Wear Debris Analysis:
The Power of Knowing More using Automated SEM/EDX

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For years, wear debris analysis has been at the forefront of predictive and preventative maintenance monitoring; ranging from applications on simple devices to extremely complex equipment and engines. At the heart, lubricant analysis has provided a wealth of knowledge to the industry – paving the way for breakthroughs in technology and a global expansion in oil analysis business. As a result, the industry thrives on having the ability to assess the changing physical and/or chemical attributes of debris as leading indicators for the amount, mode, type, and trend of degradation.

The most common techniques for assessing wear are still spectroscopic methods where the bulk chemical presence of the wear particulate is analyzed. Spectroscopic methods provide an overall assessment of the metals in the oil, while falling short on providing more detailed particle information. In contrast, oil particulate counting methods also exist. These flow counting methods rely on optical means to determine the size and shape of particulates in a flowing stream of sampled oil. The particles are enumerated and can provide some detail on the wear mechanism; however they do not provide any indication as the material source and cannot be effectively correlated with the information gained by spectroscopy.

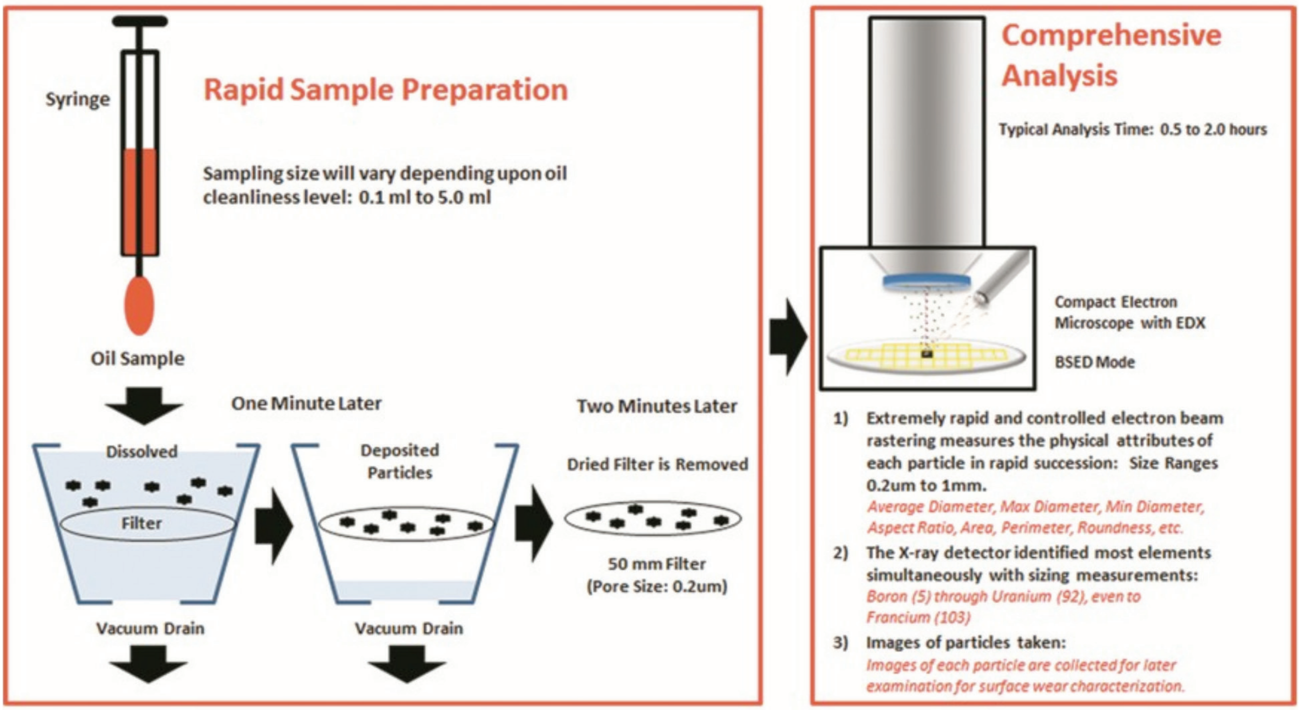
Why are both elemental analysis and particle enumeration important? Through knowledge of the elemental composition of steels, lubricants, and coating materials present within the wear environment, one can better evaluate and understand from what internal parts the various particles are being generated, confidently leading one on course with the best available diagnostic data. Having the ability to monitor such quantities over time can guide insight on the rapidity of wear, how and when to intervene, and through better documentable findings, pinpoint the changes in question to better evaluate part replacement, overall engine, gearbox, or turbine health, and the assumptions to liability. How does industry make this possible, one might ask? Well, engineers have to evaluate all the possible wear debris information that exists within an oil sample. So rather than taking an average chemistry sample, one must eventually consider going to the particles themselves and evaluating them individually. For years, scanning electron microscopes (SEM) have been utilized in lab settings to evaluate particles but, they simply take way too much time to use and often are too difficult to operate by untrained professionals. However, recent advances in the last decade have yielded more robust and automated SEM systems that are integrated with energy dispersive X-ray (EDX) spectrometers (Figure 1). These have generated a growing interest due to their speed (upwards of 20,000 particles per hour) and unattended operation. In addition, the wealth of knowledge and accuracy of data that is produced has proven to be invaluable for the diagnostic engineer.

↑ **Figure 1.** An example of a portable SEM/EDX system used for aircraft engine monitoring and Go/No Go flight status confirmation.

What makes the difference?

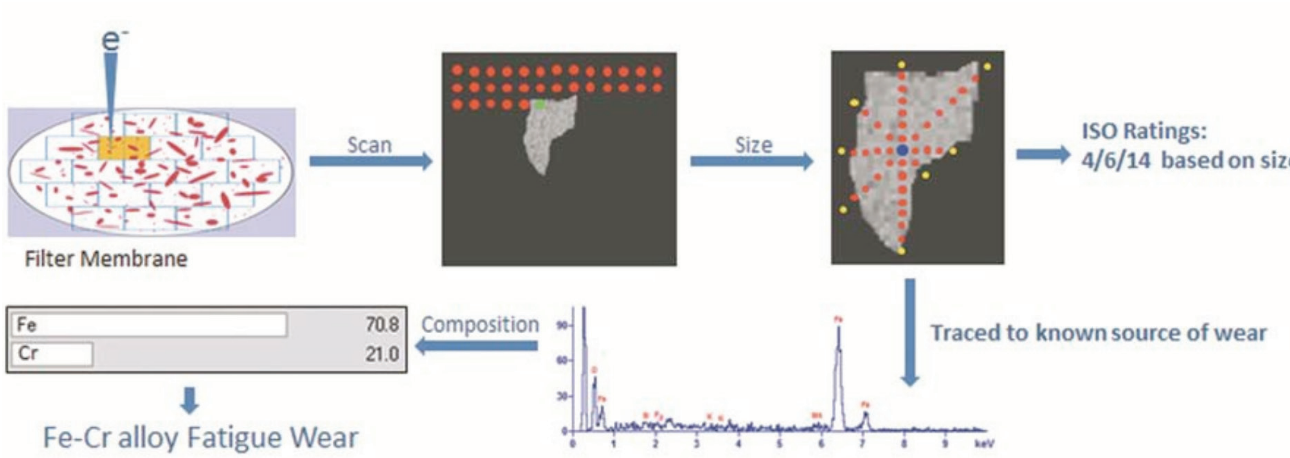
While the particles typically found in oil are much larger than the features one typically associates with an SEM, the SEM is well suited to analyzing these particles and brings with it an array of talents that are unavailable in the other instruments often used for oil analysis. In addition to being an imaging instrument, the SEM when combined with an EDX detector also has the ability to perform quick quantitative compositional measurements. Thus an SEM can not only measure and record the size and shape of a wear debris particle; it can also determine the elemental makeup of the particle thereby combining the two worlds of wear analysis, as previously mentioned, into one.

The automated SEM/EDX wear debris analysis systems use a compact single hardware control configuration for both the SEM and EDX components allowing it to be more compact and robust for industrial wear debris applications. Utilizing back-scattered electrons (BSE) on an SEM enables the system to take advantage of the strong correlation of the average atomic number of the particles and the BSE signals. Hydrocarbons and other particle types with a low average atomic number tend to backscatter fewer electrons than metallic particles and other particle types with a high average atomic number. Thus in a BSED image, metallic particles look bright while organics look dark.



↑ **Figure 2.** Shows an overview of the sample preparation and SEM/EDX analytical output capability.

One of the main differences between SEM/EDX and traditional methods is in its sample preparation. Figure 2 illustrates the sample preparation and analysis expectations of the SEM. Ultimately, a small portion of the oil sample is needed to prepare a representative sample on a filter membrane. Once loaded into the system, instead of capturing a high resolution image of the frame, these analyzers move the beam across the full field through a sequential array of fairly coarse steps – constantly searching for a particle of interest and moving to the next field. A particle is detected when the contrast intensity level of the particle exceeds the predefined threshold background set for each analysis activity. This particle-sizing sequence initiates a "rotating 16 chord" algorithm to measure the particle's morphological characteristics. At a 2048 pixel resolution, a series of chords are drawn across the diameter and through the center of the particle at equal angular spacing's. Particle size and shape measurements are then derived from these chords. All variables are then collected for each particle that the system encounters in sequence across the filter. Figure 3 illustrates the dynamic scan and rotating 16 chord algorithm sequences during the particle measurement and EDX elemental X-ray collection phase.



↑ **Figure 3.** Shows an overview of the simultaneous electron beam 16 line chord measurement raster, the particle imaging capture, the elemental spectra output, and the resulting particle classification.

After the particle is detected and measured, an energy dispersive X-ray spectrum is acquired at the center, perimeter, or along each chord for every particle detection event. Once the particle is characterized (size, shape and elemental composition) user-defined rules place them into a "class." If needed, the particles can be relocated and further examined by the operator. The system provides a customizable reporting tool and automatically generates reports of the analyses. In addition, a database stores all results of analyses for monitoring long term processing trends.

Knowing More from the Data

Automated SEMs provide a wealth of data, but the real power of an SEM is the knowledge that the data can provide. One of the biggest challenges for any engineer is in knowing how to use data – and that typically starts with visualizing and interpreting it. What follows are results obtained from an engine trial which best outlines and illustrates the various modalities that one can take advantage of using an automated SEM/EDX analyzer to better understand their mechanical systems.

Average Composition of Particles versus Size Distribution

Imagine spectroscopic and optical particle counters merging into one technique – the result would simply be distribution of compositions based on the size of the particles. In Figure 4, typical SEM/EDX data not only has the detailed chemical composition of every particle analyzed, but it also provides an extremely accurate (within ±0.1 µm) size analysis. By summarizing the data by size bins and then determining the average composition of the particles in that size range, an analyst can obtain a more clear understanding of where the source of the wear is coming. For example, it's observed that in 1-2 µm size range there is elevated Mn, Cr and Ni with a high average amount of Fe. The Cr, Mn, Ni signals all taper off at around 5 µm in size. Eventually, this Fe (potential alloyed steel) signal minimizes around 10-20 µm in size. But also, the majority of particles are sub-20 µm. So there is the possibility to believe based on the results that there is an alloying material failing, however the biggest concern is knowing which one. Herein lies one of the main differences or advantages that this type of data set can provide. If you need to know more, you simply go to the next layer of detailed information.

Size Bins	Total	Area um^2	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Co	Ni	Cu	Zn	Zr	Mo	Ag	Sn	Sb	Ba	W	Pb
1 - 2 um	84	2.1	6.6	1.0	1.5	3.5	0.4	1.1	1.5	3.2	9.0	0.3	2.3	1.0	45.1	0.6	1.4	3.3	2.8	1.0	1.7	0.7	4.0	2.2	0.9	3.6	1.4
2 - 3 um	217	3.5	2.1	1.3	2.3	8.7	0.9	1.1	1.4	2.5	12.3	0.1	1.4	0.3	53.0	0.2	0.3	1.2	2.7	0.3	1.2	0.3	1.7	1.6	0.7	0.5	1.8
3 - 5 um	244	7.6	0.5	2.1	2.9	6.1	0.7	0.8	0.8	1.7	12.7	0.7	0.9	0.6	61.3	0.1	0.1	0.7	2.0	0.1	0.9	0.1	1.1	1.3	0.3	0.4	1.0
5 - 7 um	75	15.5	0.8	2.8	3.1	10.9	1.5	1.4	1.3	3.1	15.8	1.8	0.9	0.1	48.8	0.1	0.2	0.2	1.7	0.1	0.7	0.1	2.4	1.9	0.3	0.0	0.1
7 - 10 um	44	23.0	2.5	2.9	6.5	21.5	0.7	1.2	2.9	4.5	22.8	4.4	0.4	1.4	16.0	0.2	0.1	0.8	3.3	0.2	1.6	0.0	1.7	2.5	1.5	0.0	0.5
10 - 20 um	32	68.2	1.7	3.0	4.2	15.0	0.4	2.9	1.8	4.4	13.5	2.4	2.1	0.0	35.8	0.1	0.2	0.4	2.0	0.1	1.2	0.1	3.7	1.2	3.3	0.1	0.4
20 - 30 um	9	202.2	4.5	0.7	8.2	31.5	1.2	2.5	0.3	6.6	1.4	0.7	1.2	0.1	35.3	0.1	0.0	0.2	0.9	0.5	0.7	0.0	1.2	0.5	0.0	1.7	0.0
30 - 50 um	4	494.5	3.2	2.6	16.4	62.2	0.8	3.1	0.8	3.4	1.4	0.4	0.0	0.0	4.3	0.3	0.0	0.0	0.0	0.6	0.5	0.0	0.0	0.0	0.3	0.0	0.0
50 - 70 um	1	836.4	18.7	1.5	15.7	26.4	2.0	2.9	1.1	6.8	0.0	3.6	1.4	3.2	2.2	0.0	0.0	0.0	2.3	0.0	0.0	0.0	0.0	1.4	0.0	0.0	10.7
70 - 100 um	0																										
100 - 200 um	0																										
> 200	0																										

↑ **Figure 4.** : Example data report for average composition of all particles vs. size distribution.

Classifying Wear Materials

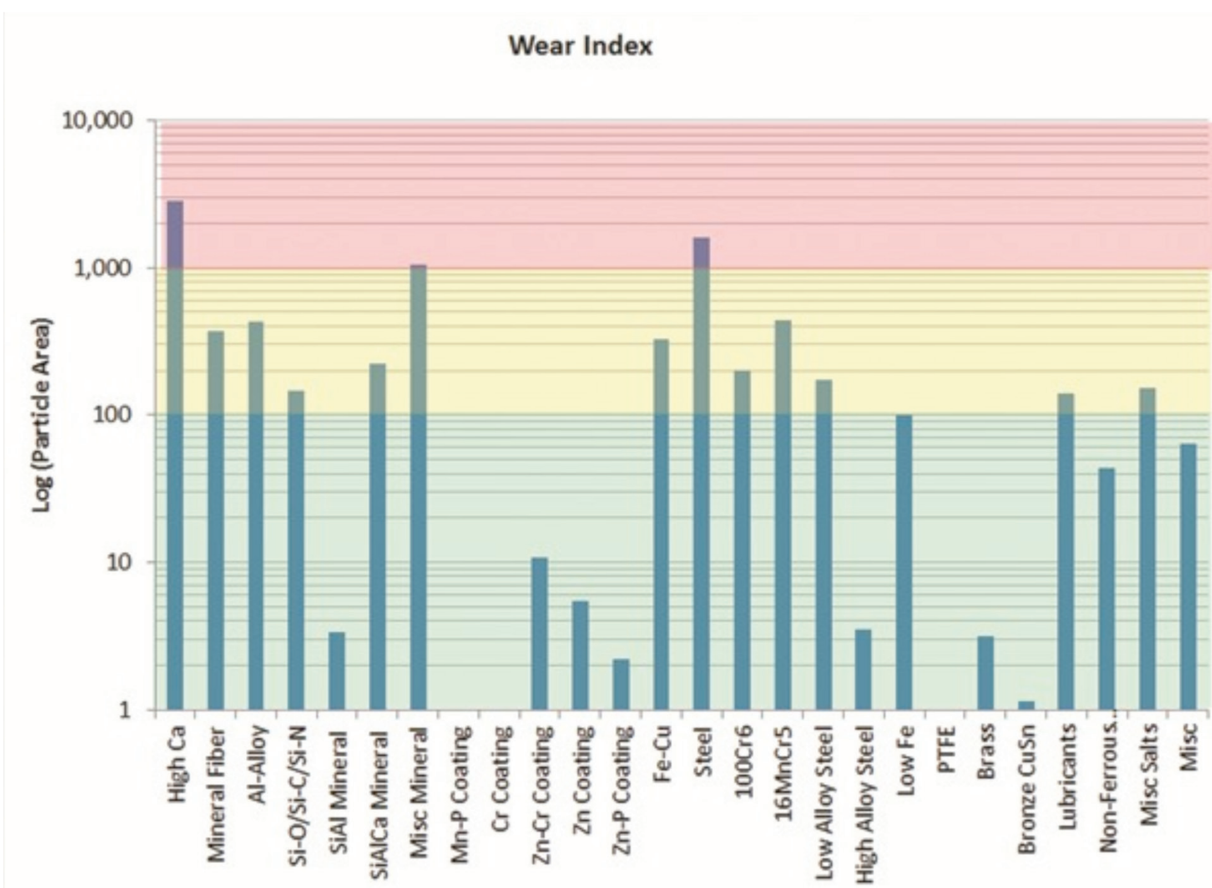
Traditionally speaking, the industry has classified wear debris into basic categories – Rubbing, Cutting, Bearing Fatigue, Gear Fatigue, etc. Characteristic shapes of larger particles often drive this determination. With recent advances in engine and gear technology, tolerances are getting smaller and material wear in the sub-5 micrometer range can be very beneficial in determining premature wear. In these cases, individual chemistries of the particles in conjunction with the sizes/shapes can be a powerful predictive model. In Figure 5, particles are grouped by chemical classification based on the individual particle-by-particle elemental chemistry. By understanding the spectral characteristics and elemental distribution of a particle, one can quickly define classification rules that allow the analyzer to automatically 'group' particles into similar material bins. The cumulative total across several particle size ranges is listed so that the particle size distribution pattern within each class can also be discerned. The counts in the left column have been normalized to represent the total particles found within the sample test volume of 1 mL that was set for each of the samples. It should be noted that with such information, ISO cleanliness values can be readily determined. In addition, a "wear index" of sorts can be generated to facilitate common and proven practices to monitor the overall wear material being generated. This is accomplished not only through counting of the particles, but also through monitoring the 'area' of wear debris particles to understand the combined 'amount' of material present in the oil (Figure 6). For instance, in the case of a jet engine, there are certain materials that when present in relatively low amounts can be alarming (certain bearings). Conversely, the general spike in the abundance of low alloy steel material can be equally detrimental. So, when monitoring an engine or process it may not always be the number of particles that are important, but also the type and amount of material present and their relationship.

Trending Wear Materials

In order to better ascertain the wear mechanisms and predicting imminent failures, the reliability of the measurement and several benchmarking measures must be determined. Typically these are also used for trending purposes to determine when the system is 'in control.' In order to understand these changes, size distribution, classification breakdowns and average chemical composition are often used. Size distribution changes or material type changes can be used to indicate additional fatigue on a product in use. When this information is stored and accessed routinely to obtain statistical trending information on the historical performance of a process, overall red flags & warning signals can also be implemented. Upon analysis, engineers can then conduct the necessary investigations into the process to improve reliability and obtain the source of the problem. This often will result in a wear debris atlas library being generated over time where the operators can quickly reference historical sources of materials and track them back to their sources and failure modes.

DMAX (um) Classification	Total Particles	[1.00-2.00)	[2.00-3.00)	[3.00-5.00)	[5.00-7.00)	[7.00-10.00)	[10.00-20.00)	[20.00-30.00)	[30.00-50.00)	[50.00-70.00)	[70.00-100.00)	[100.00-200.00)	>
High Ca	2868	221	882	882	404	331	147	0	0	0	0	0	
Mineral Fiber	74	0	0	0	0	74	0	0	0	0	0	0	
Al-Alloy	184	0	37	110	0	37	0	0	0	0	0	0	
Si-O/Si-C/Si-N	919	37	331	221	74	110	37	74	37	0	0	0	
SiAl Mineral	110	0	37	37	0	37	0	0	0	0	0	0	
SiAlCa Mineral	699	0	257	110	221	110	0	0	0	0	0	0	
Misc Mineral	3125	184	699	846	368	404	368	110	110	37	0	0	
Mn-P Coating	0	0	0	0	0	0	0	0	0	0	0	0	
Cr Coating	110	0	37	0	37	0	37	0	0	0	0	0	
Zn-Cr Coating	37	0	0	0	37	0	0	0	0	0	0	0	
Zn Coating	257	37	147	74	0	0	0	0	0	0	0	0	
Zn-P Coating	37	0	0	37	0	0	0	0	0	0	0	0	
Fe-Cu	258	37	147	37	37	0	0	0	0	0	0	0	
Steel	11765	1140	3603	5184	1177	184	404	74	0	0	0	0	
100Cr6	110	37	74	0	0	0	0	0	0	0	0	0	
16MnCr5	368	74	74	147	37	0	0	37	0	0	0	0	
Low Alloy Steel	552	74	257	110	74	37	0	0	0	0	0	0	
High Alloy Steel	74	37	37	0	0	0	0	0	0	0	0	0	
Low Fe	257	37	110	74	0	37	0	0	0	0	0	0	
PTFE	0	0	0	0	0	0	0	0	0	0	0	0	
Brass	0	0	0	0	0	0	0	0	0	0	0	0	
Bronze CuSn	74	0	37	0	37	0	0	0	0	0	0	0	
Lubricants	662	110	294	147	37	0	74	0	0	0	0	0	
Non-Ferrous Metal	699	221	110	110	74	110	74	0	0	0	0	0	
Misc Salts	1765	588	625	221	147	110	37	37	0	0	0	0	
Misc	1103	257	184	625	0	37	0	0	0	0	0	0	
Particles of interest	26103	3088	7978	8971	2757	1618	1177	331	147	37	0	0	

↑ **Figure 5.** : Classification of particles based on the chemical composition of the materials and by size distribution in micrometers. (Note: total particle counts are normalized to a 1 ml collection volume.)



↑ **Figure 6.** : Wear Index plot based on the effective area of particles detection by their classification.

Summary

Overall, automated SEM/EDX technology has come a long way. With the advent of integrated analyzers and robust packaging, the technology can now be utilized more routinely in labs or onsite. Their use is further extended due to the fact that data analysis packages are becoming more turn-key and easier to use. The tools are getting even faster with sample analysis times in minutes for a complete oil analysis, improving response times to developing situations, and ultimately, affording users the ability to run numerous samples quickly and provide detailed statistical comparisons of wear patterns between systems over time.

The initial investment in these types of tools can appear high but when compared to the knowledge that they create and the cost savings in predictive maintenance, the return on investment can be rather quick. Finally, the main advantage of these types of analyzers lies in the establishment of sampling protocols. Routine and consistent efforts to obtain samples can then lead to extremely valuable historical baseline wear profiles and provide timely indicators of changes in the status and rate of wear.

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