Particle Counting & Wear Debris Analysis In Used Oil samples

Presented by Bill Herguth

Herguth Laboratories, Inc.
People and Data You Can Count On
Overview

The Problem: Lube oil samples often reveal anomalies in the operating conditions of the machines that are sampled & tested. Further investigation is often required to determine the source and severity of the abnormality, in order to perform “Reasonable Corrective Actions”.

Historically, “further investigation” by an Oil Analysis Laboratory was inadequate. Optical Microscopy / Acid Digested Emission Spectroscopy.

The Solution: The rapid, accurate and cost effective characterization of debris found in lube oil systems. Using sophisticated instruments and software. SEM/AFA
What are Reasonable Corrective Actions

PARTICLE COUNT HIGH

A. Sample Re-tested and Confirmed?
   C. Yes .. Take a Second Sample
   D. Sample Data Now Acceptable?
   E. No… Go to Step “G”
   F. Yes …Bad Sample STOP

G. … Characterize the Severity by Size, Shape, Concentration & Precise Make-up

H. Severe Wear Iron / Brass: Inspect Gears / Bearings for Wear
   Not Severe – Filter System increase sample interval

I. Silicates
   Check Breathers
   Filter Oil
   Re-Sample

B. Not Confirmed, Bad Lab Test STOP
What are Reasonable Corrective Actions

WEAR METALS ARE HIGH

A. Sample Re-tested and Confirmed?

B. Not Confirmed, Bad Lab Test STOP

C. Yes .. Take a Second Sample

D. Sample Data Now Acceptable?

E. No… Go to Step “G”

F. Yes …Bad Sample STOP

G. … Characterize the Severity by Size, Shape, Concentration & Alloys

H. Wear is Severe Compared to Database
   Inspect Gearbox
   Check Vibration
   Increase Sample Interval

I. Wear is Not Severe Increase Sample Interval. Depending on Morphology and make up look for Hematite by X-ray Diffraction
Particle Counting

• Optical Counting ~Prior to 1970

• Light Blockage ~1968 to Present

• Pore Blockage - Flow Decay ~1980 to Present

• LazerNet Fines ~1990 to Present

• Scanning Electron Microscopy with Automated Feature Analysis 2007 to Present
ISO Cleanliness Code 4406:1999

The International Organization for Standardization created the cleanliness code **4406:1999** to quantify particulate contamination levels per milliliter of fluid at three sizes: 4µ[c], 6µ[c], and 14µ[c]. This ISO code is expressed in 3 numbers: 19/17/14. Each number represents a contaminant level code for the correlating particle size. The code includes all particles of the specified size and larger. It is important to note that each time a code increases the quantity range of particles doubles.
### ISO 4406-1999 Code

<table>
<thead>
<tr>
<th>Number of particles per millilitre</th>
<th>Scale Number</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>More than</strong></td>
<td><strong>Up to and including</strong></td>
</tr>
<tr>
<td>2 500 000</td>
<td>&gt;28</td>
</tr>
<tr>
<td>1 300 000</td>
<td>28</td>
</tr>
<tr>
<td>640 000</td>
<td>27</td>
</tr>
<tr>
<td>320 000</td>
<td>26</td>
</tr>
<tr>
<td>160 000</td>
<td>25</td>
</tr>
<tr>
<td>80 000</td>
<td>24</td>
</tr>
<tr>
<td>40 000</td>
<td>23</td>
</tr>
<tr>
<td>20 000</td>
<td>22</td>
</tr>
<tr>
<td>10 000</td>
<td>21</td>
</tr>
<tr>
<td>5 000</td>
<td>20</td>
</tr>
<tr>
<td>2 500</td>
<td>19</td>
</tr>
<tr>
<td>1 300</td>
<td>18</td>
</tr>
<tr>
<td>640</td>
<td>17</td>
</tr>
<tr>
<td>320</td>
<td>16</td>
</tr>
<tr>
<td>160</td>
<td>15</td>
</tr>
<tr>
<td>80</td>
<td>14</td>
</tr>
<tr>
<td>40</td>
<td>13</td>
</tr>
<tr>
<td>20</td>
<td>12</td>
</tr>
<tr>
<td>10</td>
<td>11</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>2.5</td>
<td>9</td>
</tr>
<tr>
<td>1.3</td>
<td>8</td>
</tr>
<tr>
<td>0.64</td>
<td>7</td>
</tr>
<tr>
<td>0.32</td>
<td>6</td>
</tr>
</tbody>
</table>
Optical Particle Counting

ISO 14/12/10
ISO 16/14/11
ISO 17/15/12
ISO 19/17/14
ISO 22/20/17

Source CC Jensen

Draw X# ml of diluted oil through 0.45 micron.

Count representative area

Minimum resolution 5 microns
Light Blockage

**Principle:**
- Obscuration of the light beam by a particle

**Signal Conversion:**
- Each peak corresponds to obscuration by a particle
- Signal height or amplitude reflects the particle size
- Threshold values determine the size bin for the particle
Sample flows through calibrated screen of a known porosity. As particles block the flow, the pressure increases. An algorithm is used to calculate the number and size of particles.

Source: Noria
LaserNet Fines

- All particles greater than 20 μm classified by a trained Neural Network
- Wear or contamination Categories: cutting, fatigue, severe sliding, nonmetallic and fibers
- Air bubbles and water droplets greater than 20 microns eliminated

Source: LNF-Particle Counting and Shape Recognition for In-service Oils
Scanning Electron Microscope (SEM)

Shape: 10 msec/feature

ISO Ratings: 18/16/13 based on equivalent circular diameter

Source National Renewable Energy Laboratory
Innovation for Our Energy Future

Full suspension solution deposited and uniformly segregated on filter membrane

Scan
SEM vs. Automatic Particle Counting

NIST traceable. ISO 12103-A
Medium Test Dust (ISO MTD)

Source Noria
Atomic Emission Spectroscopy
Limitations of Atomic Emission Spectroscopy

Particle size make straight run spectroscopy Semi-quantitative

Estimations

$\leq 8 \text{ Microns}$ 100% Recovery

$> 8 \text{ Microns}$ Recovery Diminishes
Add Strong Acid to dissolve (solubilize) wear debris
Wear Particle Concentration Techniques

Direct Reading Ferrogram

Particle Quantifier
Limitations of Methods

Acid Digestion  Applies to ALL Metals of a Given Size

Direct Reading Ferrography ONLY Measures Iron (fibers cause errors)

Particle Quantifier  ONLY Measures Iron (reliable)
Wear Particle Analysis
WEAR RATES AND MECHANISMS
Wear Particle Analysis
WEAR RATES AND MECHANISMS
Wear Particle Analysis
Silica / Sand
Wear Particle Analysis
Non-Ferrous Bronze? / Aluminum?
Wear Particle Analysis
Rust?/Friction Polymers

Rust
Friction Polymers
SEM/ AFA the Process

1. Extract debris onto filter paper affix to aluminum stubs using carbon conductive tape and placed in the SEM microscope stage.

2. Define locations, focus and analysis parameter.


4. Analyze the data.

5. Report finding.
## Rules Data Analysis

<table>
<thead>
<tr>
<th>Classification</th>
<th>Rule</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass</td>
<td>Cu + Zn &gt;50% and Cu &gt;10%</td>
</tr>
<tr>
<td>Iron Oxide</td>
<td>Fe &gt;30% and O &gt;15%</td>
</tr>
<tr>
<td>Steel</td>
<td>Fe &gt;30%</td>
</tr>
<tr>
<td>Silicates</td>
<td>Si &gt;5%</td>
</tr>
<tr>
<td>Sodium chloride</td>
<td>Na + K + Cl &gt;50%</td>
</tr>
<tr>
<td>miscellaneous</td>
<td>all remaining particles</td>
</tr>
</tbody>
</table>

## Severity

<table>
<thead>
<tr>
<th>RANK</th>
<th>Brass</th>
<th>FeO</th>
<th>Steel</th>
<th>Silicates</th>
<th>NaCl</th>
<th>misc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average all 25 Samples</td>
<td>6.51%</td>
<td>8.79%</td>
<td>25.15%</td>
<td>16.71%</td>
<td>1.32%</td>
<td>7.78%</td>
</tr>
<tr>
<td>STDEV of Data Set</td>
<td>7%</td>
<td>4%</td>
<td>10%</td>
<td>9%</td>
<td>1%</td>
<td>6%</td>
</tr>
<tr>
<td>Avg. + STDEV OK</td>
<td>13.17%</td>
<td>13.02%</td>
<td>35.25%</td>
<td>26.19%</td>
<td>2.74%</td>
<td>13.31%</td>
</tr>
<tr>
<td>Avg. +2 X STDEV WARNING</td>
<td>19.82%</td>
<td>17.25%</td>
<td>45.36%</td>
<td>35.66%</td>
<td>4.16%</td>
<td>18.83%</td>
</tr>
<tr>
<td>Avg. + 3 X STDEV ALARM</td>
<td>26.48%</td>
<td>21.48%</td>
<td>55.46%</td>
<td>45.14%</td>
<td>5.59%</td>
<td>24.36%</td>
</tr>
</tbody>
</table>
# High Brass

## High Brass Particle Classification

<table>
<thead>
<tr>
<th>Particles</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass</td>
<td>27.20%</td>
</tr>
<tr>
<td>FeO</td>
<td>6.20%</td>
</tr>
<tr>
<td>Steel</td>
<td>21.80%</td>
</tr>
<tr>
<td>Silicates</td>
<td>6.80%</td>
</tr>
<tr>
<td>NaCl</td>
<td>0.60%</td>
</tr>
<tr>
<td>misc</td>
<td>19.20%</td>
</tr>
<tr>
<td>Zinc</td>
<td>6.6%</td>
</tr>
<tr>
<td>Calcium</td>
<td>6.6%</td>
</tr>
<tr>
<td>Silicates</td>
<td>6.8%</td>
</tr>
<tr>
<td>KCl</td>
<td>5.0%</td>
</tr>
<tr>
<td>NaCl</td>
<td>0.6%</td>
</tr>
</tbody>
</table>

![High Brass Particle Classification Diagram](image)
Report Images & Analysis

Copper
Zinc
## High Steel

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass</td>
<td>1.80%</td>
</tr>
<tr>
<td>FeO</td>
<td>8.60%</td>
</tr>
<tr>
<td>Steel</td>
<td><strong>44.40%</strong></td>
</tr>
<tr>
<td>Silicates</td>
<td>22.20%</td>
</tr>
<tr>
<td>NaCl</td>
<td>1.20%</td>
</tr>
<tr>
<td>misc</td>
<td>2.20%</td>
</tr>
</tbody>
</table>

### High Steel Particle Classification

- **Steel**: 44.4%
- **Silicates**: 22.2%
- **Brass**: 1.8%
- **Zinc**: 2.2%
- **FeO**: 8.6%
- **KCl**: 9.2%
- **NaCl**: 1.2%
- **misc**: 2.2%
- **Calcium**: 8.2%
Report Images & Analysis

Iron (Fe)
# High Silicates

## High Silicates Particles by Classification

<table>
<thead>
<tr>
<th></th>
<th>Brass</th>
<th>FeO</th>
<th>Steel</th>
<th>Silicates</th>
<th>NaCl</th>
<th>misc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>2.80%</td>
<td>5.80%</td>
<td>10.60%</td>
<td>36.80%</td>
<td>0.20%</td>
<td>4.80%</td>
</tr>
</tbody>
</table>

![Pie Chart showing the composition of high silicates](chart.png)
Report Images & Analysis

Silicon
### Total Particles by Equivalent Diameter

<table>
<thead>
<tr>
<th></th>
<th>&lt;1um</th>
<th>1-2um</th>
<th>2-3um</th>
<th>3-4um</th>
<th>4-6um</th>
<th>6-10um</th>
<th>10-14um</th>
<th>14-21um</th>
<th>21-25um</th>
<th>&gt;25um</th>
<th>Total</th>
<th>%&lt;4um</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>8595</td>
<td>20436</td>
<td>14185</td>
<td>8182</td>
<td>6652</td>
<td>3144</td>
<td>669</td>
<td>355</td>
<td>77</td>
<td>79</td>
<td>62374</td>
<td>82.4%</td>
</tr>
</tbody>
</table>

### Aspect ratio >2

<table>
<thead>
<tr>
<th></th>
<th>Brass</th>
<th>Zinc</th>
<th>FeO</th>
<th>Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>34.1%</td>
<td>21.2%</td>
<td>42.2%</td>
<td>42.1%</td>
</tr>
</tbody>
</table>

### Aspect ratio >4

<table>
<thead>
<tr>
<th></th>
<th>Brass</th>
<th>Zinc</th>
<th>FeO</th>
<th>Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>5.5%</td>
<td>0.0%</td>
<td>11.0%</td>
<td>10.1%</td>
</tr>
</tbody>
</table>

- **Iron + Sulfur** (Iron Sulfide)
- **Iron + Oxide**
- **Oil Additive**
## Particle Definitions (41 Categories are available)

<table>
<thead>
<tr>
<th>Name</th>
<th>Unit</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dave</td>
<td>µm</td>
<td>The average length of the sixteen chords through the feature centroid</td>
</tr>
<tr>
<td>Dmax</td>
<td>µm</td>
<td>The length of the longest of the sixteen chords through the feature centroid</td>
</tr>
<tr>
<td>Dmin</td>
<td>µm</td>
<td>The length of the shortest of the sixteen chords through the feature centroid</td>
</tr>
<tr>
<td>Dperp</td>
<td>µm</td>
<td>The length of the chord perpendicular to the longest chord (Image #3)</td>
</tr>
<tr>
<td>ASPECT RATIO</td>
<td>1</td>
<td>The ratio of DMAX/DPERP</td>
</tr>
<tr>
<td>AREA</td>
<td>µm²</td>
<td>The area of the feature</td>
</tr>
<tr>
<td>PERIMETER</td>
<td>µm</td>
<td>The perimeter of the feature as measured from one chord end to the next</td>
</tr>
<tr>
<td>ORIENTATION</td>
<td>Degrees</td>
<td>The orientation of the longest chord. Zero is at and the angle increases clockwise</td>
</tr>
</tbody>
</table>
Conclusion

• Oil analysis is an effective tool for monitoring wear rates and contamination in most machines.

• Once data indicates an abnormal condition, further investigation is often warranted in order to take reasonable corrective action.

• SEM/AFA is a rapid, efficient and cost effective method of determining reasonable corrective action.

THANK YOU ... for your kind attention.