤ X < 20	x ≥ 20
Total counts	2935	1221	1392	265	57



The element concentration (exclude C and O)														
Average chemical composition reported in %														
Cu	N	Si	Fe	F	Al	Zn	Ni	K	Na	Cl	Mg	Cr	Mn	S
68.9	10.7	4.4	3.5	2.5	2.3	1.6	1.3	1.1	1.0	0.8	0.6	0.6	0.5	0.2

Figure 3. Statistical analysis of a PMC sample with Perception Particle Analysis Software. To better assess inorganic contaminants and eliminate the influence of the PMC itself, carbon and oxygen were excluded from the compositional statistics. Size distribution (using the maximum diameter, D_{max}), elements found on the sample (excluding C and O), and the aspect ratio (calculated by dividing the maximum diameter of a measured particle by the minimum) are shown.



The average compositional information of each class													
Average chemical composition reported in %													
Classification	**Particles**	**C**	**N**	**O**	**F**	**Na**	**Mg**	**Al**	**Si**	**Cl**	**K**	**Ca**	**Fe**
Metallic particles	335	50.2	10.7	34.5	0.1	0.2	0.2	0.5		0.1	2.1	1.1	0.3
Minerals	201	52.4	8.5	33.8	0.1	0.6	0.1	0.9	3.0		0.1	0.4	0.1
Microplastics	56	55.1	8.6	35.9	0.1				0.1			0.2	
Organics	47	55.9	8.8	35.3									

Figure 4. A simple classification of contaminant particles based on shape and composition (including carbon and oxygen). The compositional information of each class was averaged.

Cleanroom environments often contain multiple sources of contamination, many with high carbon and oxygen content (usually more than 80% in total), which makes them challenging to distinguish based on chemical composition alone. A qualitative analysis utilizing particle shape can therefore be necessary for differentiation. In particular, aspect ratio (D_{max}/D_{min}) is a simple and practical way to represent particle shape, where a larger value indicates a more oblong particle. Utilizing this shape information, microplastics (with a large aspect ratio) can be easily differentiated from organics or minerals (with a smaller aspect ratio) despite having similar chemical composition. It is important to note that this is still a qualitative assessment and there are no strict categories for microplastic aspect ratio; microplastic particles with a small ratio may exist depending on the analyzed part or application.

Figure 4 shows a simple classification of particle shape and composition with Perception Software from the same particle trap as was shown in Figure 2. The histograms generated with this approach can help answer critical questions, such as whether smaller metallic particles have a greater impact on surface cleanliness.

No universal classification and source identification can be applied, however, due to the diversity of cleanrooms. For instance, in semiconductor cleanrooms, organic particles are more likely to originate from personnel activities, whereas in biomedical cleanrooms, organic contaminants are more likely to come directly from raw materials.

Conclusions

Thermo Scientific SEMs with integrated, automated Perception Software provide a reliable, easy, and comprehensive way to analyze surface cleanliness. This approach can be used to analyze both overall cleanroom performance and for critical points monitoring, as well as to validate the effectiveness of cleaning processes. Analytical results provide valuable insight into potential contamination sources, informing the development of further control measures. Due to the various requirements of different products and cleanroom manufacturing processes, the flexibility to customize the final report can allow you to systematically address the risks related to your specific surface cleanliness needs. This will help you make protocols and controls that enhance operational efficiency, reduce product defects, and ensure compliance with regulatory requirements.

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