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Evaluation of Various Methods to Detect Metallic Wear Particles in Lube Oil  
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**EVALUATION OF VARIOUS METHODS TO DETECT METALLIC WEAR PARTICLES IN LUBE OIL FILTER DEBRIS**

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**Abstract:** Studies were conducted to evaluate methods of detecting metallic wear particles in lube oil filter debris. The methods studied were low temperature oxidation of the organic constituents in the debris, separation of ferromagnetic particles by passage through a magnetic field, and counting/sizing of metallic particles in a fluid stream using a commercially available detector. Laboratory-prepared simulations of wear debris and actual filter debris samples from operational military aircraft systems were used in the evaluation. The relative ability of each method to detect metallic wear particles is discussed, along with the effects of each method on subsequent debris examination.

**Keywords:** Wear particles; filter debris; filter debris analysis; magnetic separation; oxidation; particle counting

**INTRODUCTION:** Historically, condition monitoring of military systems in Canada has relied on Spectrometric Oil Analysis (SOA) and Ferrography. [1, 2] However, increased usage of fine lube oil filtration (10  $\mu\text{m}$  or less) has lessened the efficacy of these techniques due to a reduction of wear-related debris in the lube oil.[3] One technique that has been touted as a potential replacement or supplement for these methods is Filter Debris Analysis (FDA). [3, 4] FDA involves removal and examination of particulate debris trapped by in-line lube oil filters. Wear particle quantity, size, and morphology are used to interpret system wear condition. As the predominant wear modes vary among systems, the success of any FDA program depends on proper definition of what constitutes "normal" versus "abnormal" wear for the particular system under study.

While it is relatively straightforward for most systems to define "abnormal" wear particle sizes and morphologies, definition of "abnormal" wear particle quantity has not been simple. Filter debris contains many particles, such as oil oxidation products, fibers, atmospheric pollutants, etc., which, while they may be of diagnostic significance, tend to obscure the detection and quantitation of metallic wear particles. [3, 4] Traditionally, wear particle quantities have been estimated by visual or microscopic examination, a method requiring significant operator expertise. In the military environment this expertise has proved difficult to achieve and maintain at the field level and widespread implementation of FDA has been correspondingly hindered. Therefore development of an accurate method by which the quantity of metallic wear particles in

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a debris sample can be measured by field condition monitoring technicians has become a topic of considerable research activity. [5 - 7]

Based on the results of some preliminary work [8], three methods were chosen for evaluation as potential field methods for measuring wear particle quantity. These were: removal of organic materials by low temperature oxidation, physical separation of magnetic particles, and counting and sizing of ferromagnetic and conductive particles using a commercially available detector.

Discussion of these methods will proceed in two parts. Firstly, the ability of each method in detecting metallic wear particles will be discussed. Secondly, each method will be evaluated in terms of: effect of the method on wear particle appearance (destructive nature of method), specificity of the method for detecting metallic particles, method simplicity, cost, and analysis duration. It should be noted that this work focuses on the detection capabilities of these methods. Complete evaluation of their quantitative capabilities must await further work.

**PROCEDURES AND EQUIPMENT:** Each method was evaluated using both old lube oil filter debris samples from CH124 main transmissions and T-58 gas turbines, and simulated lube oil filter debris. Simulated debris samples were prepared from crushed activated charcoal (sieved to an average particle size of less than 75  $\mu\text{m}$ ), carbon steel and copper filings, magnetite, hematite and sodium chloride. Metal filings were sieved so as to consist of two particle sizes; average size greater than 75  $\mu\text{m}$  and average size less than 75  $\mu\text{m}$ .

Optical microscopy work was accomplished using a Zeiss Axioplan reflectance polarized light microscope using a 5X 0.15NA strain-free objective. Scanning electron microscopy (SEM) was done utilizing an ISI DS-130. Energy dispersive x-ray analyses (EDXA) were completed with a Princeton Gamma-Tech IMIX system using a light element detector mounted on the SEM. All SEM/EDXA work was done at an accelerating voltage of 20KV, a working distance of 30 mm, a specimen tilt of +20°, and a take-off angle of 38°.

**DETECTION CAPABILITIES:** Each method will first be briefly described followed by a discussion of the techniques used to evaluate detection capabilities.

**Low Temperature Oxidation:** The inorganic content of filter debris has been shown to consist of less than 30% of total debris weight. [3] It has been suggested that if the organic contribution to total debris weight could be removed, then direct correlation of debris weight to system wear condition may be possible. [3, 8] It was attempted here to remove the organic contribution by oxidation at an elevated temperature. Such a process is complicated by undesirable oxidation of metal surfaces and, if temperatures are too high, by melting of some metal particles. A temperature of 300°C was chosen as a compromise. It was thought to be sufficient to oxidize most organics but low enough to prevent melting of lead and other low-melting metals.

To determine the effect of exposure to 300°C on metal surfaces, samples of carbon steel and copper filings were placed in an oven at temperature for 8 hours. Portions were removed after 1, 3, 6, and 8 hours for examination. Figure 1 indicates that the level of oxygen detected on the copper surfaces by SEM/EDXA increases with exposure time. Oxide formation can be seen in Figure 1 to correspond to a "pebbling" of the metal surface. These results were mimicked by

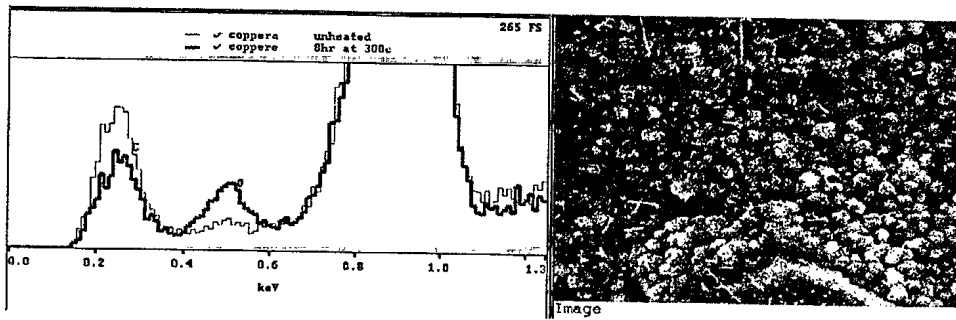


Figure 1: EDXA spectra of copper filings. Oxygen content is seen to increase in sample heated for 8 hours at 300°C (thick line) from untreated sample (thin line). Large peak is due to copper L lines. Image shows "pebbled" oxide structure that forms on heated copper surfaces.

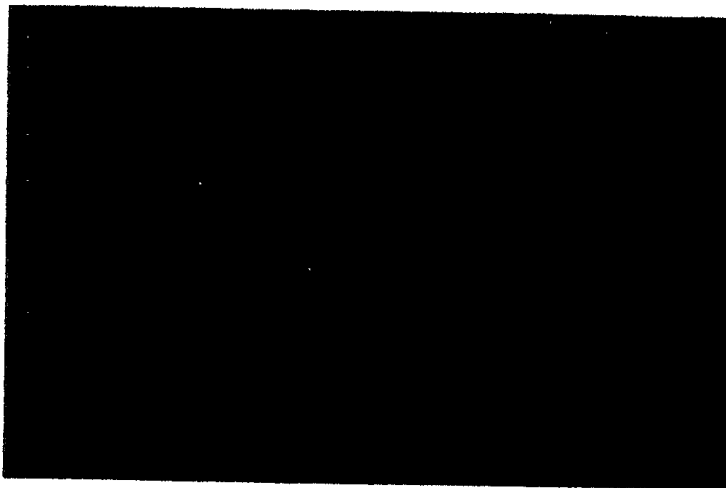


Figure 2: Copper filing after exposure to 300°C for 8 hours.

the steel surfaces, although to a lesser extent. It should be noted that the oxygen peak shown in the x-ray spectra is, even after 8 hours, a minor peak and would not lead to chemical misidentification of the particle.

Exposure to the heat also visibly darkened the metal surfaces. However, this was not pronounced and would not preclude identification of the particle as metallic, as can be seen in Figure 2.

Figure 3 shows cumulative weight losses for two different CH124 main transmission debris

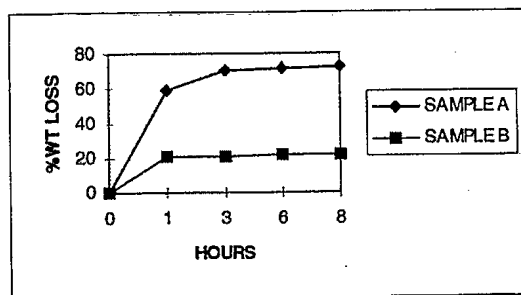


Figure 3: Cumulative weight loss curves for two CH124 main gearbox filter debris samples exposed to 300°C.

samples. Sample A was a typical filter debris sample in that it contained relatively few visible metal particles. Sample B was from a transmission which had experienced significant component wear. It should be noted that in both cases the majority of weight loss due to organic oxidation occurred within the first hour of heat exposure.

While both samples A and B experienced significant weight loss as result of the oxidation, little qualitative difference was visible in the appearance of the debris as a result. Metal particles were no easier to observe than in the untreated samples. Many non-oxidizable materials, presumably inorganic in nature, were still obscuring detection of the metal wear particles.

**Magnetic Separation:** In virtually every mechanical system the critical load-bearing components are made from steel. Therefore, quantitation of the ferromagnetic particles in a filter debris sample should be a good indicator of component wear. It is true that some nonferrous metals are used in most mechanical systems, but it is the author's experience that virtually every serious component wear situation warranting unscheduled and significant remedial action has involved wear of one or more steel components. Therefore an apparatus for separation of ferromagnetic particles from filter debris was assembled. This apparatus consisted of a shielded permanent magnet device having a magnetic field centered along a 10 cm long, 2 cm deep notch. By turning a switch the magnet could be shielded, thus essentially "turning off" the magnetic field. Field strength was 0.15T along the apex of the notch, falling to 0.025T at the roots. The separation apparatus consisted of a glass funnel suspended 30 cm above the magnet device. 0.25" I.D. Tygon™ tubing was run from the funnel, along the magnetic notch and then into a reservoir.

The apparatus was designed for separation of magnetic particles from filter debris. Generally, debris is removed from on-line lube oil filters by extracting entrapped particulates from the filter into a solvent by some physical means, such as ultrasonic agitation. At this point the solvent/debris mixture would be passed through the device with the magnetic field "on".

Magnetic particles would be held in the field and non-magnetic particles washed into the

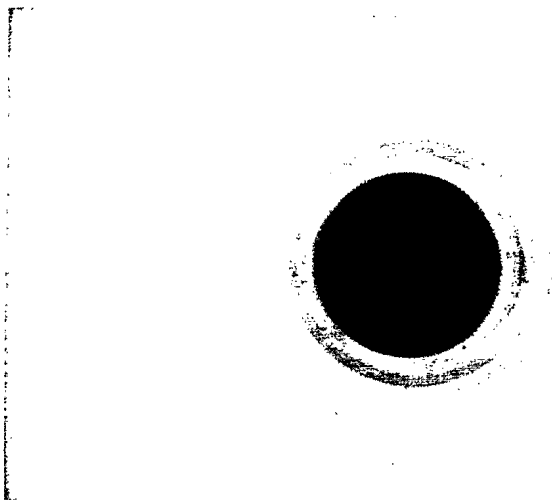


Figure 4: T-58 gas turbine filter debris before magnetic particle separation.

reservoir. Afterwards, the field would be turned "off" and the magnetic particles washed into the additional solvent into a second reservoir. In this way, the filter debris would be partitioned into two samples: magnetic and non-magnetic debris.

Various weights of steel filings were dispersed in petroleum ether and percent recoveries determined using the separation apparatus. To determine if size of ferromagnetic particle affected recovery, two sizes of steel filings (greater than and less than  $75\ \mu\text{m}$ ) were used. Essentially quantitative recoveries (average of 92%) were achieved using only one pass of the mixtures through the magnetic field. Size of steel particles had no apparent affect on recovery rate. Quantitative recoveries were also achieved using fine magnetite powder (particle diameters less than  $20\ \mu\text{m}$ ), but three passes through the magnetic field were required. It was therefore decided to employ three passes for all remaining tests.

Recovery rates using laboratory prepared filter debris samples often exceeded 100%. This was due to entrapment of nonferromagnetic material in the plug of magnetic particles held in the magnetic field and due to solvent absorption by the finely divided activated charcoal. Subsequent SEM/EDXA of the nonferromagnetic debris did not indicate the presence of ferrous species, suggesting quantitative recovery of ferromagnetic material was achieved by the separation apparatus.

The effect of the separation apparatus on old debris samples was evaluated by scraping debris from existing CH124 main gearbox and turbine samples and then dispersing the weighed debris in petroleum ether. As an example, compare Figure 4, which shows the original appearance of turbine filter debris, with Figure 5, which shows the ferromagnetic particles recovered from that debris sample. A considerable reduction in particulate numbers is evident. Quantitative analysis

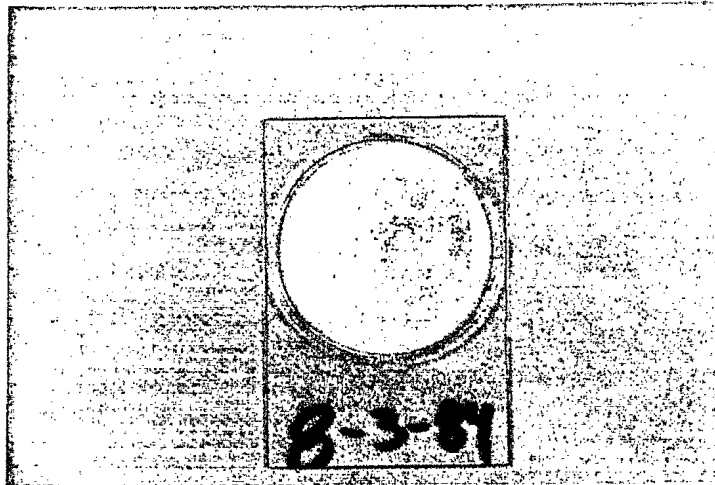


Figure 5: Magnetic particles separated from T-58 gas turbine debris shown in Figure 4.

of the ferromagnetic debris, gravimetrically or otherwise, would obviously be simpler than in the original sample. Although the relative amounts of recovered ferromagnetic particles varied, similar separation results were obtained for all operational debris samples evaluated.

**Wear Particle Counting/Sizing:** A commercially available wear particle measurement system, called MetalSCAN™, was employed as a means of counting metallic particles. The system is an inductive device which measures disturbances to an alternating magnetic field caused by the passage of a ferromagnetic or electrically conductive particle. If the passing particle is ferromagnetic, magnetic field strength is enhanced as a result of displacement of low magnetic permeability solvent. The magnitude of the disturbance is proportional to the volume of the particle. Nonferromagnetic conductive particles are detected by the formation of eddy currents within the particle which act to reduce the field strength. In this case, the magnitude of the field disturbance is proportional to the surface area of the passing particle.

The MetalSCAN™ system sizes particles by making a few assumptions about their composition. Ferromagnetic particles are assumed to be carbon steel. Nonferromagnetic particles are assumed to be aluminum. Using these assumptions, size detection limits can be determined. The manufacturer reports limits of 125  $\mu\text{m}$  for ferromagnetic particles and 400  $\mu\text{m}$  for nonferromagnetic conductive particles. [8] The instrument then counts and reports the number of particles detected within specific size ranges.

It should be noted that if the composition of the passing particle is different from the assumed composition, erroneous values will be reported for particle size. It should also be noted that the



size calculation further assumes spherical particles. As the detection limit is actually dependent on particle volume or surface area, the detection limit is considerably larger for wear particles, which tend to be relatively flat plates.

For the MetalSCAN™ system to be effective, metallic particles must individually traverse the detector. Should two particles pass simultaneously (or at least within the time-response period of the detector) a single response will be recorded. If both particles were ferromagnetic or nonferromagnetic conductive, the overall response will be additive. If dissimilar particles traverse simultaneously, the response will be more complex.

The apparatus consisted of a fixed displacement vane pump driven by a 0.018 Hp AC motor at 1440/1730 rpm which draws solvent (in this case distilled water) from a stainless steel reservoir through 5/8" ID Tygon™ tubing. Water was pumped out the discharge side through 3/16" ID tubing to the MetalSCAN™ detector and emptied into a beaker. Pump and tubing sizes were selected so as to provide a fluid velocity of 9 ft/sec, as is required by the detector.

The MetalSCAN™ detector software was configured to calculate the total weight of detected particles. This was done by determining the weight of a particle of a size at the midpoint of each range, using the composition assumptions described earlier. The total number of particles detected within each range could then be multiplied by this weight and the weights for each range summed to give total weight. Preliminary testing in this manner indicated a linear response for up to 30 mg of carbon steel particles between 250 and 300 μm in size dispersed in water. It was found during this testing that a water volume of 3.5 L was required to sufficiently disperse the particles to permit discrete counting by the detector. This volume was used for all subsequent tests.

To evaluate the efficacy of the detector, 12 CH124 main gearbox debris samples were selected. These samples were graded, using current FDA practices, into 5 severity categories, with 1 being the least severe. The categories were based on the total amount and size of metal wear particles observable with a stereoscope. Using current guidelines, samples having a severity of 3 or higher were considered significant. Each sample was then dispersed ultrasonically into a small volume of isopropanol and diluted to 3.5 L with distilled water. Each diluted sample was then examined using the apparatus described above.

Table I shows the 12 samples arranged by severity level, sample number and MetalSCAN™ results. The MetalSCAN™ results are arranged in descending order of the total weight of detected ferromagnetic and nonferromagnetic particles with the original sample number maintained for comparison. Some of the tests (Sample #1, for example) deemed acceptable by the stereoscopic examination contained among the highest levels recorded by the MetalSCAN™.

Given the past success rate of the visual/stereoscopic method, these results suggested shortcomings in the particle counting method. However, two points must be remembered. First, the stereoscopic approach counts only metallic particles that were visible within the debris while some nonmetallics, particularly ferrous oxides, would be counted in the MetalSCAN™ results. Second, it is possible, as has been discussed earlier, that low numbers of wear particles could be

**TABLE I: COMPARISON OF VISUAL RATING  
WITH METALSCAN™ RESULTS**

<u>VISUAL RATING</u>	<u>SAMPLE NUMBER</u>	<u>MAGNETIC METALSCAN</u>	<u>CONDUCTIVE METALSCAN</u>
		SAMPLE # (MG)	SAMPLE # (MG)
5	12	1 (14.4)	5 (31.4)
	11	5 (6.7)	1 (11.1)
4	10	6 (4.3)	6 (8.2)
	9	3 (3.7)	9 (3.6)
	8	7 (2.1)	3 (3.4)
3	7	2 (1.4)	7 (1.8)
	6	9 (1.2)	11 (1.4)
	5	12 (1.1)	12 (1.3)
2	4	11 (1.1)	2 (0.6)
	3	10 (0.8)	10 (0.2)
1	2	8 (0.2)	8 (0.2)
	1	4 (0.1)	4 (0)

erroneously reported by the visual method due to obscuration of metallic particles by nonmetallic particles.

Figure 6, which shows Sample #1 prior to any treatment, indicates that this filter debris sample did in fact contain a significant amount of nonmetallic particles that may have obscured metallic particle detection by the stereoscopic method. SEM/EDXA examination of the magnetic particles separated from this debris indicated that the majority were ferrous oxides, which would not have been counted by the visual method.

As a further check, the debris samples utilized in this testing were magnetically separated using methods similar to those described earlier. The weight of magnetic debris recovered in each test was quite similar to the ferromagnetic MetalSCAN™ results, further supporting the validity of the MetalSCAN™ results. It is true that ferromagnetic particle counting will detect metal oxides (which, strictly speaking, aren't metallic wear particles), but it would seem beneficial to have such particles detected as this will provide some measure of system component corrosion.

**METHOD EVALUATION:** As stated earlier, the criteria used to rank the methods for further field testing were: destructive nature of the technique, specificity, simplicity, cost, and analysis duration. Each criterion will be discussed separately and then the overall evaluation summarized.



Figure 6: Sample #1 used in MetalSCAN™ evaluation, prior to any treatment.

The magnetic separation and wear particle counting methods are non-destructive and do not affect wear particle appearance or the overall composition of the debris. The oxidation method is destructive as organic materials, which could be of diagnostic significance, are oxidized. Also, the color of wear particles can be altered. Specifically, copper alloys tend to blacken while steels take on straw or bluish hues, depending on alloy type. It should be noted that this change does not affect identification of the particles as "metallic". Further, compositional analysis by SEM/EDXA is not significantly affected.

None of the methods are entirely specific for metallic particles. Both the magnetic separation and particle counting methods record magnetic ferrous oxides as wear particles. However, this is not necessarily a disadvantage as it provides a measure of system corrosion. The particle counting method also detects nonferrous metals and therefore provides a greater degree of specificity than magnetic separation. The oxidation method is not very specific. Any inorganic material, and even some temperature resistant organics, will be detected and counted as metals.

The oxidation and magnetic separation methods would be quite simple to use. Both would require preparation of filter patches using current techniques and simple correlation of patch weight with system component condition. The particle counting is somewhat more complicated in that analyst familiarization with the detector's operating system is necessary.

Cost for implementing any of the methods would be relatively minor. Start-up costs for both the oxidation and magnetic separation methods should be less than \$1K. Start-up costs for the

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counting method will be somewhat higher, but should not exceed \$10K. Operational costs for all three methods should not be significantly greater than that required for current FDA practices.

In terms of analysis duration, both the counting and magnetic separation methods should be only slightly greater than current practices, as both require only passage of the debris/solvent mixture through a detection apparatus prior to patch preparation. The oxidation method would add approximately 2 hours to the analysis time given a 1 hour duration in the oven and 1 hour cooling before weighing.

**CONCLUSIONS:** Separation of wear particles by passage of a debris/solvent mixture through a strong magnetic field and counting/sizing of metallic particles using a commercial detector both hold promise as field methods for measuring wear particle quantity in lube oil filter debris. Field trials utilizing operational equipment should begin to determine the quantitative capabilities of these techniques. The oxidation method is not recommended for further evaluation due to increased analysis time requirements, poor specificity and destruction of potentially significant organic constituents.

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