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Rotrode Filter Spectroscopy: A Recently Improved Method to Detect and Analyze Large Wear and Contaminant Particles in Fluids

ABSTRACT: Routine spectrochemical analysis of used oil samples with rotating disc electrode (RDE) or inductively coupled plasma (ICP) emission spectrometers has become standard practice in machine condition monitoring programs to determine elemental content. Unfortunately, both of these techniques become less responsive to particles as they increase in size. To overcome this limitation, several complementary methods and analytical techniques to detect large particles in used oil have been applied, including acid digestion, ferrography, and X-ray analysis. Unfortunately, these techniques are not practical or economical to implement in a high sample throughput machine condition-monitoring program. One technique that has demonstrated success as a complementary method for detecting large particles is rotrode filter spectroscopy (RFS). RFS extends the capability of rotating disc electrode (RDE) spectrometers to detect particles greater than 10 μm in size.

KEYWORDS: Rotrode Filter Spectroscopy (RFS), rotating disc electrode spectrometer, RDE, used oil analysis

Introduction

Spectrometric oil analysis has been applied for more than 40 years as a routine and cost-effective condition monitoring technique [1]. It is used to determine the elemental concentration in parts per million of wear metals, contaminants, and additives in a used oil sample. Commercial oil analysis laboratories report on as many as 20 different elements. With the knowledge of the wear metal and contaminant limits for the machine or engine being monitored, a determination may be made as to whether or not that equipment is operating properly.

A fact that has recently become more widely understood is that spectrometric oil analysis detection efficiency decreases as the wear particle size increases [2]. This partial limitation is not a problem with reciprocating engines that generate mostly small particles, but it is sometimes a problem in fatigue failures of rolling element bearings, such as those in military gas turbines, which generate large particles at the outset of failure without generating many small particles. In addition, some wear modes such as spalling, severe sliding wear, and cutting wear generate large particles that may go undetected by spectroscopy [3]. This is true to a greater degree for certain types of spectrometers than others. It is also more important for certain types of machines and engines.

Atomic absorption spectroscopy (AAS) and inductively coupled plasma (ICP) spectrometers suffer the most from particle detection inadequacies. Rotating disc electrode (RDE) spectrometers are responsive to somewhat larger particles, but the upper limit is still approximately 10 μm [4]. Ferrography and X-ray analyzers address this limitation and are

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capable of identifying large wear particles as a complementary technique. However, these instruments are not always practical or cost-effective in a used oil analysis laboratory environment that competes on the price of samples and may process hundreds or even more than 1000 samples per day.

The RFS method makes use of the fact that carbon disc electrodes used in RDE spectrometers are porous and can be used as a filter to trap particles in oil samples. Early versions of the RFS fixture used standard disc electrodes with some success and acceptable sensitivity [5]. However, standard disc electrode geometry caused the process to be slow, and lack of manufacturing control of disc porosity resulted in poor repeatability. Due to its ease of use and inherent advantages, the RFS concept was recently revisited with the intent of further improving performance. The updated RFS technique uses custom ring-shaped graphite electrodes and a semi-automatic process to improve sensitivity and repeatability and to reduce sample preparation time.

This paper describes the theory of operation of the RFS technique, the evolution of the concept based on experience, and how it is applied in commercial used oil analysis laboratories.

Theory of RFS

A microscopic inspection of the carbon disc electrodes used in rotating disc electrode (RDE) spectrometers reveals that they are porous. Rotrode filter spectroscopy (RFS) makes use of this fact, and a funnel-based fixture (Fig. 1) is used to clamp the discs so that used oil samples can be drawn through the outer circumference of the disc when a vacuum is applied. The filtering process through the disc captures the particles in the oil. The oil is then washed away with solvent, and the disc is allowed to dry. The particles are left on the outer circumference of the disc electrode so that they are vaporized and detected when sparked on the RDE spectrometer.

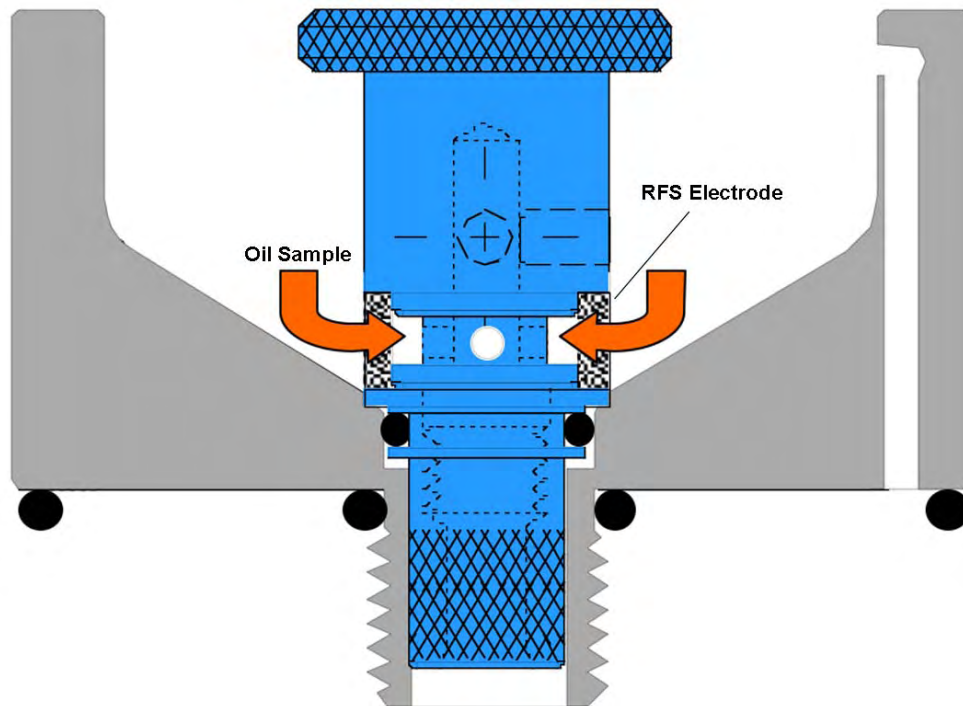


FIG. 1—RFS disc electrode filtering oil through its circumference.

The RFS technique is used as a comparative method due to the unavailability of oil standards with known gravimetric concentration of particles for each element measured by the spectrometer. In practice, a used oil sample is first analyzed using the standard RDE technique, which provides an analysis of dissolved and small wear particles. A second analysis of the same sample using the RFS technique detects large particles. The two analyses provide an indication of the wear particle size distribution in the sample. A sudden presence of large wear particles will not be seen by conventional analysis alone. Their presence, however, will be readily evident through the RFS analysis.

The RFS Fixture

The original RFS fixture to prepare electrodes for the analysis of large particles was first developed in the early 1990s (Fig. 2). The fixture used standard disc electrodes, which were clamped in the funnels to filter and capture large particles. It also had five stations so that five samples could be processed simultaneously. A vacuum pump was used to pull the sample and filter it through the electrode. A manual rinse with heptane was used to wash away any remaining lubricant. Sample preparation time of an electrode for RFS analysis varied with the viscosity and contamination of the oil sample. Preparation time could be as short as 4–5 min for relatively clean, used lubricating oil samples, such as from turbines, electric motor bearings, and hydraulic systems. Engine oil samples with high soot levels require the longest filtration times, sometimes half an hour or more.



FIG. 2—*Early RFS fixture.*

It has always been a problem that standard disc electrodes are not manufactured with consistent porosities, which affects the capture efficiency of the particles. This variability hurts repeatability and thus makes data trends, on which used oil analysis maintenance recommendations are based, more difficult to establish. Furthermore, long sample preparation times for highly sooted samples from diesel engines were not practical for some laboratories. These factors led to a new system that addressed these concerns and offered additional improvements. The updated and improved system is known as automated rotrode filter spectroscopy (A-RFS) (Fig. 3). It greatly improved repeatability through the use of controlled electrodes, automation, and a vacuum/pressure pump to reduce sample preparation times.



FIG. 3—A-RFS system.

The A-RFS system is a semi-automated sample preparation instrument with a five-station fixture of funnels. It works on the same principle as the original RFS system. However, it uses a special electrode manufactured to more consistent porosity specifications, it has an automated cleaning cycle, and its filtration times are reduced by the application of a vacuum/pressure pump to both pull and push the used oil sample through the electrode.

Conventional off-the-shelf electrodes were used as a filter in the original RFS method. Experimental data over time confirmed that the porosity of these electrodes was not controlled in the manufacturing process, and thus it had a detrimental effect on repeatability when new batches of electrodes were purchased. The new A-RFS sample preparation process begins with a specially designed RFS electrode. The RFS electrode is custom manufactured to consistent porosity specifications. Thinner walls improve repeatability and reduce filtration times. The dimensions of the standard disc electrode used for conventional RDE analysis and the new RFS electrode are shown in (Fig. 4).

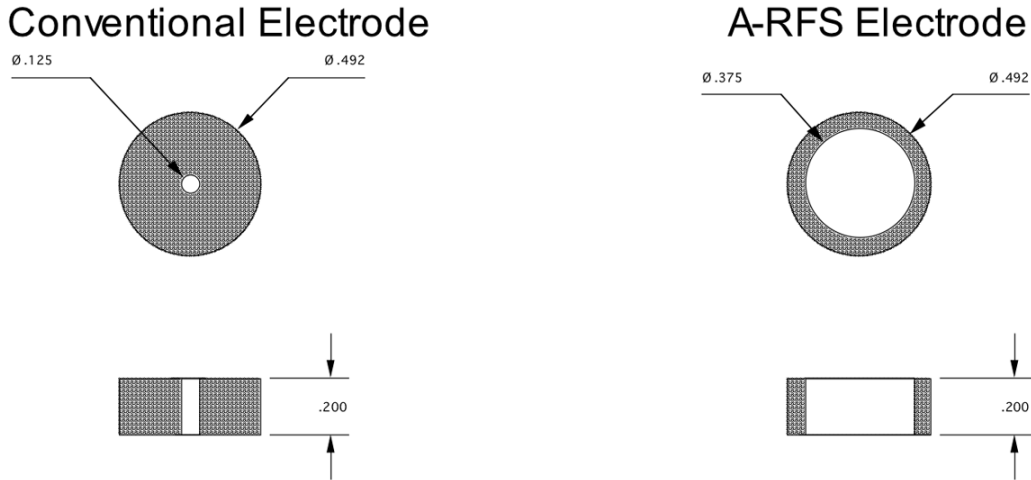


FIG. 4—Conventional electrode and RFS electrode.

In routine operation, the A-RFS disc electrodes are mounted on an electrode clamp assembly for installation in one of the five preparation stations. The electrode clamp assembly is made of two separate pieces so that a new electrode can be mounted on it directly from its container, thus limiting the possibility of contamination through handling (Fig. 5a.). The clamps are numbered so they can be easily identified and associated with their matching oil sample.

The electrode clamp assembly with a fresh carbon disc is then installed in one of the RFS sample funnels. An oil sample is poured into the funnel, and the funnel is clamped shut (Fig. 5b). With the start of the automated process, a vacuum/pressure pump creates a pressure drop of approximately 5 atm across the disc electrode, causing the sample to flow through the disc electrode. The filtration process captures and concentrates large wear particles on the surface of the electrode. A sensor determines when the sample has been filtered through the electrode and automatically starts the electrode cleaning cycle followed by a drying process. Heptane is used as a solvent to wash the remaining oil from the RFS electrode and to re-suspend any particles remaining in the funnel so that they can be filtered and captured.

The RFS preparation process is complete when all of the oil has passed through the disc electrode, residual oil has been washed away, and the electrode is dried. The operator then removes the electrode clamp assembly and installs the shaft complete with the electrode in the spectrometer for analysis (Fig. 5c).



FIG. 5—Preparation process of the A-RFS electrode.

RFS Data Interpretation

Unfortunately, calibration standards prepared with actual wear metal particles at various sizes and concentrations are not available. Also, even if they were available, shelf-life, particle settling, and the actual distribution of particle sizes could be problematic. Thus, in order to provide the basis for benchmarking RFS calibration and to assure that data are consistent, spectrometers are calibrated with certified commercial or military metallo-organic calibration standards. This insures the spectrometer has the same response over time and permits trending of wear metals and contaminant particles. It is important to note that RFS data is therefore not in true parts per million (ppm), but in scalar units that the spectrometer can reproduce over time.

RFS is a powerful analytical tool because it provides additional information on actual wear and contaminant particles in an oil sample. In routine operation, laboratories perform two analyses on a used oil sample. The first is conventional oil analysis that provides quantitative and qualitative analysis of dissolved and small particles in the sample. The second is RFS analysis that provides an indication of large particles and their elemental composition. Taken together, trends of the two analyses can be used as a trend to provide a much clearer picture as to the mechanical health of oil wetted systems.

A typical data trend from an aircraft engine is shown in Tables 1 and 2. The first set of analyses (Table 1) is with conventional oil analysis for dissolved and small particles in the oil samples over a period of about 2 months. Conventional oil analysis shows no trends or significant concentrations of wear metals and contaminants. The second set of data (Table 2) is for the same samples but with RFS analysis for large wear particles. Note that the concentrations of several elements such as iron, magnesium, sodium, and silicon are significantly higher than conventional analysis. In addition, other wear metals such as aluminum, chromium, nickel, lead, and zinc are now detected. The RFS analysis also identifies a major trend change at 299 h denoted by a big jump in the iron, magnesium, and silicon concentrations. This signifies that there is a big increase in large particles. Conventional analytical data (Table 1) showed no trend or major concentrations for any of the elements. The aircraft was taken out of service at 312 h, the engine was inspected, and a bearing was replaced. Continued service could have resulted in complete failure.

TABLE 1—*Aircraft engine oil analysis trend, conventional spectroscopy analysis.*

Date	H	Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	B	Mo	Zn	V
12/10/03	262	0	0	0	0	0	0	0	0	0	0	4	1	0	0	0	0
12/12/03	268	1	0	0	0	0	0	0	0	0	0	4	1	0	0	0	0
12/15/03	271	0	0	0	0	0	0	0	0	0	0	5	1	0	0	0	0
12/16/03	278	1	0	0	0	0	0	0	0	0	0	4	1	0	0	0	0
12/18/03	293	1	0	0	0	0	0	0	0	0	0	4	0	0	0	0	0
01/05/04	299	1	0	0	0	0	0	0	0	0	0	3	1	0	0	0	0
01/08/04	307	0	0	0	0	0	0	0	0	0	0	3	0	0	0	0	0
01/15/04	312	0	0	0	0	0	0	0	0	0	0	3	0	0	0	0	0

TABLE 2—*Aircraft engine oil analysis trend, RFS analysis.*

Date	H	Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	B	Mo	Zn	V
12/10/03	262	24	0	2	1	1	18	14	2	2	43	0	2	1	1	7	0
12/12/03	268	15	1	6	1	1	9	9	1	2	19	0	1	1	1	2	0
12/15/03	271	10	0	3	1	0	10	8	0	1	22	0	1	0	0	8	0
12/16/03	278	18	0	6	1	2	7	3	1	2	18	0	1	0	1	6	0
12/18/03	293	14	0	0	1	0	4	1	1	2	12	0	0	2	1	1	0
01/05/04	299	52	0	3	3	1	30	18	4	0	80	0	3	1	0	8	0
01/08/04	307	58	0	7	3	3	47	30	3	1	113	0	4	2	1	11	0
01/15/04	312	106	1	4	8	2	19	14	9	3	45	0	2	2	1	12	1

It is common knowledge that an analysis is only as good as the oil sample. This is even more important with RFS analysis since RFS analyzes large wear and contaminant particles. It is thus important to always take the oil sample from the same place and in the same manner to improve analytical performance. Variations in RFS analysis also will be observed due to particle settling in the sump if samples are taken at varying times after a system is shut down.

RFS analyses, as shown by this example, are capable of identifying and quantifying large particles that would normally be missed by conventional spectrometric oil analysis.

A-RFS Field Experience

RFS has shown itself to be a valuable additional tool to military oil analysis programs. However, it started in industrial applications and continues to provide rapid and valuable information for in-house and commercial laboratories. Experimental data from military application and a commercial case history will be reviewed in this section.

Experimental Data with Metal Powders

In order to verify that the RFS technique can detect particles greater than 10 μm, the U.S. Department of Defense Joint Oil Analysis Program Technical Support Center (JOAP-TSC) prepared samples using M50 alloy particles. M50 alloy is often present in aero-derivative turbines. Some failure modes of J52 engines appear to be sudden catastrophic failures where the root cause began with the lack of lubrication of the 4½ bearing area. Initially, analysis of the samples by RDE spectroscopy did not indicate the failure mode [6]. The composition and specifications for M50 alloy are as shown below (Table 3).

TABLE 3—*Composition of M50 alloy.^a*

Fe	Cr	Ni	Si	Mo	V
89.8	3.75–4.5	0.3 max	0.2–0.6	3.9–4.75	0.8–1.25

^a in % concentration.

The M50 alloy was sieved into known particle ranges and mixed at several concentrations in MIL-L-23699 oil in groupings of 25–38 μm and 45–63 μm . The samples were analyzed using conventional spectroscopy and also RFS [7].

Conventional atomic emission spectroscopy (Table 4) did not detect any of the M50 alloy mixed into the new MIL-L-23699 oil. This was to be expected and confirms the inability to detect large particles in an oil sample.

The same samples also were analyzed with the new A-RFS technique (Table 5). The data verify the capabilities of RFS in detecting large particles and the presence of the M50 alloy, a frequently used alloy for roller bearings in aviation gas turbines. The response for particles in the 25–38 μm range is good. The data analyst can readily detect the presence of large particles, and the alloy can also be identified by its metallurgy. As the particle size increases to the 45–63 μm range, the excitation potential of the spectrometer is insufficient, and only a portion of the particles is vaporized.

TABLE 4—*Conventional spectrometric analysis of M50 alloy particles in MIL-L-23699 oil.*

M50 Particle Size / Vanadium Concentration	Fe	Cr	Ni	Si	Mo	V
25–38 μm / 3 ppm	0.0	0.0	0.0	0.0	0.0	0.0
25–38 μm / 12 ppm	0.9	0.1	0.0	0.0	0.0	0.0
25–38 μm / 20 ppm	1.5	0.1	0.0	0.0	0.0	0.0
45–63 μm / 3 ppm	0.0	0.1	0.0	0.2	0.0	0.0
45–63 μm / 7 ppm	0.1	0.1	0.0	0.0	0.0	0.0
45–63 μm / 15 ppm	0.0	0.0	0.0	0.1	0.0	0.0

TABLE 5—*RFS analysis of M50 alloy particles in MIL-L-23699 oil.*

M50 Particle Size / Vanadium Concentration	Fe	Cr	Ni	Si	Mo	V
25–38 μm / 3 ppm	463	17.5	25.1	15.0	21.2	3.2
25–38 μm / 12 ppm	1454	59.6	86.6	17.5	81.6	12.1
25–38 μm / 20 ppm	3083	157	219	42.9	227	37.1
45–63 μm / 3 ppm	26.9	0.9	1.2	1.4	0.9	0.0
45–63 μm / 7 ppm	21.1	0.6	0.8	2.0	0.7	0.1
45–63 μm / 15 ppm	25.8	0.9	1.0	2.1	0.4	0.3

Microscopic inspection of the RFS electrode as well as an automatic particle counter and shape classifier have been used to verify that the effective RFS capture range of particles from a used oil sample is from 2 μm to more than 100 μm . However, the upper particle size limit that can be completely volatilized by the spectrometer's arc is closer to 50 μm . This does not preclude the RFS analysis from detecting larger particles, because even partial volatilization of a particle above 50 μm will add signal and concentration to the analysis. The purpose of RFS analysis is to detect and trend the presence of large particles in a used oil sample that are missed by routine spectrometric analysis, and not to quantify the exact size or concentration of these

particles. The mere presence of large particles is most significant, and the RFS analysis directs the data analyst to additional analytical tests.

It also may be observed that the RFS technique is very sensitive. Just a few ppm of particles in the oil give a big response by the spectrometer. This is due in part to the RFS filtration process concentrating particles on the disc surface from a relatively large oil volume for subsequent vaporization and detection.

Commercial Case History

The following is a typical case history [8] from a Northeastern power company. The item being sampled is a condensate vacuum pump. In this case, the commercial laboratory provided oil analysis services for lubricant physical properties and metals including RFS for larger particles. Although the laboratory provides data for 20 wear metals, contaminants, and additives, a trend was observed only for iron and silicon (Fig. 6).

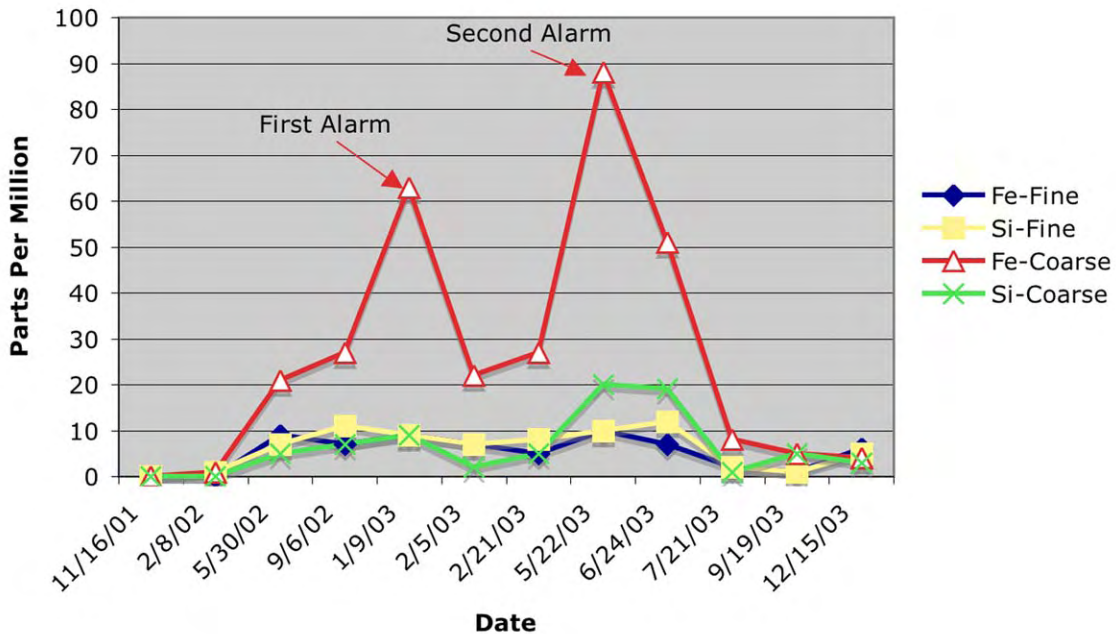


FIG. 6—Spectrometric analysis trends for normal (fine) and RFS (coarse) analyses.

Normal spectrometric analysis, “fine,” did not show significant wear. In most industrial systems such as this pump, concentrations of wear metals will fluctuate in accordance with oil added and can vary by as much as 10 ppm. The wear as indicated by normal spectrometric analysis therefore did not exceed expected limits, and did not result in any maintenance recommendations. On the other hand, the RFS analyses as indicated by “Coarse” in (later in Fig. 8) showed a clear trend resulting in maintenance recommendations.

The silicon readings by themselves were not cause for alarm, but they were when combined with RFS data for iron. The RFS trend for iron jumped enough for the laboratory to issue a severe alarm after the January 9, 2003 sample. The maintenance response was to “feed and bleed,” i.e., drain some lubricant and top it off. The next two RFS analyses showed a corresponding reduction in the iron analyses. However, the May 22, 2003 sample indicated that

there was still a problem, and a severe alarm was again generated. This time the maintenance personnel opened up the pump and found that a bearing had a loose fit and was fretting on the bearing housing, bearing, and shaft (Fig. 7). The RFS data also prompted the laboratory to prepare a ferrogram of this sample. Ferrographic analysis verified that large particles were present and that cutting wear (Fig. 8) was taking place.



FIG. 7—Bearing housing, outer, and inner race fretting due to a loose fit.

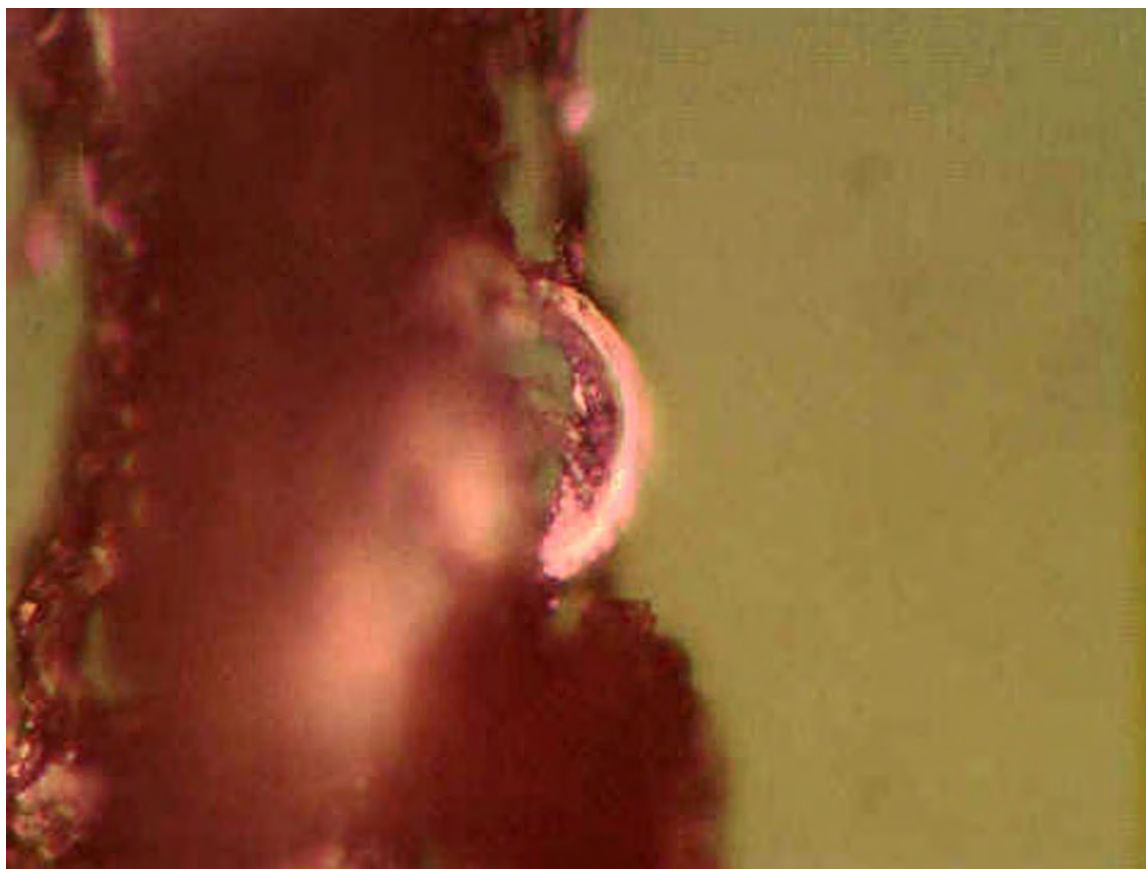


FIG. 8—Ferrogram showing cutting wear.

In this example, normal spectrometric analysis did not detect the large wear particles emanating from the bearing-fretting problem. The RFS analysis, however, did provide early warning to repair, clean up, and solve the problem before it could lead to a major failure with possible secondary damage. The next three samples show a return to a normal trend after the lubricant was cleaned and replaced.

Conclusion

RFS is an important enhancement to standard RDE analysis. It provides new information on the condition of the machine being monitored by expanding the particle detection capability of the RDE technique. Sometimes it is also used to screen samples to determine if more time consuming analysis, such as ferrography, is required.

Rotrode Filter Spectroscopy (RFS) is a recently improved technique that has been shown in actual field applications to provide important additional information about large wear particles, information that may be missed with conventional techniques. It is applied with existing instruments to provide two analyses on the same used oil sample. The first, using the standard RDE atomic emission technique, detects and quantifies dissolved and small wear metals and contaminants. The second analysis, by RFS, qualifies and semi-quantifies larger particles. This paper has shown how the early version of the RFS system has been improved and updated to provide better sensitivity, repeatability, faster sample throughput, and improved particle capture capability.

RFS analysis is not used by itself, but as a second test that expands the particle detection capability of spectroscopy to provide a comprehensive tool to direct the laboratory to identify and solve a maintenance problem. Frequently, the RFS analysis is also used as a screening tool to determine if additional tests, which are too time-consuming or expensive to perform on all samples, should be performed. Supplementary techniques such as analytical ferrography or particle shape characterization and particle counting are then applied to verify a severe wear condition.

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