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SEM-EDS

ADVANCED WEAR DEBRIS ANALYSIS FOR OPTIMIZED MAINTENANCE

AUTOMATED SEM-EDS ANALYSIS FOR ROUTINE CONDITION MONITORING

by

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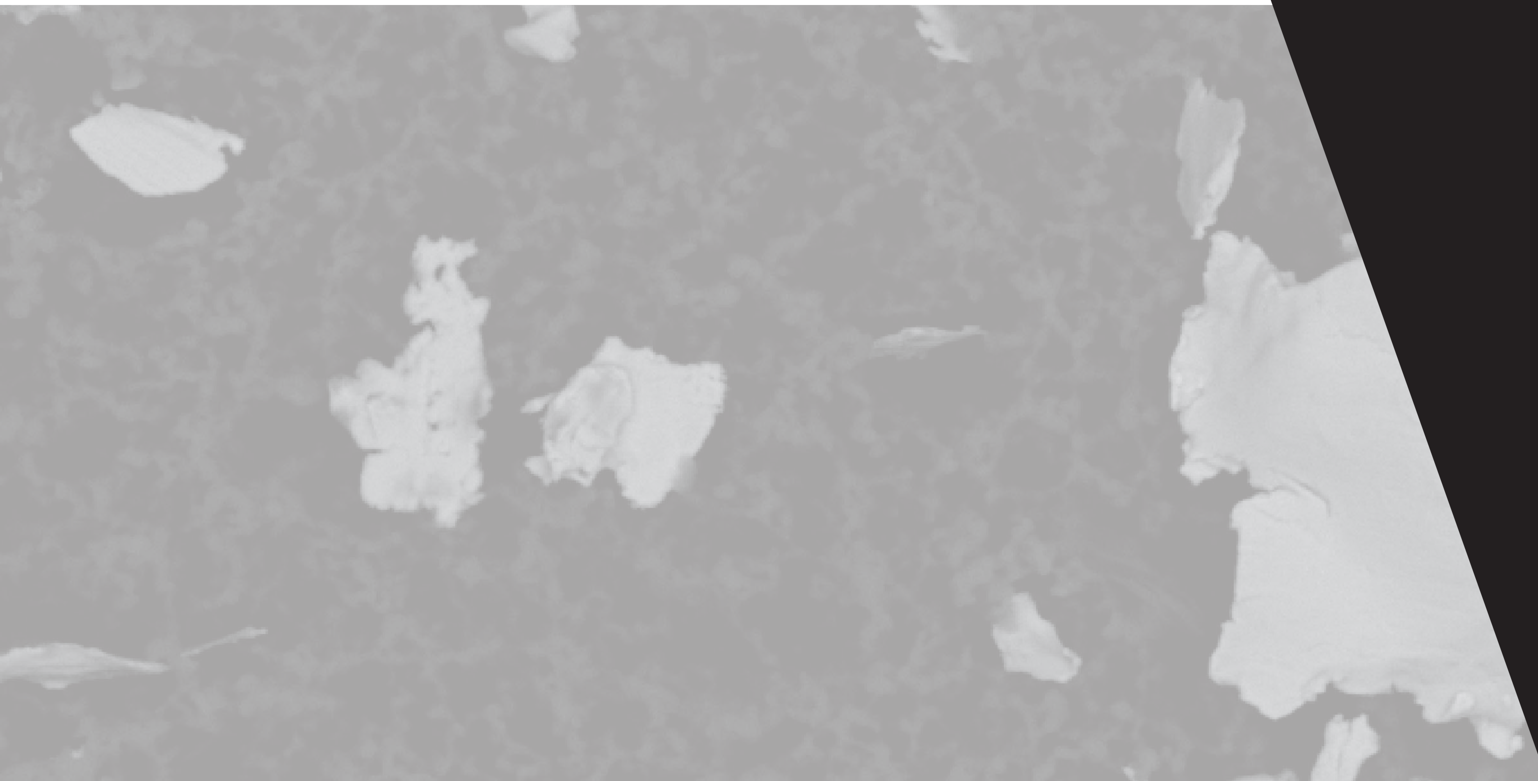
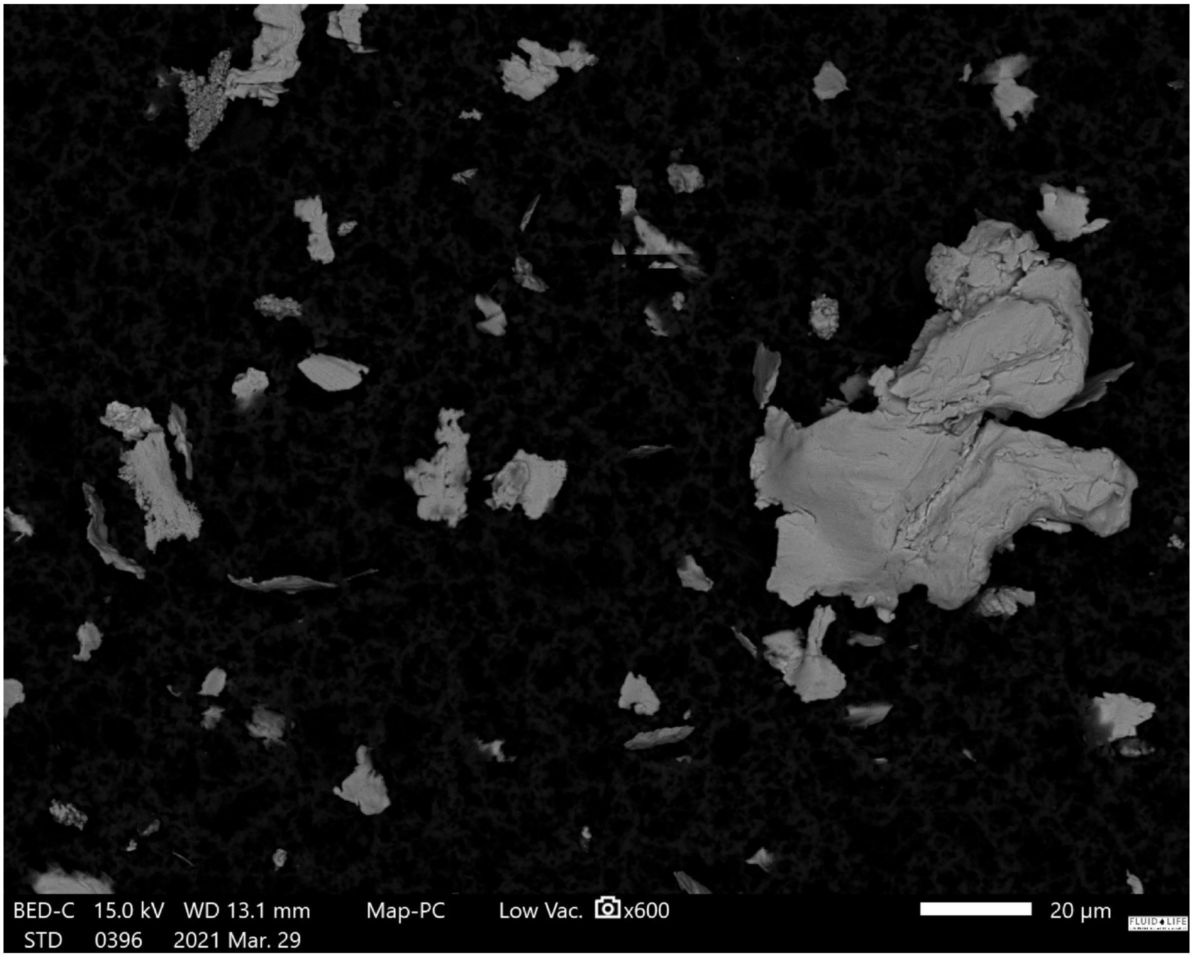


ADVANCED WEAR DEBRIS ANALYSIS FOR OPTIMIZED MAINTENANCE

Executive Summary

Traditional SEM analysis requires time consuming manual analysis by skilled technicians effectively making SEM analysis impractical and too expensive for routine usage in condition monitoring of mobile and fixed plant equipment. With the addition of automation software, big data and AI, SEM-EDS Wear Debris Analysis is a superior method in determining the size, shape, and composition of particles in lubricating oil, grease, filters, and process materials. In this new method, SEM Wear Debris Analysis can analyze hundreds of particles per sample, providing a clearer picture of contamination and wear compared to traditional SEM. Combined with world class laboratory automation, this method enables complete SEM-EDS analysis to become a cost-effective pillar of any routine condition monitoring program and provide critical component protection.

This article will provide the reader with an overview of this technology and demonstrate its practicality as a routine, triggered or advanced test within their engineering and maintenance arsenal.



WHAT IS SEM-EDS

Scanning Electron Microscopy (SEM) uses electrons instead of light (photons) to analyze and visualize the surface features of a material. Electrons have a significantly smaller wavelength than light which results in the ability to study materials at high magnification and with excellent depth of field. Images are presented in greyscale which can reveal additional topographical or composition information depending on the type of detector used.

Energy Dispersive X-Ray Spectrometry (EDS) can be combined with SEM to provide compositional microanalysis of particles and surfaces. EDS is used to determine the chemical composition of a sample including what elements are present along with their distribution and concentration in Mass% and Atomic%.

Combining SEM and EDS (SEM-EDS) enables investigators to determine the size, shape and composition of particles found in oils, greases, filters, DEF, swabs, magnetic plugs, filter screens and process fluids.

TECHNOLOGY OVERVIEW: CONDITION MONITORING USING SEM-EDS

Traditional SEM analysis requires careful and time-consuming sample preparation. Typically, the solid debris from a sample of oil, grease or filter is concentrated on a filter patch and affixed to a conductive substrate. This patch is typically sputter coated with gold or another conductive metal to apply a nanometer scale layer of conductive material on the surface of the sample. As a result of higher operating voltages, this conductive layer is required to provide a conductive pathway for electrons to travel when using traditional SEM instruments under high vacuum.

During SEM analysis, the electron beam, detector, and EDS parameters must be carefully selected and optimized for compatibility with each material. An analyst can then observe, image, and determine the elemental composition of individual particles to gauge their severity and source. This practice has been used for many years to analyze filter debris and oil samples from critical assets such as aircraft engines and gearboxes.

Computer controlled scanning electron microscopy systems have been in use for decades, but these automated systems were expensive to purchase and required considerable expertise to set-up and operate. Modern systems utilizing laser-induced breakdown spectroscopy (LIBS) are also available, but these rely upon a pre-existing library of wear metals for identification, which limits their wider application to unknown sources of wear debris, additives, and contamination for mobile and fixed machinery.

Modern SEM instruments can operate under lower vacuum conditions using a low operating voltage and a backscatter detector which reduces the steps required in sample preparation. Wear debris, contamination and other particles of interest can be isolated from samples of oil, coolant, DEF, grease, process fluids and filter media by filtration through a membrane filter with an appropriate pore diameter. Generally, membrane filter patches are mounted onto an aluminum stub and analyzed. Additional preparation steps, including particle fixation and mounting, may be required depending on the application.

Fluid Life, provider of equipment reliability solutions and lubrication analysis, developed a new protocol for the application of automated SEM-EDS analysis to mobile equipment and fixed asset condition monitoring programs. This new tool is used to diagnose and monitor machinery health and provide actionable recommendations. This process is the result of an industry first combination of automation, software AI, the domain knowledge of analytical chemists, and mechanical engineers, and the practical knowledge of experienced heavy-duty mechanics and planners. By automating and streamlining sample preparation, analysis and particle classification, SEM-EDS analysis is more cost effective and practical for routine condition monitoring of mobile and fixed assets.

In this largely automated method, SEM-EDS Wear Debris Analysis determines the size, shape, and composition of hundreds of particles per sample, providing a clearer picture of contamination and wear versus Ferrography or traditional SEM which are limited to only a handful of manually analyzed particles per sample. The SEM-EDS results are compared against a database containing more than 12,000,000 maintenance and oil analysis records. The resulting reports are reviewed by experienced planners, engineers, and heavy-duty mechanics. This combination of practical domain knowledge and SEM-EDS data leads to actionable recommendations.

COMPARISON OF SEM-EDS TO ROUTINE METHODS

A variety of methods and technologies (Figure 1) are employed in condition monitoring including vibration, oil analysis, ultrasonic, thermography and visual inspections. Oil analysis is typically used to monitor the condition of the lubricant, quantify contamination and monitor wear. A basic oil analysis program may include Spectrometry (ICP or RDE), FTIR analysis, viscosity, a particle count, a test for magnetic iron (Total magnetic iron or PQ index), and tests for specific contaminants such as glycol and water.

	ICP-Spectrometry	Magnetic Iron / PQ Index	Automated Particle Count	X-ray Fluorescence Spectrometry	Analytical Ferrography	SEM-EDS
Particle Size Detection	~ 0-10um	> 40um	~4um - 120um	All	>5um	> 0.1um
Particle Count	-	-	Yes	-	-	Yes
Particle Sizing	-	-	Yes	-	Limited	Yes
Particle Shape Classification	-	-	Limited	-	Yes	Yes
Elemental Composition of Individual Particles	-	-	-	-	Visual Subjective	Yes

Figure 1. Comparison of analytical methods used in Oil Analysis. Source: Fluid Life.

ICP Spectrometry has a well-known loss of sensitivity to particles larger than 10um and is unable detect particles or contamination composed of carbon or other organics. Interpreting the results of an ICP Spectrometry-based oil analysis report can be difficult because the method does not differentiate between sources of the same element, particle sizes or severity. As a result, accurate wear rate and trending is impractical or impossible to implement using ICP analysis alone.

The gap left by spectrometry-based oil analysis, ignores large particles which are a critical, but often neglected, element of an effective oil analysis and condition monitoring program. Particles with diameters larger than 10um provide key information regarding component wear and contamination. These larger particles may provide early warning of abnormal wear and can themselves be abrasive and cause secondary wear.

XRF Spectrometry can detect these larger particles and has been used for decades to analyze wear debris in oil and fuels. Modern EDXRF or WDXRF instruments (Figure 2) can analyze samples of lubricants taken directly from a piece of equipment as well as filter patches prepared from filters and magnetic plug debris. XRF analysis has been shown to detect failure modes involving large particles missed by ICP Spectrometry. These studies relied on detailed knowledge of the metallurgy and maintenance histories of the equipment to determine the sources of wear metals This limited applying the methodology to routine condition monitoring as it is simply not practical to know the metallurgy of every component.

Figure 2. WDXRF Spectrometer used for analysis of Oils and Filter. Source: Fluid Life.

Using Spectrometry alone (ICP, RDE, XRF), it is not possible to pinpoint the specific source of most elements such as copper, which may include material from bearings, brass, bronze, cooler core pacification, paints, anti-seize, mine dust and other sources. Some modes of copper generation can hide or obscure abnormal wear. For example, an engine with a new cooler core may see a rise in copper concentrations in the oil because of cooler core pacification. This generally harmless source of copper may obscure a serious bearing failure generating copper wear debris and requiring action. Spectrometry based oil analysis cannot differentiate between steel housings and steel bearings, gears, and shafts or between outside contamination such as iron containing process dust and wear of steel components.

Other methods (Figure 3) including an automated Particle Count can quantify the number of large particles and provide a means to implement cleanliness and filtration targets. An Analytical Ferrography can be performed to provide information regarding the approximate size of particles and morphological information about the wear mode. The interpretation of an Analytical Ferrography is subjective and dependent on the skill of the analyst. In most cases it is not possible to determine or differentiate specific alloys or sources using an Analytical Ferrography.

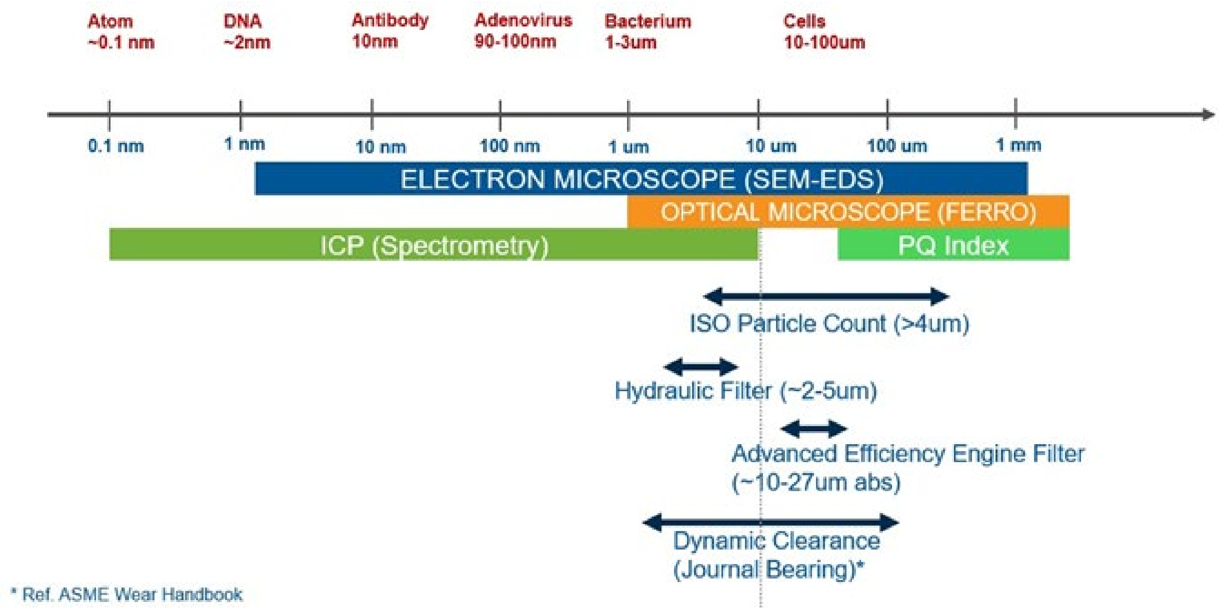


Figure 3. Visual representation of the applicable particle size range for different analytical methods of oil condition monitoring. A visual comparison of common filter sizes is included for reference. Reference: ASME Wear handbook.

The ability of SEM-EDS Wear Debris Analysis to determine the size, shape, and composition of hundreds of particles per sample (Figure 4) and differentiate between wear, contamination and additives overcomes the limitations of traditional oil analysis. From this data the source, size and severity can be determined quantitively and enables informed decision making.

Streamlining sample preparation and automating the analysis and classification process reduces the overall cost to perform SEM-EDS making it practical for routine condition monitoring. Routine sampling is necessary to detect abnormal operating conditions before serious damage can occur and to ensure the that wear particles themselves are retrieved and analyzed shortly after their formation and before their composition and morphology are changed by actions within the system itself. Analysis of both the oil and filter media, where present, allows operators to understand both the current and past machinery health as well as overcome the limitations of oil analysis alone.

Class name	Total Particles (particles / ml)	Summary (Each size - Maximum length [um])			
		10 ≦x < 20	20 ≦x < 40	40 ≦x < 100	100 ≦x < 5000
Steel	615	537	60	15	3
(Al/Si/K) – Outside Contamination	154	147	7	0	0
Silica - Fiber	18	14	4	0	0
Copper Alloy	12	5	0	0	0
Aluminum Alloy	4	3	1	0	0
Brass (Cu/Zn)	3	3	0	0	0
Stainless Steel - AISI 303	3	3	0	0	0
Steel (Cr <2%)	3	1	2	0	0
Bronze (Cu/Sn)	3	2	1	0	0
Contamination (Al/Si/K)	1	0	1	0	0
Contamination (K/Cl)	1	0	1	0	0
Contamination (Na/Cl)	1	1	0	0	0
Steel - (Ni >2.5%)	1	1	0	0	0

Figure 4. Example abbreviated summary output of automated SEM-EDS analysis showing particle size, count, and simplified classifications. Note particles can be classified by source, composition, or alloy. In some cases, different alloys can be separated and distinguished. Distinct sources of outside contamination and metal alloys are separately identified. Source: Fluid Life.

Applications of Routine Automated SEM-EDS include:

- Routine Monitoring (wear and contamination)
- Filter Analysis
- Life Cycle:
    - Early onset failure
    - Mid life catastrophic failure
    - Post Failure Analysis
- Turbine Flush
- Component life extension
- Condition based change-outs
- Monitor Bearing Wear progression

CASE STUDY #1:

Determine the Source of Copper in an Industrial Engine (CAT3412 Generator Set) and if Maintenance Actions are Required

Situation:

ICP Spectrometry results (Figure 5) for the oil obtained from an industrial engine (CAT3412 Generator Set) showed increased levels of copper and iron. This generator set was a critical piece of equipment providing electrical power to a remote site. It was noted that the ICP-Copper signature is often due to cooler core leaching which does not require intervention. However, the possibility of other sources of copper, including severe bearing wear or oil pump failure, could not be eliminated using ICP analysis.

The client was concerned this diagnosis was not definitive as there was no way to determine if the machine was failing without taking the unit out of service for an inspection. The potential consequences of inaction including an unplanned failure of a critical piece of equipment were not acceptable.

Sample Date	Al	Si	Na	K	Fe	Cu	Pb	Sn	Cr	Ni	Ti	Ag	V	Sb	Be	Mn	Cd
June 22, 2021	3	7	1	0	14	85	1	0	0	0	0	0	0	0	0	0	0
June 07, 2021	2	8	1	0	13	89	0	0	0	0	0	0	0	0	0	0	0
March 03, 2021	0	5	0	0	7	48	0	0	0	0	0	0	0	0	0	0	0
December 16, 2021	2	3	0	0	7	14	0	0	0	0	0	0	0	0	0	0	0

Figure 5. ICP-Spectrometry results obtained from the oil from an in-service CAT3412 generator set. Concentrations are presented in units of ug/g (mass). Results are presented in abbreviated summary form. The results for elemental copper are highlighted to draw attention to the reason the service engineer was concerned about this piece of equipment. Source: Fluid Life.

Analysis & Results:

Automated SEM-EDS Analysis was performed on the oil and filter from the engine and did not identify any signs of abnormal wear in the oil or filter. It was concluded that the ICP-Copper signature was due to engine oil cooler core leaching which is harmless in solution. This type of leaching commonly occurs due to new cooler break-in, overheating or changing the brand/formulation of oil. A previous oil sample confirmed that the additive levels had recently changed which supported the conclusion that a change occurred in the brand/formulation of oil.

The operator confirmed that the cooling system was functional and checked for signs or causes of overheating. A new oil sampling port was added to retrieve consistent hot oil samples. The operator continued to monitor the engine oil and filter for this unit using SEM-EDS Analysis, was able to avoid unnecessary maintenance by making an informed decision supported by evidence.

CASE STUDY #2:

Two 40MW Class Gas Turbines (Power Utility)

Situation:

Basic oil analysis was performed on oil samples submitted from two 40MW Class Gas Turbines operated by a power plant supplying electricity to a city in North America.

Routine ICP analysis (Figure 6 & 7) showed an absence of any wear metals, but the particle count was fluctuating outside of limits from sample to sample. The operator was concerned that the particle count results were not consistent with the ICP results for Silicon and Aluminum which were assumed to be outside contamination. Basic oil analysis results did not provide the level of assurance required by the operator to determine if maintenance was required for either turbine.

Turbine #	UNIT	Sample Date	Al	Fe	Cu	Pb	Sn	Cr	Ni	Ti	Ag	V	Sb	Be	Mn	Cd
1	0714-980	2021/06/22	0	0	0	0	0	0	0	0	0	0	0	0	0	0
		2021/01/05	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	0714-970	2021/06/22	0	0	0	0	0	0	0	0	0	0	0	0	0	0
		2021/01/05	0	0	0	0	0	0	0	0	0	0	0	0	0	0

Figure 6. ICP-Spectrometry results for wear metals for samples of oil removed from two separate 40MW class gas turbines. The Turbine number is noted in the first column. Results are presented in abbreviated form with units of ug/g (mass). Source: Fluid Life.

Turbine #	UNIT	Sample Date	Na	K	Si	Al		ISO Code	>4um Count	>6um Count	>14um Count
1	0714-980	2021/06/22	0	0	2	0		17/15/13	690	260	60
		2021/01/05	0	0	1	0		18/16/14	1662	570	112
2	0714-970	2021/06/22	0	0	1	0		18/17/15	2200	910	220
		2021/01/05	0	2	5	0		17/15/12	1100	260	40

Figure 7. ICP-spectrometry and laser particle count results for samples of oil removed from two separate 40MW class gas turbines. The Turbine number is noted in the first column. Results are presented in abbreviated form with units of ug/g (mass) and particles/ml respectively. Reference: ISO 4406:2001 ISO Code. Source: Fluid Life.

Analysis & Results:

Automated SEM-EDS analysis was performed on filters removed from both units. The filters had comparable hours and similar operating conditions. The analysis showed an order of magnitude difference in contamination levels for the two turbines. The filter from Turbine 1 showed 42 total particles (Figure 8) versus 630 total particles detected in the same analysis area from Turbine 2 (Figured 9 & 10).

Most of the particles in the filter from Turbine 2 (Figure 11) were sourced to outside contamination (Silicon, Aluminum) with some traces of carbon steel, stainless steel, and Silver (Ag) overlay. The filter removed from Turbine 1 (Figure 12) contained traces of outside contamination and traces of carbon steel and stainless steel like that found in the filter from Turbine 2.

CASE STUDY #2: CONTINUED

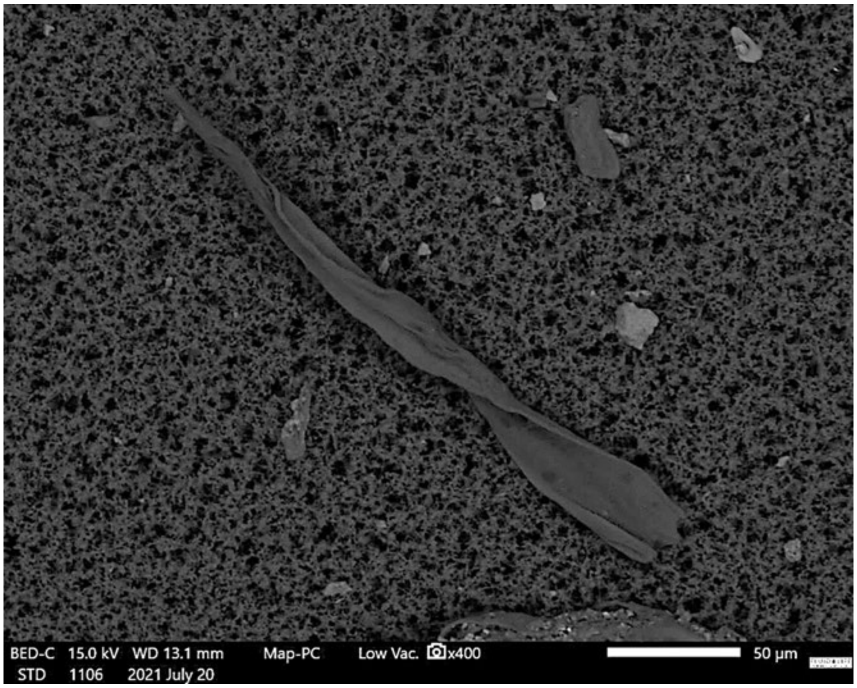


Figure 8. SEM micrograph showing debris isolated from TURBINE 1. The overall debris field shows traces of fibers, organic debris and other outside contamination including silicon dioxide (quartz/sand).

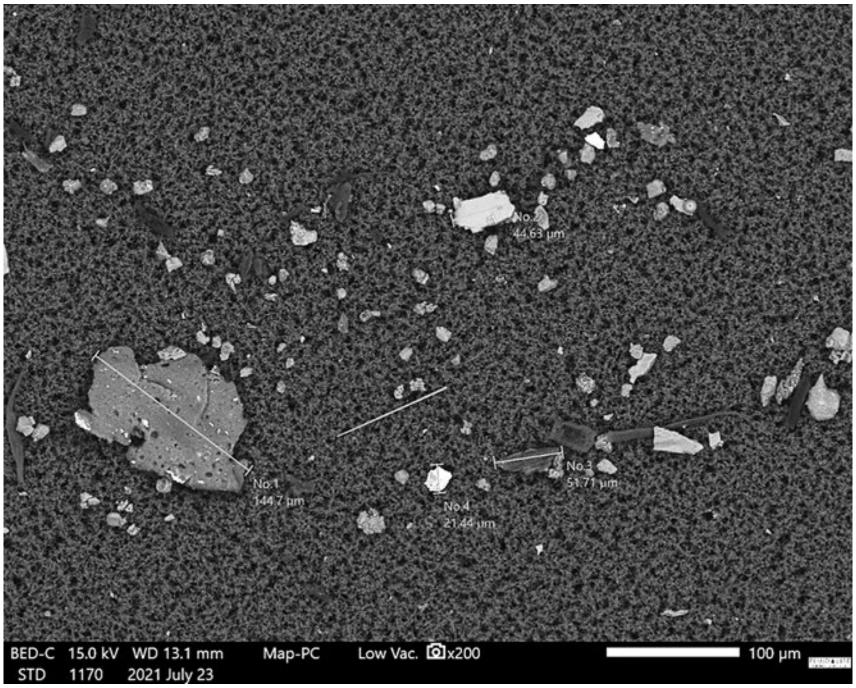


Figure 9. SEM Micrograph showing debris isolated from TURBINE 2. The overall debris field shows elevated levels of outside contamination and wear debris. The largest particle in this figure has an observed chord diameter of approximately 144um and is composed of silicon dioxide (quartz/sand) with embedded wear particles. The remaining wear particles observed include particles with chord diameters in the range of 20-50um. These larger particles would not normally be detected by routine ICP-Spectrometry.

CASE STUDY #2: CONTINUED

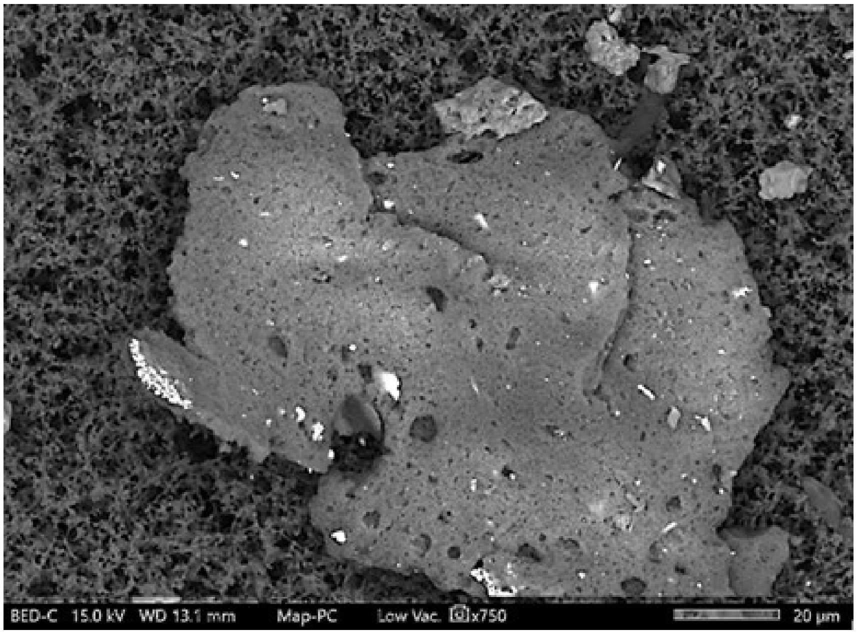


Figure 10. SEM micrograph of particles isolated from Turbine 2. This figure shows a central large particle with a chord diameter of approximately 144um composed of silicon dioxide likely from outside contamination. The lighter shaded particles visible embedded in the large particle are composed of various alloys of steel and lead.

Class name	Total particles	Summary (Each size - Maximum length [um])			
		10 ≦x < 20	20 ≦x < 40	40 ≦x < 100	100 ≦x < 5000
All classes	630	349	91	12	
Contamination (Si)	417	330	75	12	
Contamination (Al/Si/K)	192	8	6		
Steel - Carbon Steel	11	5	6		
Silica - Fiber (Filter Media)	3	1	2		
Additive - PTFE	2	1	1		
Lead	2	2			
Stainless Steel - AISI 303	2	1	1		
Ag - Bearing Overlay	1	1			

Figure 11. Abbreviated summary SEM-EDS analysis data for TURBINE 2. The SEM-EDS analysis shows an order of magnitude increase in outside contamination as compared with the same data from TURBINE 1 (below). Lead and silver were detected and attributed to bearing liner and overlay respectively. Source: Fluid Life.

Class name	Total particles	Summary (Each size - Maximum length [um])			
		10 ≦x < 20	20 ≦x < 40	40 ≦x < 100	100 ≦x < 5000
All classes	42	36	5	1	1
Contamination (Si)	15	13	1	1	
Outside Contamination	9	8	1		
Contamination (K/Cl)	5	4	1		
Steel - Carbon Steel	7	5	2		
Contamination (Al/Si/K)	3	3			
Stainless Steel - AISI 303	3	3			1

Figure 12. Abbreviated summary SEM_EDS analysis data for TURBINE 1. The data shows traces of outside contamination and wear consistent with the SEM image analysis. Source: Fluid Life.

CASE STUDY #2: CONTINUED

Based on the conclusions in the SEM-EDS report, the operator determined that maintenance was required for Turbine 2, but not for Turbine 1. The unit was inspected to ensure proper contamination control was present at fill points, breathers, seals; for new fluids/filters; and when sampling. The oil was filtered offline (kidney loops), and cleanliness was confirmed and monitored using SEM-EDS.

CASE STUDY #3:

Planetary Gearbox

Situation:
Basic oil analysis was performed on oil samples submitted from a planetary gearbox. The concentrations of Copper and Iron were found to be increasing as determined by ICP Analysis.

The operator was concerned that the unit might require replacement and that failure of this gearbox would substantially disrupt operations. Basic oil analysis was inconclusive, so an automated SEM-EDS analysis was performed on an oil sample obtained from an appropriate sample location.

Analysis & Results:
Based on the SEM-EDS Analysis, it was determined that most of the Iron detected by the ICP could be attributed to normal gear wear. The Copper, indicated in the basic oil analysis report, was attributed to normal wear of copper bushings which are inspected regularly and can be replaced. SEM images showed traces of abrasive wear from a bearing race composed of 52100 (Figure 13) and the presence of silicon based external contamination. SEM-EDS also identified wear from the nickel gear overlay and bronze from a thrust washer (Figure 14).

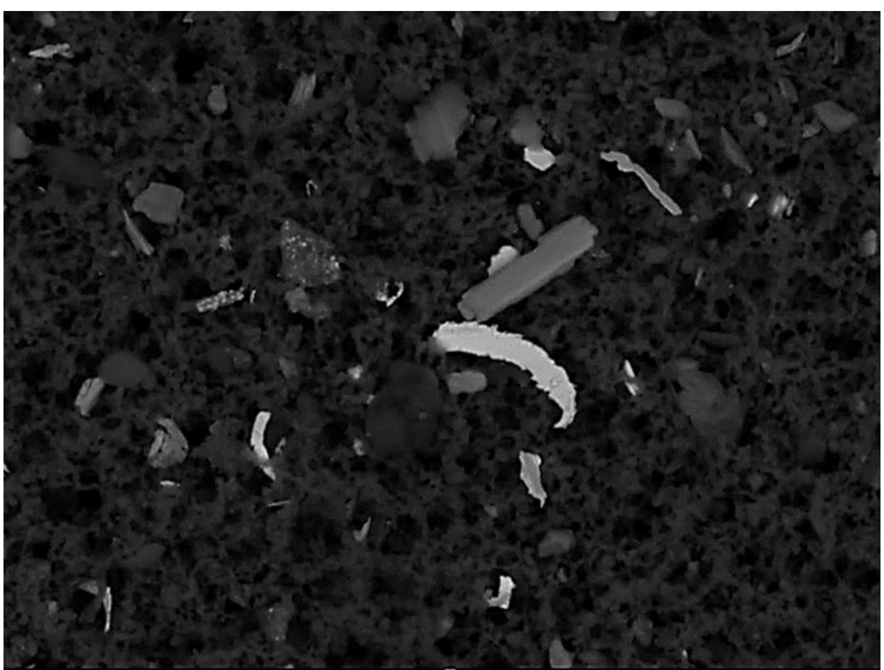


Figure 13. SEM micrograph showing particles isolated from a planetary gearbox including 52100 Steel (bearing Race) and copper from thrust washers. Source: Fluid Life.

CASE STUDY #3: CONTINUED

Class name	Total Particles (particles / ml)	Summary (Each size - Maximum length [um])			
		10 ≦x < 20	20 ≦x < 40	40 ≦x < 100	100 ≦x < 5000
All classes	771	686	79	3	3
Steel	293	258	33	1	1
Steel (4142) - Gears	215	193	20	1	1
Outside Contamination – Silica based	139	133	6	0	0
Steel (52100) - Bearing/Bearing Race	80	65	13	1	1
Silica – Fibers	18	14	4	0	0
Additive - PTFE	9	9	0	0	0
Aluminum Alloy	4	3	1	0	0
Brass (Cu/Zn)	3	3	0	0	0
Copper Alloy	3	3	0	0	0
Steel - (2%<Cr<10%)	3	3	0	0	0
Nickel Overlay	3	1	2	0	0
Bronze (Cu/Sn)	1	1	0	0	0

Figure 14. Abbreviated summary data from the automated SEM-EDS analysis of oil from the planetary gearbox. Steel (52100) was detected along with silica based outside contamination likely causing or accelerating wear of the bearing race. Nickel overlay and steel (4142) were detected indicating normal wear of the gears. Note that the analysis was able to distinguish between various alloys of copper (brass, Bronze, and copper alloy) as well as various alloys of steel. Source: Fluid Life.

Analysis & Results:

It was determined that the gearbox could be left in-service if the abrasive contamination was removed with kidney loop filtration. The ongoing cleanliness and wear of the gearbox were monitored using SEM-EDS Analysis triggered if the particle count, copper, or iron increased as observed using basic oil analysis.

CONCLUSIONS

Spectroscopy- and Ferrography-based oil analysis methods have demonstrated shortcomings and are limited in diagnosing machine health or detecting potential failure. Early attempts at automated SEM-EDS Analysis required specific knowledge of equipment metallurgy, maintenance records and correlational data which limited the application to a wider range of industrial and mobile equipment.

With advancements in software and technology, automated SEM-EDS is now a cost-effective pillar of a routine condition monitoring program. The three case studies presented demonstrate how automated SEM-EDS Analysis, combined with relevant domain knowledge, can eliminate the gaps found in traditional oil analysis programs and provide empirical evidence required for operators to make informed decisions on equipment health and maintenance.

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