



Detecting particles in oil

by Alistair Geach, B. Sc.

In the last technical bulletin we looked at four of the seven techniques used in Wearcheck's oil analysis laboratories to detect and quantify particle contamination, as well as the value and limitations of these methods in detecting abnormal wear situations. In this issue we examine the remaining three techniques.

5. Particle counting (sample cleanliness)

So far we have referred to particle sizes and concentrations without much quantification. However this is to be expected because to provide such information from manual microscopic examinations would be a slow process and potentially inaccurate where high particulate concentrations are involved. Automatic particle counters, on the other hand, are able to provide this data very rapidly and inexpensively, but the data is limited to particle size and concentration only. Little if anything at all can be learned about the nature of the contamination from the output of these instru-

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ments and they are primarily used to determine oil cleanliness.

The quickest and most common automatic particle counters found in laboratories are laser optical counters that work on the 'light blockage' principle. Although these instruments produce very specific size and count data, the bulk of the data generated is often ignored in reports and only the cleanliness code is reported. Cleanliness codes are normally calculated according to the ISO 4406 specification. The most recent version of ISO 4406 (1999) requires oil cleanliness to be reported in terms of a cleanliness code comprising a set of three numbers representing the approximate concentrations of contaminating particles that fall into the 4, 6 and 14µ size ranges.

Optical particle counters can count and size particles from a couple of microns up to about 500μ in size. They are easy to use and can quickly provide count and size data as well as calculate the applicable ISO 4406 cleanliness code.

Particle counters calculate the cleanliness code.



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The instrument typically contains a laser source and detector separated by an optical cell through which the oil and the laser beam pass at 90°. As particles in the oil pass through the laser beam, they block off some of the beam and cause a shadow to move across the laser detector. The detector's electronics are able to sense the shadow and then output a voltage related to the size of the shadow falling on the detection surface. This results in a voltage pulse being generated that can be counted as a particle and also measured against 'voltage' calibration data to determine the size of the particle that caused the pulse.

Cleanliness data is useful for trending. In practice there are many limitations with this technology that make many samples unsuitable for testing. Samples containing soot, water, or very high levels of contamination cannot be tested accurately as they yield grossly erroneous results. High soot levels block out the laser beam completely while high levels of con-



Figure 1. Cleanliness code

tamination result in two or more particles being counted as one as they appear in the beam together and are detected at the same time. One may expect the laser to pass through water droplets making them invisible to the detector but. in practice, light is scattered and refracted by the droplets so they cause a detector response and incorrect count and size data. The sensitivity of the technique is also an issue when sampling very clean systems, as the elimination of external contamination becomes a primary concern. With these systems, sample bottles certified to a specific cleanliness should be used and special sampling procedures put into practice. The testing laboratory also needs to be aware of the nature of the sample so suitable handling procedures can be put in place to eliminate the potential for contamination of the sample within the laboratory itself.

In spite of these drawbacks, optical particle counting remains a useful technique to obtain cleanliness data for trending purposes and to screen for samples that should be selected for a more indepth particle analysis. If particle count data is to be trended it is important to have a high level of standardisation in sampling and testing methods and it is advisable to use the same lab and the same instrumentation for all tests. Interlaboratory correlation is known to be poor for particle counting tests and although ISO 4406 (1999) has gone a long way to improving repeatability between labs, many labs have yet to adopt the new standards. Adoption of the new standard throughout the industry is going to be slow because calibration procedures have become complex and expensive to perform and many of the existing instrumentation cannot meet the requirements of the new standards. Wearcheck is investigating new particle count-

STANDARD APPLICATION	PREVIOUS	CURRENT
Calibration of automatic particle counters for liquids	ISO 4402	ISO 11171
Method for coding the level of contamination by solid particles	ISO 4406 (1987)	ISO 4406 (1999)
Calibration material	ACFTD	ISO MTD

Table 1. Old and new ISO standards

ing technology to address these issues.

6. Particle shape classifier

A new and interesting instrument has been under evaluation in Wearcheck's main laboratory since



Figure 2. Conceptual diagrams - Optical particle counter vs Particle counter and classifier

April 2002. This instrument uses state of the art technology to combine a particle counter and a particle shape classifier into a single instrument. Internally it has a similar geometrical format to a conventional optical counter with a laser beam, an optical cell and a detector, but the individual components are quite different. The continuous laser beam is replaced with one that is pulsed, the optical cell is constructed differently with a larger 'optical' aperture and the shadow detector is replaced with a lens and a digital camera.

To take a measurement, the oil sample is pumped slowly through the optical cell where it is illuminated by flashes of light from the laser diode. Each time the sample is illuminated, an image of the oil is captured as a magnified image by the digital camera. Although the oil is moving through the cell, any oil debris is frozen in space in the recorded image as a result of the illuminating flash from the laser diode. A typical analysis involves capturing images over a two-minute period at a rate of 30/sec. During this time about 1ml of oil flows through the cell and in excess of 3 500 images are collected containing 'pixel maps' of all the particles that were 'photographed' in the optical cell. These images are processed through image processing software and a neural network to generate the final data reported on the sample.

The instrument not only provides accurate particle count data that can be reported to a number of industry standards, including both the old and new ISO 4406 standard, but also categorises particles greater than 20μ from their shape characteristics. Particles are categorised as sliding, fatigue, cutting, non-metallic, fibres, water droplets or unknowns. Water New instrument will significantly enhance diagnosis.

droplets are identified and eliminated from count data and the software can be set up to calculate the percentage of water in the sample if required. Other advantages over conventional counters are a higher count threshold allowing highly contaminated samples to be successfully analysed, and an independence of new and demanding ISO APC calibration procedures. Calibration depends on the physical size of the camera pixels, the magnification of the lens and the physical position of the various components relative to one another. Calibration fluids are not required but remain useful as they can be run as samples to periodically check the instrument's results against the standard's certified values to confirm the system is operating correctly.

Although this technology is new and exciting it will take some time to evaluate and commission the instrumentation as the accuracy and value of the particle classifying technology needs to be established for the variety of oil samples supplied from the wide range of equipment sampled by Wearcheck's customers. This exercise is essential if the full potential of this instrument is to be realised.

7. Spectrometer

The value of identifying abnormal wear situations from particle counting and classifying techniques is limited from a troubleshooting point of view unless one can establish with some degree of confidence where the abnormal wear particles are being generated.

To make decisions of this nature, one needs to know the composition or metallurgy of the particles and have a working knowledge of the design and material make-up of all the lubricated components within the equipment sampled. Determining the composition of individual particles is very costly and seldom carried out by commercial oil analysis laboratories unless their services extend to include equipment failure analysis. A commercial lab will however routinely conduct a spectrometric analysis as a primary test that yields data from which some deductions can be made about the source of particulate contamination. Many labs use an ICP spectrometer for this purpose.

An ICP (Inductively Coupled Plasma) spectrometer is an atomic emission spectrometer and is used to determine the total elemental concentrations derived from oil additives, wear metals and contaminants in a diluted oil sample. When an oil sample is analysed on an ICP spectrometer it is diluted with solvent and aspirated into the central region of an extremely hot Argon plasma (electrically charged gas) where the elemental emissions that are measured by the spectrometer occur. For particles to be included in measurements they have to be small enough to be carried inside minute sample droplets into the plasma. In practice, only particles of about 5µ in size or less are able to complete this journey. Particles greater than about 5μ are either left behind on initial dilution, fall out of suspension after dilution or exceed the carrying capacity of the aerosol droplets during aspiration and do not make it to the plasma.

Because additives are dissolved in oil, elemental concentrations associated with ICP measurements on additive elements (eg. Ca, Mg, Zn and P) are representative of the additive levels in the sample. However, the same obviously cannot be said for wear metals and other particulate contaminants. Any ICP results that apply to wear metals and contaminants only apply to those particles in the sample that are about 5μ and less in size and this needs to be a consideration when interpreting results. Fortunately abnormal wear situations are more often than not reflected in ICP results because, when abnormal wear occurs, the generation of large abnormal particles tends to be accompanied by an acceleration in the rate of small particle generation as well.

It is also very important to realise that ICP results represent the total concentration of the element reported, and skill is needed when interpreting these results. A single result on an element may contain contributions from numerous sources and, for this reason, there is seldom any value in looking at a result in isolation. Often, results on a number of elements need to be assessed together if a meaningful conclusion is to be drawn. A higher than normal lead (Pb) reading on a sample from a petrol engine run on unleaded fuel for example, should raise the following questions: Is abnormal bearing wear taking place? Has the driver filled up with leaded fuel in error, or was the unleaded fuel contaminated with leaded fuel? Is the result possibly a combination of contributions from both sources? Was the sample gun washed out with petrol after the previous sample? Looking at the lead value relative to the values for copper and tin could go some way to answering these questions.

High silicon levels on the other hand could indicate that dust or dirt particles have entered the system, but could equally result from the leaching of silicone-based gasket sealer into the oil. If the high silicon level is not accompanied by increased wear metal levels across the board and the silicon to aluminum ratio is fairly high, it is unlikely that dust particles are the source of the contamination

HIGH LEAD READING FROM ICP SPECTROGRAPHIC ANALYSIS				
Leaded Fuel used	d Sn Reading	Pb:Sn Ratio	Possible Conclusion	
Yes	Normal	N/A	Lead reading from fuel combustion by-product. (Expect ±1000ppm /1000km)	
No	Normal	N/A	Lead reading from combustion by-products of fuel contaminated with lead. (Expect <<1000ppm/1000km)	
No	Abnormal	Ratio consistent with bearing wear.	Big end or main bearing wear and if lead reading is very high, a contribution from contaminated fuel cannot be ruled out too.	
HIGH SILICON READING FROM ICP SPECTROGRAPHIC ANALYSIS				
Al Reading	Other Wear Metals	Si:Al Ratio	Possible Conclusion	
Normal	Normal	N/A	Silicon contamination with Silicone from gasket sealer.	
High	High	Ratio con- sistent with dirt entry	Silicon contamination consistent with dirt entry.	

An ICP measures total elemental concentration.

Table 2. Multiple sources of Pb and Si New instrument will increase usage of analytical ferrography.

Need troubleshooting advice ?

Wearcheck sells two troubleshooting charts for display in workshops, training centres and technical departments - one for engines and one for drivetrains. These are durable laminated checklists in A2 size (Product Code WA2C) or A4 size (Product Code WA4C) which detail common problems and possible corrective action. Also view them on Wearcheck's web site under Publications.

Conclusion

While seven techniques to detect and measure particulate contamination in used oil have been described, they all have limitations in specific areas and one ideally needs to look at all the test data as a whole to make valid assumptions and arrive at suitable conclusions concerning machine health. The single most powerful technique discussed is analytical ferrography but this is not carried out routinely as cost and time constraints prevent the service from being widely used in oil analysis programmes. A one-off ferrographic analysis has its place but ideally the machine being assessed should have well established baseline trends for particulate contamination that can be used in interpreting the seriousness of a developing problem.

Wearcheck's new particle counter/classifier has the potential to help increase the usage of analytical ferrography, as basic shape data will be routinely recorded. Previously this has been unobtainable cost-effectively. This data is expected to trend well and provide reliable information that will facilitate the identification of samples for which in-depth ferrographic analysis is likely to be most beneficial. Realisation of this expectation will largely depend on how consistently the shape classifications derived from pixel images of particles match the classifications derived from traditional ferrography. With sufficient correlation, baseline trends derived from pixel images can be used in conjunction with one-off ferrographic data to help in assessing the health of a machine or a machine component.

Even without the added advantage of analytical ferrography, the new shape classification data will add significantly to the information available to a diagnostician to help him make an informed decision on machine health and, ultimately, it must have a positive impact on the quality of these decisions.

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