Image Analysis in the Field of Oil Contamination Monitoring

Examensarbete utfört i Bildanalys vid Tekniska högskolan vid Linköpings universitet av

Ema Ceco

LiTH-ISY-EX--11/4467--SE

Linköping 2011
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Monitoring wear particles in lubricating oils allows specialists to evaluate the health and functionality of a mechanical system. The main analysis techniques available today are manual particle analysis and automatic optical analysis. Manual particle analysis is effective and reliable since the analyst continuously sees what is being counted. The drawback is that the technique is quite time demanding and dependent of the skills of the analyst. Automatic optical particle counting constitutes of a closed system not allowing for the objects counted to be observed in real-time. This has resulted in a number of sources of error for the instrument.

In this thesis a new method for counting particles based on light microscopy with image analysis is proposed. It has proven to be a fast and effective method that eliminates the sources of error of the previously described methods. The new method correlates very well with manual analysis which is used as a reference method throughout this study. Size estimation of particles and detection of metallic particles has also shown to be possible with the current image analysis setup. With more advanced software and analysis instrumentation, the image analysis method could be further developed to a decision based machine allowing for declarations about which wear mode is occurring in a mechanical system.

Keywords: contaminants in oil, counting particles, image analysis, oil condition monitoring, sizing particles
Abstract

Monitoring wear particles in lubricating oils allows specialists to evaluate the health and functionality of a mechanical system. The main analysis techniques available today are manual particle analysis and automatic optical analysis. Manual particle analysis is effective and reliable since the analyst continuously sees what is being counted. The drawback is that the technique is quite time demanding and dependent of the skills of the analyst. Automatic optical particle counting constitutes of a closed system not allowing for the objects counted to be observed in real-time. This has resulted in a number of sources of error for the instrument.

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Last but not least, I owe my deepest gratitude to my family for always motivating and supporting me.

Ema Ceco
Linköping 2011
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Abbreviations

ANN Artificial neural network
CCD Charge coupled device
EDS Energy dispersive spectroscopy
EDM Euclidean distance map
FLT Fuel and lubricant testing
ICP Inductively coupled plasma mass spectroscopy
IMPACT Image analysis particle counting
ISO International standard organisation
MAT Medial axis transform
PDMS Polydimethylsiloxane
RGB Red, green, blue
SEM Scanning electron microscopy
SIT Siemens industrial turbomachinery
Preface

The importance of oil condition analysis can be explained by a simple example. Oil in a mechanical system can be resembled to blood in a human being. If something is suspected to be wrong, the first step to remedy is to take a blood sample from the person, analyze the blood constituents and finally evaluate possible anomalies. The principle for a mechanical system is the same; oil samples, instead of blood, are tapped, preferably on a regular basis, in order to detect and foresee deviations. Background information about the mechanical system is, just as in the medical case, crucial since different systems tend to evolve characteristically depending on oil and system type. Oil from a gearbox normally shows significantly more contamination than oil from a turbine system. The difference lies in the extent of which the system is closed or open and the mode of interaction between components. In order to perform analyses tailored for the system at hand, this information is essential.

The methods for lubricant analysis are many. No one method alone holds the key of all information sufficient to make a reliable diagnosis of a mechanical system. The most commonly used methods today complement each other; where one has its limits another one takes by. One analysis that is regularly performed on most types of oil is particle counting. Particle counting lays the foundation for this Master’s thesis.

Welcome to the exciting world of oil condition monitoring with image analysis!
Chapter 1

Introduction

Monitoring wear particles in lubricating oils allows specialists to evaluate the health and functionality of a mechanical system. It is also a great indicator of when it is time to change oil in a system. The following studies are based on lubricating oils kindly provided by the staff at the oil and gas division at Siemens Industrial Turbomachinery (SIT), in Finspång. The oil, tapped from a testing rig for gas turbines, is sent to the Fuel and lubricant testing (FLT) division of Exova, an analysis institute specialized on fuel and lubricant condition monitoring.

Testing rigs are used to confirm functionality and quality of gas turbines before they are shipped out for final assembly on end-point location. One gas turbine is online at the testing rig for 2-3h in order to generate valid guidance data. About 20\(m^3\) of lubricate is during this time continuously pressed through the system. To ensure long, trouble-free operation in the test rigs and to control quality of the gas turbines it is necessary to, among other analyses, monitor contaminant levels in the oil [1]. Contaminants in oil are roughly defined as particles that were not present when the oil was added fresh to the system [2]. Throughout this thesis work a novel method for acquisition of quantitative data of particle contaminants in lubricating oils was developed. The method is based on image analysis aided particle counting. The system will from here on be referred to as IMPACT (IMage analysis PArticle CounTing).

1.1 Purpose

Today, manual and optical counting are employed at FLT, Exova in order to quantify the amount of particles in oil from the effects of wear, misuse and outer contaminants. Manual particle counting in particular is a very time consuming process. It requires a lot of experience and practice to be executed according to standards ISO 4406 [3] and ISO 4407 [4]. An analysis of a single sample can take up to three hours to conduct. Because of the immense amount of time spent on counting particles, this method is quite costly why it is usually rather avoided. Only customers with very sensitive and expensive systems use the analysis technique. These customers cannot afford having certain contaminants in their systems
contributing to system failure. The later method, automatic optical particle counting, is stable and fast. It is based on the principle of light blockage [5]. That is, whenever a particle in the flow cell passes the illuminating laser beam, a scatter pattern is sent to the detector surface (Figure 1.1). The detector relates the size and shape of the pattern to a voltage output [6].

![Figure 1.1. Automatic optical particle counter. Image borrowed from [7]](image)

The issue with the automatic optical quantifier is that one cannot be sure what is being measured since it is a closed system. Oil samples containing air bubbles have been studied to generate results slightly higher than the true value [6]. Samples containing soot have shown to be troublesome since soot tends to block the laser beam, generating non-reliable data [6]. Additives in oil have proven to generate differing values as well [8].

In order to be able to offer purity analysis of oil cheaper, faster and to a wider clientele, a new method based on microscopy with image analysis has been developed and implemented. In the long run, the hope is that more customers will be able to afford the analysis. From Exova’s point of view, the ability to profile themselves with a wider, more affordable variety of services, will result in more competitive solutions for the company.

### 1.2 Objective

The work presented in this thesis aims to optimize, validate and compare a new method for quantifying particles at FLT, Exova. The new method based on optical microscopy and image analysis is expected to be able to

- Quantify particles in oil larger than 4µm
1.3 Outline

- Separate particles into different size classes
- Separate and quantify fibres
- Separate and quantify metallic particles

The validation will follow the guidelines of standards ISO 4406 [3] and ISO 4407 [4] in order to acquire results comparable to manual analysis, which is used as a reference method throughout this study. Oils from gas turbines are relatively clean from contaminants. Gas turbine oils are normally expected to last for a long time, sometimes reaching up to 20 years [1]. IMPACT will not be limited to gas turbine oils, although, at this initial stage, it will be validated and verified using oils from a gas turbine testing rig.

1.3 Outline

This thesis is divided into three main parts, Theory, Experimental, and finally Discussion and Conclusions. The reader will in the first few chapters (Chapters 2-3) be introduced to fundamental concepts of tribology and image processing which are important for the comprehension of the following study. In Chapter 4 the imaging system is presented which concludes the Theory part. The following chapters, the Experimental part, will treat the work method for IMPACT (Chapter 5), validation of the method (Chapter 6), and a comparison with automatic optical particle counting (Chapter 7). Finally in Discussion and Conclusions, the study will be summed up with a discussion (Chapter 8) and conclusions (Chapter 9) of the model at hand, topping it off by presenting possible improvement areas for the future (Chapter 10).
Part I

Theory
Chapter 2

Tribology

“...the science and technology of interacting surfaces in relative motion and of related subjects and practices...”

*Peter Jost et al.*
Government Report, March 9, 1966 [9]

This chapter aims to introduce the term tribology. First the reader will gain knowledge about some background and basic concepts in tribology. Later sections will briefly discuss different parts of tribology, namely friction, wear, and lubrication which are the founding elements for understanding how wear particles are generated in lubricating oils. Wear is in this thesis the central concept for the formation of contaminants in oil. There are nevertheless other sources of contamination like poor handling procedures or seal failure.

The term tribology was coined by Peter Jost in 1966 [9] as he and his colleagues saw a lack of a cohesive term, describing a field of study that many before him, e.g. Leonardo da Vinci, Euler, Coulomb, and other great scientists, had engaged themselves in and devoted their lives to [10]. The word tribology is derived from the Greek word “tribos” which simply means “rubbing”[11]. Bluntly, one might say that without tribology no motion would occur. Interestingly enough, in the field of tribology one, in general, cannot account for or predict the outcome of friction or wear in a system. Since this is a multidisciplinary field of science, mechanics, physics, chemistry, and metallurgy are all essential for explaining tribological phenomena [10].

2.1 Friction

“...the resistance to motion that arises from interactions of solids at the real area of contact.”

*W.F. Gale and T.C. Totemeir*
Smithells Metals Reference, 2004 [12]
It is estimated that a third of all energy spent in the world today is used to overcome frictional problems. As a result, 200 billion dollars are spent annually in the U.S. on necessary material replacements and increase in fuel consumption in mechanical systems [11]. It is by the action of friction that many wear particles are formed. Oils are conditioned and optimized to minimize frictional forces. If a lubricant on the other hand has an advanced ongoing ageing process, it may no longer withstand the environment under which it is operating. At that point extensive frictional tendencies might prevail resulting in wear particles which will increase both in size and quantity.

2.2 Wear

“The progressive loss of substance from the surface of a solid body due to mechanical action, i.e. the contact and relative motion of a solid, liquid or gaseous counterbody.”

W.F. Gale and T.C. Totemeir
Smithells Metals Reference, 2004 [12]

The definition of wear indicates that it is an ongoing mechanical and chemical process between two interacting surfaces. The system which this thesis will focus on, a gas turbine system, mainly consists of metal alloys in close interaction. Wear is conveniently divided in two subsections, mechanical wear and oxidative wear [13]. Oxidative wear is a product of reoccurring chemical and electrochemical reactions. Oxidative and mechanical wear are often synergistic processes. Where mechanical wear takes place, the chance for oxidative wear is significantly increased and the other way around [14]. A simple example can explain this train of thought; a rust protective layer in a system is gradually torn off by mechanical wear. This leaves unprotected areas, favourable for electrochemical reactions, i.e. oxidative wear, to transpire. Mechanical wear is what is most often thought of when speaking about wear. It is affected by a couple of mechanisms where wear particles are often formed. By these wear mechanisms; abrasion, fatigue, and, adhesion, mechanical wear can be further subdivided [13] (Figure 2.1).

2.2.1 Abrasive wear

Abrasive wear is often considered the most critical type when studying industrial problems. There are two different types of processes generating abrasive wear, two-body abrasion and three-body abrasion. Two-body abrasion appears when a harder surface slides over a softer surface and chops away parts of it. Three-body abrasion covers the case when free material or particles are present between the two opposing surfaces, e.g in the lubricant, causing damage to them [10]. Another type of three-body abrasion is caused by erosion. Erosion is a result of high flow of particles or liquids towards a surface continuously removing material from it. This
2.2 Wear

type of wear is consequently affected by heightened particle levels in lubricants. If the particle concentration is high, erosion can become an issue in a system. Erosive wear is not only dependent on the velocity of the liquid or particles but also on the particles stiffness, shape and angle of impact [10]. Typical particles formed by erosion can be found in Table 2.1. Erosive wear reveals itself in many mechanical applications, an example being at the blades of gas turbines caused by dust clouds in the air [14]. Severe types of cutting wear are produced during abrasion. The particles are in the size range of $25\mu m$ and are therefore serious when found in lubricants (Table 2.1). Laminar particles are also caused by erosive wear, e.g. when particles intrude a rolling contact. Particles resulting from abrasive wear processes have a characteristic appearance that can be seen in Table 2.1. Rubbing abrasive wear are the particles that are normally found in circulating lubricants. Cutting abrasive wear is the particle that raises the most concern if detected in a lubricant system. A few particles detected are not that alarming but if the amount raises up to hundreds, there is reason for proactive measures. Laminar wear particles may also be a result of abrasive wear in the system.

2.2.2 Fatigue wear

Surfaces in close contact always exert a certain amount of force on each other. Fatigue is associated with cyclic loading meaning that the surface experiencing the force is loaded and unloaded continuously in cycles which causes stress to the material. If the stress level exceeds a certain level repeatedly, cracks will occur near the surface which causes deformation of the material. Spherical particles are smaller than $5\mu m$ and may be a cause of fatigue wear. Other spherical particles that may be present in the lubricant may instead be derived from oxidative wear. Also caused by fatigue wear are fracture particles. Fracture particles are up to $100\mu m$. They are alarming when found in lubricants because of their size (Table 2.1).

2.2.3 Adhesive wear

Finally, adhesive wear occurs where two surfaces slide or are pressed towards each other and friction occurs. The process is adhesive meaning that asperities from one of the surfaces attract or tear away fragments from the other surface by atomic and molecular forces. If the particles torn away do not attach to the attracting surface enough they can be flushed away by the lubricant [10]. Adhesive particles have a unique appearance which can be found in Table 2.1. Sliding adhesive wear is commonly found in lubricants and does in general not result in alarming actions if detected in oil. It is characterized by particles in size range, $20 - 50\mu m$ (Table 2.1).
2.3 Lubrication

“Lubricants minimize friction and wear in rubbing contacts by reducing metal-metal contact, removing wear debris and carrying away frictional heat.”

_W.F. Gale and T.C. Totemeir_  
Smithells Metals Reference, 2004 [12]

Reducing friction in a mechanical system is the main reason for introducing a lubricant. The friction, hence the energy losses, are decreased dramatically after introduction of a lubricant. Apart from reducing friction, the lubricant also provides the metal surfaces with a protective layer against wear and corrosion, and conveys heat generated during operation. A lubricant can be a liquid (oil most often), a grease, a solid material, or a gas.

### 2.3.1 Lubricating oils

The majority of lubricants used today are oils. Oils constitute of long hydrocarbon chains. They are separated into three main classes; mineral oils, synthetic oils and biological oils. Apart from the hydrocarbon chains, the oil is usually blended with additives for performance enhancement. Throughout this thesis a mineral oil is studied and evaluated. Though, both mineral and synthetic oils may be used as turbine oils.

**Mineral oils**

Mineral oils are the most common type of lubricant used. They are extracted from crude oil which is available all over the world. Crude oil is distilled by fractional distillation in high towers, i.e. the oil is separated into constituents, fractions, depending on their respective boiling points, see Figure 2.2. Trays are placed at different heights in the distillation column enabling separation of fractions by con-

![Figure 2.1. Wear mechanisms, modes, and particle types. Remake of figures in [13] and [15].](image)
2.3 Lubrication

Table 2.1. Common particles in lubricating oils. Remake of figures in [16] and [17].

<table>
<thead>
<tr>
<th>Particle type</th>
<th>Profile and edge</th>
<th>Colour</th>
<th>Composition</th>
<th>Texture</th>
<th>Size [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing</td>
<td>Golden, partly dark or black</td>
<td>Cu, (Zn) Fe, (Ni)</td>
<td>Thin and smooth</td>
<td>&lt; 15</td>
<td></td>
</tr>
<tr>
<td>Cutting</td>
<td>Golden, bright yellow, Fe, (Ni) Cu, (Zn) Sn, Sb, Cu</td>
<td>Black dots</td>
<td>25 – 100 (1:9)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laminar</td>
<td>Golden, bright yellow</td>
<td>Cu, (Zn) Fe, (Ni)</td>
<td>Flat particles</td>
<td>20 - 50</td>
<td></td>
</tr>
<tr>
<td>Sliding</td>
<td>Dark, golden, bright yellow Fe, (Ni, Cr) Cu, (Zn) Fe</td>
<td>Sliding marks</td>
<td>20 -50, &gt; 50</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fatigue</td>
<td>Bright, golden, black with dots Fe, (Ni) Cu, (Zn) Sn, Sb, Cu</td>
<td>Thick, irregular shape</td>
<td>&lt; 100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spherical</td>
<td>Bright or brownish, dark, golden Fe, (Ni) Fe, (Ni) Cu, (Zn)</td>
<td>Smooth, sliding marks</td>
<td>&lt; 10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fibre</td>
<td>Transparent, bright yellow</td>
<td>Cellulose</td>
<td>Polymer, long, branched</td>
<td>&gt; 100 (1:10)</td>
<td></td>
</tr>
</tbody>
</table>

densation. Depending on the fractional volatility, different fractions will condense at different heights in the column. The most volatile fraction will condense at the highest tray while the least volatile compound will condense at the lowest. Fractions which are commonly extracted by fractional distillation are found in Figure 2.2. Depending on the origin of the crude oil, mineral oils differ from each other in viscosity, chemical form, and sulphur content. The backbone in all oil types is a hydrocarbon chain. Depending on the functional groups prevalent in the oil, it is categorized by the three classes described next.
Paraffinic oils - constituting of hydrocarbon chains which may be branched in numerous ways

Naphthenic oils - composed of hydrocarbon chains which also contain cyclic hydrocarbon groups

Aromatic oils - containing unsaturated cyclic hydrocarbon formations in the hydrocarbon chain

The lengths of the hydrocarbon chains determine the internal tendency to self-entangle, i.e. the viscosity of the oil. More about viscosity will follow in a Section 2.3.2. Sulphur content in mineral oils is also dependent of the origin of the oil. It is preferable to have approximately 1% sulphur content in the mineral oil as it enhances the lubricants' ability to withstand wear. Higher amounts of sulphur may on the other hand favour oxidative processes [14].

Figure 2.2. Crude oil fractioning in a distillation column. Image borrowed from [18].

Synthetic oils

Synthetic oil is chemically produced in a highly controlled environment for specialized applications. It is a substitute for mineral oil and can be modified to contain certain functional groups which enhance the oils’ performance. Synthetic
oils are manufactured by cracking of petroleum, meaning that petroleum is refined into more well-defined, smaller fragments of hydrocarbons. These fragments are then allowed to polymerize and produce lubricants with the appropriate attributes. The reaction can be directed in a couple of different manners depending on the oil requirements of the oil, e.g. it can be carried out in the presence of halogens or catalysts. Halogenation, produces lubricants appropriate for use in fire hazardous environments, while silanization in the presence of catalyst results in lubricants that are liquid over a longer temperature span than others. This can be useful under high temperature operation [14].

2.3.2 Viscosity

“...a fluid's resistance to flow and is primarily a consequence of the internal friction of the fluid.”

G. E. Totten
Fuels and lubricants handbook: technology, properties, performance, and testing, 2003 [19]

In lubricating oils, viscosity is the determinant factor of the oils' ability to reduce friction and energy dissipation, and to withstand corrosion and wear. In general in lubricating oils, the longer the hydrocarbon chains forming the oil, the higher the viscosity of the oil. The hydrocarbon molecules, per se, do not interact with each other to any greater extent. Only weak physiochemical forces are present between the molecules. The length of the hydrocarbons on the other hand, is a great factor affecting the viscosity of the fluid. Long hydrocarbon chains randomly entangle rendering higher resistance to motion, in other words higher viscosity. This can be compared to water molecules which are small and compact with no room for entanglement, hence the lack of viscous properties. Viscosity can be described with a simple example. Suppose you feel the urge for a cup of tea with honey. You place your tea bag in your cup and pour boiling water over it. Water is a thin liquid which flows without any resistance and reaches the cup almost instantly. Then you grab a spoon full of honey and let it pour down into the cup. It takes longer time for the honey to reach the cup. Honey is a thick liquid with an internal resistance to flow. It is a viscous fluid. The fluidity of a viscous fluid increases with temperature. That is why it is important to consider the operation conditions in a device when choosing an appropriate lubricant. Gas turbines, having relatively high operation temperature, might need oils with viscosity enhancing additives to maintain the viscous properties over a longer temperature span. Viscosity is also important from the point of view of counting particles. The higher the viscosity of the oil, the slower the sedimentation of particles inside the lubricant. Slow sedimentation is favourable since it allows for accurate statistical counting of particles in lubricating oils [14].
2.3.3 Additives

Additives are, as mentioned earlier, used as performance enhancers in lubricating oils. Usually the term base oil is used for lubrication oils before any adherence of additives. There is a wide selection of different types of additives and additive packs specialized for oils with various areas of application. There are some general types of additives, added to most base oils to improve properties like viscosity and the ability to withstand oxidation, while others, like extreme pressure additives and anti-foam agents, are used in systems operating under special conditions. [14] In this thesis, additives prevalent in circulating turbine oils will be considered. Gas turbines usually operate under special conditions where the additives in the lubricant hold certain requirements. According to Rudnick et al. [20] they need to

- Improve bearing lubrication.
- Convey heat through circulation.
- Serve as hydraulic fluid for governor and other equipment.
- Lubricate reducing gears.
- Prevent corrosion.
- Allow rapid separation of water from the oil.
- Resist foaming.
- Resist oxidation.

In Table 2.2 a handful of additives used in circulating turbines oils are described. As can be seen, all requirements are covered by the presented additives. Additives are often synergistic chemicals, enhancing one another. One issue in newly produced turbines is that residues of industrial oil may be left in the system after production [8]. When different types of oils are mixed together in a system it may sometimes not be a favourable combination. Zinc and calcium are common constituents of additives in industrial oil, in the form of calcium sulphonate and zinc sulphonate. These metallosulphonates are highly reactive with acidic residues in anti-oxidation/extreme pressure (EP) and anti-rust additives which are frequently used in turbine oils. When reacted, an insoluble salt normally precipitates [20]. Wanke and Michael [8] showed in 2008 that by adding different types of additives to base oil, certain additives heighten the contaminant levels in the base oil. The study was performed to evaluate the effect of additives in automatic optical particle counters. Polydimethylsiloxane (PDMS), an anti-foam additive, was proven to cause the greatest deviations [8]. Anti-foam additives function by forming micellar structures in the oil, preventing it to foam at the air/oil interface [8]. The micellar structures are in $4 - 10\mu m$ size range.
2.3 Lubrication

Table 2.2. Common additives used in circulating turbine oils. Remake of table in [20].

<table>
<thead>
<tr>
<th>Type of Additive</th>
<th>Additive compound</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Antioxidant</td>
<td>Diaryl amines</td>
<td>Controls free radicals and terminates radical reactions.</td>
</tr>
<tr>
<td></td>
<td>Hindered phenols</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Organic sulfides</td>
<td></td>
</tr>
<tr>
<td>Rust inhibitor</td>
<td>Alkylsuccinic acid derivatives</td>
<td>Creates a protective film by adsorbing polar constituents on metal surfaces.</td>
</tr>
<tr>
<td></td>
<td>Ethoxylated phenols</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Imidazoline derivatives</td>
<td></td>
</tr>
<tr>
<td>Foam inhibitor</td>
<td>Polydimethylsiloxanes</td>
<td>Alters the surface tension of lubricants and facilitates separation of air bubbles which retards foam formation.</td>
</tr>
<tr>
<td></td>
<td>Polyacrylates</td>
<td></td>
</tr>
<tr>
<td>Metal deactivator</td>
<td>Triazoles</td>
<td>Form inactive film on metal surfaces by joining with metallic ions (e.g. iron and copper).</td>
</tr>
<tr>
<td></td>
<td>Benzotriazoles</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2-Mercaptobenzothiazoles</td>
<td></td>
</tr>
<tr>
<td>Mild antiwear/</td>
<td>Alkylphosphoric acid esters</td>
<td>Reacts with metals to form films of lower shear strength than the metals, thereby preventing metal-to-metal contact.</td>
</tr>
<tr>
<td>Extreme pressure</td>
<td>and salts</td>
<td></td>
</tr>
<tr>
<td>additive</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Demulsifier</td>
<td>Polyalkoxylated phenols</td>
<td>Enables fast water separation from oil by promoting coalescence of water droplets.</td>
</tr>
<tr>
<td></td>
<td>Polyalkoxylated polyols</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Polyalkoxylated polyamines</td>
<td></td>
</tr>
</tbody>
</table>

2.3.4 Contaminants in lubricating oils

In gas turbines, oil circulates through a lubrication system. The circulation time for the lubricant studied in this thesis is about 7 minutes which is relatively fast considering the large volume of lubricant travelling through the gas turbine. In the circulation system, the oil passes a filter system that filters it properly and catches wear debris. One can imagine that this first test-run in the testing-rigs is very important for turbines fresh off production lines, as there still might be fragments and particles left in the system from production. In the early stages of device operation an increase in particle concentration in the lubricant is often observed [13]. This is called the running-in period which is an acceptable development where a stable wear regime is established in the system (Figure 2.3). Later leaps in the rate of particle contaminants are not acceptable and may instead be indicating wear out. In Figure 2.3 the relationship between particle size and number of particles
present in the oil during different stages of operation are presented. During the first period, the running-in period, there are a lot of small particles present in the oil. If the system should begin to fail, an increase of both particles and size would take place.

![Graph showing particle quantity and size dependence of operation time](image)

**Figure 2.3.** Particle quantity and size dependence of operation time. Adaptation of graph from [21].

It is difficult to decide what a normal rate of increase of particle contamination is as it depends on several different factors e.g. device material, lubricant, operating temperature, but also on the surrounding environment. In order to be able to distinct significant changes in the wear rate, the most effective method is to continuously trend the contaminant concentration levels. Apart from contaminants caused by wear there are other types of particles e.g. fibres and sand which might be found in lubricating oils. Fibres most often originate from defective filters while sand is a typical contaminant caused by poor handling of the lubricant.
Chapter 3

Image analysis in the field of particle monitoring

“Distinguishing image processing from image analysis is easy. Image processing always uses an image as an input, and the result of each of its functions will be an image. On the other hand, image analysis will also use an image as input (this can be a gray, color, or binary image), but the output is always numbers (single or multiple, gray value, color value, area or perimeter of a particle, etc.).”

Dr. Wolf Malkusch
Quantitative Image Analysis Methods and Limitations, 2002 [22]

Today advanced image analysis systems can identify a number of different particle properties e.g. quantity, form, edge detail, size, color, ratio and reflectivity [23]. By combining these properties, often using artificial neural networks [24], fuzzy logic [25], or other knowledge based systems [26], advanced image analysis software can conclude which types of particles are present in an oil sample. Thereby, qualitative data, as well as quantitative, are possible to extract. As mentioned in Chapter 2 different types of wear particles originate from different kinds of wear processes. The possibility of determining the origin of wear particles present in a lubricant helps experts to decide which type of wear processes are ongoing in a mechanical system. In this chapter, image processing procedures used to quantify particles as well as extract qualitative data describing form, edge detail, size, color, and reflectivity of the particles will be treated. Most of the chapter is based on the findings of John C Russ in his book The image processing handbook [27]. Where no other references are found the reader is referred to this book.

3.1 Image preprocessing

The reason why image enhancement is executed before extracting desired information is simple; well-defined raw data are more feasible for further analysis. The
raw input signal from a camera sometimes results in images with somewhat blurry edges (Figure 3.1a). In particle analysis the main concern might be blurriness of the particles' edges. To remedy this issue a Laplace filter is applied to the signal (Figure 3.1b). The Laplace filter is a so called edge enhancement filter, see for example [28]. Briefly it can be described as providing sharper or faster transitions between background and object. In the filters attempted to be analyzed in this thesis, irregularities from the filter pores prevail in 200x magnification. These pore structures might in some cases become a source of error since they take on low intensities as opposed to the rest of the filter which is often white. The lowest intensities of the filter pores sometimes correspond to the brightest intensities of the particles. As we apply a Laplace filter to enhance the edges of particles, the pore structures become slightly more visible as well. This turns out to be a problem when an appropriate threshold needs to be set. A possibility is to use an averaging filter, see for example [28], which is a local pre-processing method (Figure 3.1c). It is used to suppress image noise. When both filters are applied simultaneously (Figure 3.1d), the Laplace filter enhances the particle edges while the averaging filter smoothens out the background. The result is an enhanced image, more suitable for further analysis.

**Figure 3.1.** a) Original image b) Image sharpening with a 5x5 Laplacian. c) Image processed with a 3x3 average filter. d) Combining these filters results in well-defined particle boundaries easier to threshold.
3.2 Segmentation

Segmenting objects for analysis might be the most critical step of the image analysis. The particles to be analyzed throughout this thesis work are in the size range of $> 4\mu m$ meaning that the smallest ones are quite difficult to capture. The magnification of the microscope needs to be 200x to ensure sufficient enhancement of the smallest objects. In this size range, the optical microscope has some limitations like halo and distortion effects which limit the lower detection range [22]. In order to segment interesting particles from background a manual step needs to be conducted. Thresholding may be performed manually by choosing the minimum between two intensity peaks in a histogram. A histogram presents the number of pixels representing a certain intensity, see Figure 3.2a). Our filters, being white,

![Figure 3.2](image)

**Figure 3.2.** The histogram in a) is easy to threshold since both background and intensity peaks are well defined. The objects in b) are very small relative to the background, why no peak originating from the objects can be studied.

will result in a peak with brighter pixel intensities, close to 255 (white), while the particles, often being black, will result in darker intensities closer to 0 (black). Manual thresholding cannot be executed solely based on the histogram of the image since the proportion of background area to particle area is very high when analyzing filters. This results in a large peak of the background intensities while the intensities of the particles are quite few resulting in a tiny top when compared to the background, see Figure 3.2b). A method, called half-amplitude thresholding, described in ISO 13322-1:2004 [29], is used throughout this thesis. The half amplitude method is a tool for setting a manual threshold since the software PicEd Cora does not include automatic thresholding. Half amplitude thresholding is performed by selecting a region just outside the boundary of a representative object. A threshold is set so that half of the pixels in the region are segmented or thresholded. The same procedure is performed just inside the visible boundary of the desired object. The average of these two values is selected as a threshold in the image. To improve the accuracy of the thresholding this procedure can be repeated for a couple of representative particles and the average of the results can be selected as a global threshold of the image [29]. The image is now binary, the particles represented by the value 1 whilst the background is labelled 0. In Figure 3.3, the pre-processed particle from Figure 3.1 is thresholded and the binary
image analysis in the field of particle monitoring

image is superimposed in yellow over the original image in Figure 3.1d). The line in Figure 3.3 shows the maximum Caliper dimension described further in Section 3.3.2.

![Image](image.png)

**Figure 3.3.** Object segmented with half amplitude thresholding.

### 3.3 Image analysis

#### 3.3.1 Quantification

In order to count the number of particles which have been segmented in the picture, i.e. objects that hold a value of 1, labelling is executed. There are a number of different algorithms which can be applied in order to label and count objects in an image. Next one common method will be discussed, the run track algorithm, see Figure 3.4.

The run track algorithm scans picture elements in an image from the top left corner. Wherever a pixel intensity changes from 0 (background) to 1 (object), the pixel valued 1 is labelled. The label is spread south if the pixel below also holds the value 1. When the right scan is finished a scan starting from the left takes place spreading the labels created in the right south scan to the left of the labelled pixels, of course if the pixel value is 1. The process is repeated until all pixels valued 1 are labelled. The last part of this algorithm performs a re-labelling step where all neighbouring pixels obtain the same label, see Figure 3.5. Labelling algorithms contain a label counter, continuously tracking the number of different labels put in the image, thus counting the number of objects in the image [30].

#### 3.3.2 Size

According to ISO 4407 [4] it is desirable to measure the maximum length of a particle and thereby classify them according to size. The main issue with image analysis is that there are a number of different methods for evaluating the maximum diameter of a particle. The form of the particle is important which will become clear in this section. Next two methods will be considered which may be used when analysing contaminants in lubricating oils.
3.3 Image analysis

Figure 3.4. Binary image after thresholding.

Figure 3.5. Labelled objects in image.

Caliper dimension

Caliper dimension or Feret diameter is a measure of the greatest distance between any two pixels on the periphery of an object. The Caliper dimension is determined
by sorting through the coordinates of the pixels along the objects boundary. The
two pixels with the largest and smallest coordinates are chosen and the euclidean
distance (3.1) between these two points is calculated, rendering the caliper di-
mension. The euclidean distance is defined as the linear distance between two
points in an image, given by the Pythagorean Theorem. Two points $A(x_A, y_A)$
and $B(x_B, y_B)$ are separated by the euclidean distance,

$$D(A,B) = \sqrt{(x_A - x_B)^2 + (y_A - y_B)^2}. \quad (3.1)$$

Consider an s-shaped cellulose fibre (Figure 3.6) which is a common bi-product
of defective filters in lubricating systems, also a contaminant of great interest
in particle analysis. In this thesis one of the main goals is to separate fibres
from other particles. Generally a fibre is defined as a particle of at least
100 $\mu$m with a width to length ratio of 1:10 [4]. If analyzed solely based on the caliper
dimension algorithm, the s-shaped fibre shown in Figure 3.6a) will render a fibre
length which differs from the true length of the fibre. This will result in erroneous
width-to-length ratio and consequently an incorrect classification of fibres. The
Caliper diameter holds a poor estimation of fibre length and should therefore not
be implemented for analysis of fibres.

Euclidean distance map with skeletonization

In a euclidean distance map, EDM, the pixels within an objects boundary hold
values of the euclidean distances to the nearest neighbour in the background. If
an object pixel has two such distances, i.e. is equally separated from both bound-
aries, the object pixel is a part of the medial axis transform, MAT. The MAT is
another name for the skeleton of the object, see Figure 3.6b). In an EDM the
intensity value of each pixel corresponds to the distance from the boundary of
the object. If the EDM is multiplied with the MAT, the data can be illustrated
in a histogram where the amount of pixels vs. the pixel intensity is presented.
Averaging these values for all points on the skeleton gives the fibre width (Figure

\[\text{[Figure 3.6. Comparison between measuring particle size with a) Caliper dimensions versus b) skeletonisation with euclidean distance maps. Adaption of figure from [27].]}\]
To estimate the length of the fibre there are a couple of different methods used based on skeletonization. In the most accurate method the fibre length is evaluated by fitting a smooth curve to the skeletonised pixels. Another method for length estimation depends on the number of pixel pairs and their orientation in the skeleton. Assume a pixel, with an edge length of one arbitrary unit. The distance between the centres of mass between two orthogonal neighbouring pixels equals in this case to one. Then consider the same pair of pixels now ordered diagonally, the distance between the centres of mass of the pixels equals to $\sqrt{2}$. This estimation has later on been optimized since it produces a slight overestimate of the length. The optimized constants estimate the length as

\[
\text{Length} = 0.948 \cdot (\text{Nr. of orthogonal pairs}) + 1.340 \cdot (\text{Nr. of diagonal pairs}) \quad (3.2)
\]

with a mean error of approximately 2.5% [27].

![Orthogonal pixel pair](image1.png) ![Diagonal pixel pair](image2.png)

**Figure 3.7.** Distance comparison between orthogonal and diagonal pixel pairs. Pixel pairs ordered orthogonally as opposed to pixel pairs ordered diagonally have longer distance between the pixel centres.

**Area**

To measure the area of a binary image, i.e. a thresholded image, the procedure is fairly straightforward. The number of pixels valued 1 are counted and multiplied by the area of one pixel. Intuitively, with higher resolution of the object, the area becomes more accurate. The reasoning is depicted in Figure 3.8.

**Perimeter**

At a first glance it might be thought of as a simple process to calculate the perimeter, one of the most well-defined properties of a particle. This is however not the case. In the early days of perimeter algorithms, systems estimated the boundary length by counting the number of pixel edges that touch the background. Just like in the case of fibre length estimation, the pixels touching the background are counted pair wise and classified by orthogonal or diagonal orientation. The perimeter length is subsequently calculated by (3.2). Nowadays, the most accurate perimeter values are extracted by fitting smooth curves to the feature boundaries. The greatest difficulty in estimating the perimeter of an object is that it is highly dependent of the imaging resolution. Image objects with high resolution, i.e. objects covered by many pixels, reveal more
boundary irregularities than those of low resolution. High resolution particles would intuitively render larger values of the objects’ perimeter. Because of this dependence, current ISO standards state that an object should be covered by at least five pixels in length for accurate size estimation. For evaluation of shape which will be discussed in the following section, this lower limit lies at about ten pixels. The main reason for this is that the accuracy of perimeter estimations is crucial because of its’ use in many shape descriptors.

3.3.3 Shape

In particle analysis, shape descriptors are defined by a number of different properties. The oldest class of shape descriptors are obtained from some fundamental particle properties like perimeter, area, fibre length, fibre width, maximum length and breadth (See Figure 3.6). The most widely used shape descriptors for classification of particles are presented in Table 3.1. The form factor, (3.3), is used to evaluate the edges of particles while the aspect ratio, (3.4), is more concerned with the elongation of the particle. An example of the relationship between form factor and aspect ratio can be found in Figure 3.9. The roundness (3.5) describes the circularity of a particle. A perfectly round particle, has a roundness descriptor equalling 1. A compactness measure estimates just what is implicates, how compact the area of the particle is. If the particle is branched the compactness is lower than that of a perfectly spherical particle. Two shape descriptors easily predict a fibre, the elongation and curl, (3.7) and (3.8). If an object has a length to width ratio, i.e. elongation of at least 1:10 and a size of at least 100 µm it is classified as a fibre according to standard practice [4]. Further on, the curl of a fibre can help to distinguish certain fibre types from each other (Figure 3.10). More strictly compactness measures the object boundary closeness to the centre of mass of the object. No single shape descriptor uniquely describes the shape of
3.3 Image analysis

Table 3.1. The most common shape descriptors for particle analysis. Table adapted from [27].

\[
\text{Form factor} = \frac{4\pi \cdot \text{Area}}{\text{Perimeter}^2} \tag{3.3}
\]

\[
\text{Aspect ratio} = \frac{\text{Length}}{\text{Breadth}} \tag{3.4}
\]

\[
\text{Roundness} = \frac{4 \cdot \text{Area}}{\pi \cdot \text{Length}^2} \tag{3.5}
\]

\[
\text{Compactness} = \sqrt{\frac{\frac{4}{\pi} \cdot \text{Area}}{\text{Length}}} \tag{3.6}
\]

\[
\text{Elongation} = \frac{\text{Fibre length}}{\text{Fibre width}} \tag{3.7}
\]

\[
\text{Curl} = \frac{\text{Length}}{\text{Fibre length}} \tag{3.8}
\]

a particle. From combinations of descriptive parameters the appearance of the particle can be predicted. Experts have proven that by applying shape descriptor algorithms combined with decision making software it is possible to render accurate results about the type or types of mechanical wear prevalent in a system.

3.3.4 Color

Color extraction from an image might deceive, when thought of simple but in practice it holds quite a few obstacles to be overcome. A color image is stored as three integer values in each pixel, one from the red channel, one from the green, and a last one from the blue. This constitutes a so called RGB color scheme. Each of these three colours normally have intensities varying from 0 to 255. The three colours are mixed together in every pixel of the image, to form a certain color in the visible spectrum. The number of intensity and color combinations possible reflects the difficulty of setting an appropriate threshold to select a certain color from an image. An object, say a red particle, often holds at least a dozen shades of red depending on the topological structure. In order to threshold a red particle
it is therefore necessary to select a span of red intensities to be thresholded. This can be done manually by choosing intensities for the red, green, and blue channel and iteratively slimming down the intensity span until the desired features are thresholded. Another method for thresholding an image in the RGB space is by using the Euclidean distance. If we know the average color that is desired for segmentation, the color may be denoted by a vector, \( c \), in the RGB space. In order to specify a range of intensities for thresholding it is necessary to have a measure of similarity. One of the simplest measures is the Euclidean distance. We say that \( z \) is similar to our average vector \( c \) if the distance between them is less than the specified threshold \( D_0 \). The Euclidean distance between the points for
3.3 Image analysis

Figure 3.11. Threshold based on Euclidean distance measure. Adaption of figure from [31].

comparison, c and z is

\[
D(z, c) = \sqrt{(z_R - c_R)^2 + (z_G - c_G)^2 + (z_B - c_B)^2}
\]  

(3.9)

where R, G, and B denote the RGB components of the vector c. The spherical threshold rendered from the Euclidean distance measure is depicted in Figure 3.11. All points within or on the surface of the spherical threshold, i.e. \( D(c, z) \leq D_0 \), are pixels in the selected intensity span.
Chapter 4

System overview

Briefly, the sample preparation consists of filtering certain amounts of oil (normally 100ml) through fine pore filters. The particles on the filters are analysed with an Olympus BH-2 UMA optical microscope. The microscope table is connected to an automated control box which allows scanning in x and y directions. Images are acquired with a monochromatic Sony AVC-D5CE CCD camera, generating a gray scale digital video stream. The images are stored with 8 bits per pixel, rendering $2^8 = 256$ possible intensity levels in gray scale. Also, a Kappa Argon SDC 212C CCD color video camera was implemented at the end of the experimental study for acquisition of color images. This camera captures 12 bits per pixel resulting in $2^{12} = 4096$ possible intensity levels in the color scheme. An MV Sigma/Delta SLC frame grabber card is used to capture frames from the digital video stream and display them in the computer, enabling further processing. The software PicEd Cora [32] is used for image processing and analysis.

PicEd Cora is limited in functions available for the image analysis procedures introduced in Chapter 3. In PicEd Cora there are functions allowing for counting and classification of particles according to size. Also, it is, according to the manual [32], possible to identify and count fibres and metal contaminants. Fibre detection in the software is based on the the conditions introduced in Chapter 3, a minimum length of $100\mu m$ and a length to width ratio of at least 1:10 is sufficient for fibre distinction. Thereby, quantification, size, shape, and brightness are parameters that are taken into consideration throughout the thesis. Whether PicEd Cora is powerful enough to obtain this information accurately or not will be evaluated in the following chapters. The software implements Caliper dimensions for maximum length estimations of objects. Fibres analyzed by those algorithms will most likely not be evaluated correctly (remember Figure 3.6). For further information about the capabilities of the software the reader is referred to the manual [32].
Figure 4.1. IMPACT setup. The image analysis system consists of a light microscope connected to an automatically controlled scan table and a PC with image analysis software.
Part II

Experimental
Chapter 5

Method

Next, the complete procedure, from filter preparation to filter analysis is described more thoroughly. The methods for filter preparation and image analysis are based on a combination of current standard methods. For validation and method comparison, other analysis methods will be employed which will be described in upcoming sections.

5.1 Sample series

Seven oil samples were tapped from a testing rig for gas turbines by personnel at SIT. The oil has before this sample point passed a filter system, filtering the lubricant from most of the contaminants. The samples were sent to FLT, Exova for oil condition monitoring where among other analyses, automatic optical particle counting is regularly performed. The goal in this thesis is to study how quantification of particle contaminants correlates between manual and image analysis primarily, and later compare the results with data from the automatic particle counter. Three sample series were prepared with seven samples in each, resulting in a total sample size of 21 filters.

5.2 Oil sample filtration

In order to analyze oil samples with IMPACT, the first step is to filter them. Gridded nitro cellulose Millipore filters with 47mm diameter and a pore size of 0.8µm are commonly used for oil filtration. The filter is clamped between a glass funnel, with a calibrated diameter, and a filtration setup with a vacuum pump, see Figure 5.2. Normally 100ml of oil is poured onto the filter. Another 100ml of solvent, here petroleum ether, is added and mixed with the oil in order to clean the filter free from oil residues and discolorations prior to analysis. Vacuum pressure is then engaged to press the oil through the filter, leaving contaminants at the filter surface. A final step before analysis is to thoroughly clean the filter from oil by adding more solvent. The filter should have an even light gray color after
filtration for optimal image analysis. It should be left in room temperature to dry before further analysis. A flow chart describing the filtration steps can be found in Figure 5.1.

![Flow chart of the filtration procedure](#)

**Figure 5.1.** Flow chart of the filtration procedure. Adapted from flow chart in [29].

![Filtration setup](#)

**Figure 5.2.** Filtration setup.
5.3 Light microscopy technique

Optical microscopy is a common tool when analyzing particles in oil. In theory, an optical microscope has a submicron detection limit. In practice this limit is higher since halo effects are profound when analysing subjects that small [22]. In this thesis the attempt is to analyse particles as small as $4 \mu m$.

5.3.1 Magnification

The magnification is a key setting when analyzing particles in the lower micron range. According to current standards ISO 4407 [4] and ISO 16232 [33], at least ten pixels detecting the desired object are necessary for accurate estimation of the particles’ dimensions. When passing to a particle size in the lower micron range, < 20 $\mu m$, five pixels are sufficient. In the system studied in this thesis, a magnification of 200 times equals a pixel size of $0.6983 \mu m/pixel$. Five pixels enable detection of particles larger than 3.5 $\mu m$. With a pixel size of $0.4702 \mu m$, a magnification of 300 times allows particles as small as 2.4 $\mu m$ to be detected. Throughout this study a magnification of 200 times will suffice to analyze particles larger than 4 $\mu m$. An initial study was performed in order to evaluate the performance of three different magnifications (Figure 5.3). The view fields were examined over a filter, each by three different magnifications, 50x, 100x, and 200x. The study confirms that a magnification of 200x allows for the best detection and the most accurate size separation of particles.

![Figure 5.3. Magnification effect on particle count.](image-url)
5.3.2 Brightness

An evaluation of the effects of light intensity on the total number of particles was performed by analyzing five fields of view at 200x magnification at six different light intensities. Results of the analysis are found in Figure 5.4. The study indicates an optimal brightness at 80%. At this intensity a threshold is set without difficulties and the background color does not inhibit the analysis. This intensity allows detection of the largest total of particles. As can be concluded from Figure 5.4, particles larger than $6\mu m$ are not affected by changes in light intensity to any greater extent. At intensities < 80 %, the background intensities coincide somewhat with the edge pixel intensities of the particles, complicating accurate particle identification. At 90% intensity the particles are overexposed by the light which slightly diminishes their size.

5.4 Image analysis

5.4.1 Particle counting

A filter area of at least $20mm^2$ and at least 300 particles need to be scanned in order to render a statistically correct analysis according to ISO 4407 [4]. A flow chart of IMPACT procedures is found in Figure 5.5. The user of IMPACT first needs to modify the illumination in the microscope manually and adjust the focus so that optimal conditions, described in Sections 5.3.1 and 5.3.2, are reached on screen. During this thesis the settings used were 80% brightness intensity and a maximum focus which is achieved by trial and error. The following steps consist
of the user performing either a manual scanning with the help of the coordinate table or an automatic scanning where the table and the software on their own scan a selected filter area. Throughout this study, manual sequences were acquired for optimal focus over all images. Manual scanning allows for the user to adjust the focus between images whereas the automatic scanning does not. This may with high magnifications yield unfocused areas in the images. The software PicEd Cora automatically processes the images according to the steps described in Chapter 3 which are further discussed next.
Method

- **Live image** - Live image after selecting magnification and brightness parameters, see Figure 5.6.

- **Gray scale image processing** - Consists of the pre-processing procedures described in Section 3.1. A Laplace filter is used for sharpening the object boundaries while an averaging filter is applied for evening out background irregularities, see Figure 5.7.

- **Segmentation** - In the segmentation part half amplitude thresholding is performed, according to Section 3.2. The binary image is superimposed in yellow over the pre-processed image, shown in Figure 5.8.

- **Binary image processing** - Sizing the objects in the image by measuring the Caliper dimensions described in Section 3.3.2. The result is found in Figure 5.9.

The results of the measurement of each particle are collected in a table which can be saved.

![Figure 5.6. Raw image from camera.](image1)

![Figure 5.7. Image pre-processed with Laplace and averaging filter.](image2)
5.4 Image analysis

Figure 5.8. Image thresholded with the half amplitude method.

Figure 5.9. Objects in image counted and analyzed with Caliper dimensions.

5.4.2 Metal detection

Metal particle analysis is conducted by double scanning the desired area. The first scan is regular, detecting all light that is reflected off the filter surface, i.e. all particles abundant on the filter, just like described in the previous section (Figure 5.10). During the second scan, Figure 5.11, a planar polarized optical filter is applied. Only reflective materials, e.g. metals, will be reflecting light. An appropriate threshold is set on the first, regular, scan detecting the total amount of particles at the filter. As both scans correspond exactly to each other, it is now known what is a particle and what is not. Within the threshold of the particles, the software analyzes the second, polarized scan for intensities higher than a certain predefined value as to determine whether or not any reflections are present within the particles’ boundaries.

5.4.3 Fibre detection

Fibre detection is conducted according to Section 5.4.1. An additional setting of the length to width ratio (1:10) is applied, which should by definition only include fibres. The length is measured with Caliper dimensions, described in Section 3.3.2 which is proven not to be an appropriate size estimation for fibres, see Figure 3.6.
Figure 5.10. Regular filter scan.

Figure 5.11. Filter scan with optical polarization filter.
Chapter 6

Results - Validation

To verify the accuracy of the image analysis system, manual counting was performed of the same membrane filters as a reference method. Manual counting follows ISO standards 4406 and 4407. The membrane filters were manually analyzed with a light microscope at 400x magnification. In one of the microscope oculars a ruler is found enabling size estimation of particles.

The ruler was swept past randomly selected squares at the gridded filters and all particles larger than 4 µm were counted and separated into three size classes, >4µm, >6µm, and >14µm. At least 20 squares on a filter need to be analyzed and at least 300 particles should be counted for correct statistical quantification. It is important to select squares representative of the particle distribution over the filter.

6.1 Total particle count

A total of 21 filters were counted both manually and with IMPACT. To evaluate if the results from the image analysis correlate with the manual particle analysis, a scatter plot is created and a correlation factor is calculated. A correlation factor of 0.996 (Table 6.1) indicates that there is strong correlation between manual counting and IMPACT which can also be seen in Figure 6.1. All data points follow a linear trend.

The two methods were also compared with a pair wise two-sampled t-test to evaluate if they in fact do obtain the same expectance values, µ. For the complete analysis data set the reader is referred to Appendix A. A two sampled t-test is a hypothesis test which helps in determining whether the results from both analysis techniques coincide or not. It tests if the difference between two normally distributed populations,

\[ y_j = x_{Mj} - x_{Ij} \]  \hspace{1cm} (6.1)

, is a result of randomization (null-hypothesis) or if the populations do not render the same results. In the equation M stands for manual analysis and I for IMPACT. A null-hypothesis, stating that the difference, \( y_j \), between manual analysis and
IMPACT is nonexistent, is tested against the opposite statement, that there is a bias in the results.

The following hypothesis is tested:

\[ H_0 : \mu_{y_j} = 0 \]  
\[ H_1 : \mu_{y_j} \neq 0 \]  

(6.2) \hspace{1cm} (6.3)

The test is performed with a confidence level of 95%.

If

\[ 0 \in \text{Confidence interval, } CI \]  

(6.4)

then the null hypothesis in (A.2) cannot be discarded, i.e. it is possible that the results from both analyses coincide. The results of the t-tests are found in Table 6.1. The t-test cannot reject the zero-hypothesis that the methods have the same expected values since \( 0 \in CI \). The two methods for particle counting may have the same expectance values i.e. it is possible that they do give the same results in \( \geq 95\% \) of the counts.

![Figure 6.1. Correlation between manual and image analysis for particles larger than 4\( \mu m \).](image)

6.2 Size distribution

Particles in the mid-size class, \( > 6\mu m \), also show good correlation (Figure 6.2). The sample points follow a linear trend line. Particles are sufficiently prevalent in the oil for accurate statistical quantification. Still, there is a slight difference compared to the correlation for the total particle count. A t-paired test is performed on
6.2 Size distribution

Table 6.1. T-paired test of size distribution.

<table>
<thead>
<tr>
<th>Size Class</th>
<th>N</th>
<th>Mean</th>
<th>StDev</th>
<th>SE Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 4µm</td>
<td>21</td>
<td>32005</td>
<td>16715</td>
<td>3852</td>
</tr>
<tr>
<td></td>
<td>21</td>
<td>31309</td>
<td>17280</td>
<td>3771</td>
</tr>
<tr>
<td></td>
<td>21</td>
<td>696</td>
<td>1659</td>
<td>362</td>
</tr>
<tr>
<td>CI</td>
<td>-59; 1452</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-Value</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>P-Value</td>
<td>0.069</td>
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<table>
<thead>
<tr>
<th>Size Class</th>
<th>N</th>
<th>Mean</th>
<th>StDev</th>
<th>SE Mean</th>
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<td>4650</td>
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The same hypothesis is set as for the total particle count. Results of the t-tests for three different size distributions are found in Table 6.1. The hypothesis that both manual and image analysis do render the same results for particles larger than 6µm cannot be rejected since 0 ∈ CI. Analyses may thereby correspond to each other even when counting particles larger than 6µm. Particles larger than 14µm are usually not that prevalent in this oil type under normal conditions. When evaluating particles counted in the largest size class (> 14µm), the area analyzed becomes significant. A single particle’s difference between image and manual analysis can affect the recalculated result up to 50% why the correlation between these results is relatively poor, Figure 6.2. This is most likely a consequence of multiple variables, statistical counting being the most significant. If the complete filter area was analyzed both manually and with image analysis, the correlation would most likely be heightened. The t-test shows that even if the correlation is significantly worse than for the smaller size classes, the expected values still might agree between the two methods. The poor correlation (see Table 6.1) can merely be a factor of statistical uncertainty when counting the particles.
6.3 Metal detection

For metal particle detection, samples containing iron particles smaller than 70µm were prepared. Oil was filtered through 0.8µm Millipore filter before mixing it
6.4 Fibre detection

with iron particles. The expected ratio of metal particles versus the total amount of particles should theoretically reach close to 100% i.e. close to all particles at the filter should be reflective. This will however not be the case since contaminants from the vials used, the environment, and the oil handling are always apparent in the experiments. Only 36% of the particles between 4µm and 6µm are detected as metal particles and the correlation is quite poor, Figure 6.4. For particles in the size range of between 6µm and 14µm about 66% are detected as metal particles, Figure 6.5. Particles smaller than 14µm seem to reflect the planar polarized light poorly, making it difficult to detect them in the smaller size range. Particles larger than 14µm analyzed with 50x are separated adequately with a ratio of 96% of the particles being identified as metals, while the same size range analyzed in a 100x magnification detects about 92% of the iron. When comparing the effect of magnification (Figures 6.6 and 6.7) it is clear that with 50x magnification, the amount of metallic particles detected improves. The deviation in the 50x magnification, of 4%, might be a result of topological irregularities of the particles hindering the light to be reflected, or contaminants in the oil.

6.4 Fibre detection

To evaluate the capabilities of PicEd Cora for fibre separation, a classical fibre shape was drawn in standard paint software. The image was re-opened in PicEd Cora and analyzed to illustrate how the software generally measures length. Figure 6.8 shows the result after evaluating a fibre shape automatically which is the standard of use. In Figure 6.9 the length of the fibre is measured by applying a polygon manually through the fibre. PicEd Cora seems to be measuring the length of the fibre just as hypothesized in Chapter 3. It measures the maximum Caliper dimension which is a poor length estimation of a curled fibre. A difference in length of about 900µm is studied in the experiment which is a large deviation from the true fibre length.

6.5 Color detection

Red particles are frequently appearing in lubricating oils. Red particles may be copper alloys or originate from corrosive wear, i.e. rust. An attempt was made, with the new Kappa CCD camera, to select the intensities of a red particle in Figure 6.10. Red intensities were selected and thresholded by trial and error in Figure 6.11. If the same basic procedure as for metal particle detection could be implemented, it would theoretically be possible to detect particles of a certain color. In practice this is not the case since that is not a function of PicEd Cora today.
Figure 6.4. Detection of metallic particles, sizes 4 – 6\(\mu m\).

Figure 6.5. Detection of metallic particles, sizes 6 – 14\(\mu m\).
6.5 Color detection

Figure 6.6. Detection of metallic particles, > 14µm with 50x magnification.

Figure 6.7. Detection of metallic particles, > 14µm with 100x magnification.
Figure 6.8. Fibre length by Caliper dimension. The length is measured to $650\mu m$.

Figure 6.9. Fibre length by polygon fitting. The length is measured to $1555\mu m$. 
6.5 Color detection

Figure 6.10. Color image from IMPACT.

Figure 6.11. Detection of a red particle. The red particles in the image are thresholded and the binary image, i.e. the red pixels are a binary mask superimposed over the original image.
Chapter 7

Results - Comparison to optical automatic particle counting

All oils analyzed manually and with image analysis were previously analyzed with a third method, namely the automatic optical particle counter. The automatic optical particle counter was introduced in the first chapter as the most frequently used analysis technique for counting particles today. For the turbine oils examined here, the automatic particle analysis is a standard method. The oils were therefore already analyzed previous to image and manual analysis. In Figure 7.1 average results for particles larger than 4µm from manual and image analysis are compared to the results from the automatic particle counter. Full data used in the following figures may be found in Appendix B.

The automatic particle counter gives a systematic multiple factor error while image analysis and manual analysis correspond very well. A mean error of approximately 78% is studied with the automatic particle counter compared to manual and image analysis, Figure 7.2. This might be the phantom particles which can be seen at the filter as well (Figures 7.3 and 7.4). When filtering the samples, transparent amorphous aggregates consisting of smaller elements can be seen trapped in the pore structure. The appearance is not consistent with the typical manifestation of wear particles.
Results - Comparison to optical automatic particle counting

Figure 7.1. Comparison of Manual analysis, IMPACT and automatic particle counting.

Figure 7.2. Ratio of phantom particles in sample series.
Figure 7.3. Phantom particles on Millipore filter, 100X magnification. The phantom particles are the reflective amorphous aggregates. In the top and bottom left corner, two fibres are also found. Note that these are not phantom particles.

Figure 7.4. Phantom particles on Millipore filter, 400X magnification.
Results - Comparison to optical automatic particle counting
Part III

Discussion and Conclusions
Chapter 8

Discussion

When analysing wear particles in oil, the optimal information about the contaminants would lead to a conclusion about what is happening at interfaces in a system. With the software and machine limitations in mind, IMPACT can be classified as a stable and quite adequate analysis technique for the demands of the customers today. It is powerful enough to count particles, as well as classify them in appropriate size classes. The ability to separate metallic particles is quite sufficient for particles larger than $14\,\mu m$. Metallic detection could be further optimized by acquiring a high resolution camera which is more sensitive to brightness intensity changes. What needs to be kept in mind is that this function is highly dependent of the surface texture of the particle. Therefore it is practically impossible to completely eliminate the sources of error when separating metallic particles.

As mechanical systems develop and become more sensitive, and as image analysis technology in combination with artificial neural networks or fuzzy logic expands, the demand for qualitative information from oil analyses will increase. IMPACT is a real time analysis technique, allowing the analyst to continuously follow what is being detected. An advantage with this is that complicated particles or discolorations of the filter can be avoided simply by selecting another part of the filter for analysis. Comparing to the closed automatic particle counter, it is proven in Chapter 7 that the counter is quite limited in this sense. The automatic particle counter seems to count amorphous structures which have been studied on filters as well. These phantom particles are by definition not contaminants in the oil and therefore not counted in IMPACT. Researchers have shown, as presented in Chapter 2, that this may be a result of aggregated additives in the lubricant. Not knowing which additives are added to the turbine oil makes determining the cause of the faulty results from the automatic particle counter quite difficult. An initial elemental analysis with scanning electron microscopy-energy dispersive spectroscopy, SEM-EDS, was conducted of the phantom particles on the filter previously presented in Figures 7.3 and 7.4. The complete SEM-EDS analysis can be found in Appendix C. The results indicate traces of silica, sodium, and sulphur which are all common elements in additives. Silica residues may be an indication of micellar formations in the lubricant, a likely cause of anti-foam additives. As presented in Chapter 2,
anti-foam agents tend to give the largest increase of particles in optical particle counters, deviations which are studied throughout this thesis as well. It is however important to be aware that the aforementioned structures are present in the oil. As previously illustrated in Figures 7.3 and 7.4, these amorphous structures tend to aggregate and stick to the filter. In large scale mechanical systems, where the lubrication system often also contains a filter system, this can lead to plugging of filters and thereby costly energy losses. Nonetheless, from the point of view of pure wear, the structures are most likely irrelevant.

Particle counting with image analysis renders more accurate analysis data of wear rate. It should therefore be the analysis of choice when analysing wear particles. A combination of image analysis aided particle counting and automatic particle counting could be an appropriate, indirect method of estimating the amount of amorphous additive structures found in the lubricant. In the turbine oil analyzed throughout this thesis, the fraction of phantom particles seems to be relatively stable, with a mean of about 78% of the total particle count in the optical counter (see Figure 7.1).

Dealing with the capabilities of the image analysis software, PicEd Cora, they are slightly limited especially when it comes to fibre detection. The ability to automatically detect and count fibres rapidly is a desirable property of the image analysis setup. The current software configuration is not accurate and needs some modifications to work properly for fibre detection. When this is achieved, the system will be powerful enough to substitute manual counting at FLT Exova completely, working according to the guidelines of ISO 4407.
Chapter 9

Conclusions

Throughout this thesis it is shown that particle quantification with the aid of image analysis is a quite potent tool in the field of lubricant analysis. IMPACT correlates very well with manual particle counting. The imaging system is powerful enough to accurately

- count the number of particles on a filter surface
- estimate the particle sizes
- classify the particles according to ISO standard practice
- separate and quantify metallic particles $> 14\mu m$

The method today is on the other hand not capable to calculate fibre lengths correctly. Therefore, separation of fibres should be further investigated. IMPACT does not correlate with automatic optical particle counting. Interfering amorphous structures are suspected to be the cause of the deviation.
Chapter 10

Future potential

The analysis setup studied in this thesis holds the basics for particle analysis; counting and sizing particles. As discussed in Chapter 8 the image analysis software is limited in functions for qualitative analysis of particles. More advanced software would allow for studies about wear mode and origin of particles. Still, the image analysis setup, in its present configuration meets the demands of many customers today and is in fact a powerful tool and support for experts. With advancing microscopy techniques, the methods for analyzing particles are expected to increase. The demand for more information concerning constitution, texture, density, and thickness of particles will increase. Peng et al. (1999)[25] showed that by combining numerous particle properties with the help of fuzzy logic, it is possible to conclude which type of wear particles are present in the lubricant and their origin. Myshkin et al. [24] instead studied the power of artificial neural networks ANN, for decision based analysis of particles. ANN being slightly more advanced than fuzzy logic, also showed great success in particle analysis. Throughout this thesis it is proven that with the help of a standard light microscope and image analysis software, results comparable to manual analysis can be achieved. To lessen the expenses of buying advanced image analysis software specialized on particle analysis there is open software operating under the general public licence, which may be used both commercially or non-commercially, and can be adapted for analysis of particles. One very powerful program is ImageJ provided by the National Institutes of Health. The software was originally developed for blood cell analysis. The same algorithms and operations for size, shape, and quantity are implemented in ImageJ allowing, with small modifications, for particle analysis purposes. Throughout this study it has been concluded that PicEd Cora does not apply an accurate algorithm for analysis of fibres. The length estimation is based on the maximum Caliper dimension instead of the euclidean distance map with skeletonization while the width seems to be estimated accurately by counting pixels. Yet another advantage of ImageJ is that both algorithms may be applied and combined for particle analysis. Finally there are open source plug-in programs for decision based analysis, either by ANNs, fuzzy logic or principal component analysis, allowing for wear mode and origin conclusions of particles. Color im-
Future potential

age acquisition is still somewhat an unexplored territory. The color camera was implemented by the end of the experimental study in order to analyze the possibilities of color screening in particle analysis. The first few experiments with the color camera show great potential in the field even if it, at this first stage, is difficult to distinct a certain color and set an appropriate threshold for that color only. Further studies about frequently appearing coloured particles in lubrication oils are essential. It is necessary to distinct the colours and find unique color bands for each particle type. This would in the end render another descriptive parameter for implementation in the decision based analysis. After meeting with current clients with an interest in particle analysis techniques, it is clear that the resolution of current techniques is rather poor. There is still a too wide gap between inductively Coupled Plasma analysis (ICP), an elemental analysis technique which detects particles in the size range of < 2.5\(\mu m\) and optical and image analysis detecting particles > 4\(\mu m\) according to ISO 4406 [3]. The new color camera, improving the resolution with about 25\%, theoretically allows for detection (counting and sizing) of particles as small as 2.6\(\mu m\). During this thesis, a couple of different paths for further studies were observed. The main one being the need for a comparative study between image analysis and automatic optical particle counting. The analysis data from the two methods are not comparable for all oil types, a factor which many customers may not be aware about. Studies, one of which is the one performed during this thesis, have shown that there are substances in lubricants, added deliberately or not, which may cause heightened particle levels even though these are not traditional particles desired for analysis. If we beforehand know which types of additives, or outer factors that contribute to phantom particles in the automatic particle counter the method can be avoided in favor of image analysis of filters.
Bibliography


Appendix A

Statistical evaluation with pair wise two-sample t-tests

A two-sample t-test is used to evaluate how well both analysis methods, manual analysis and IMPACT, correlate. A test statistic

\[ y_j = x_{Mj} - x_{Ij} \]  

(A.1)

is set as the difference between both methods. The data set consists of 21 filters, each filter analyzed with both manual analysis and IMPACT. The particle counts are separated into three size classes, > 4\( \mu m \), > 6\( \mu m \), and > 14\( \mu m \). The test-data is found below for each size class. The same hypothesis is used for all three tests

\[ H_0 : \mu_{y_j} = 0 \]  

(A.2)

\[ H_1 : \mu_{y_j} \neq 0 \]  

(A.3)

is tested with a confidence level of \( \geq 95\% \). Excel is used for executing the t-tests. The results are found in Chapter 6.
Statistical evaluation with pair wise two-sample t-tests

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Appendix B

Analysis data - automatic optical particle counter

Next the analysis data used in Figures 7.1 and 7.2 are presented. The analyses were performed when the oil samples arrived to the lab, that is between six months to two weeks previous to the experiments performed during this thesis.

Table B.1. Data set - automatic optical particle analysis.

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Appendix C

SEM analysis

A SEM analysis of the filter presented in Chapter 7, Figures 7.3 and 7.4 was performed at the materials department of Exova. The results are found in Figures C.2 and C.1 and the graph on the next page. The results and conclusions of the analyst were the following:

“It was not possible to statistically detect the elemental composition of the phantom particles. I do see the phantom particles in the microscope but analytically it is difficult to detect what separates the amorphous structure from the surrounding filter. It does however seem to contain traces of sodium, silica, and sulphur. The carbon, nitrogen, and oxygen peaks originate from the filter”.

In Figure C.1 it is clearly seen that the amorphous particles are very thin which supports the conclusion of the analyst that there are no significant amounts to be detected.

Figure C.1. SEM image of phantom particle.
Figure C.2. Graph of the SEM analysis.
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