Image Cover Sheet

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TITLE

POLARIZED LIGHT MICROSCOPY AS A FILTER DEBRIS ANALYSIS TOOL

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POLARIZED LIGHT MICROSCOPY
AS A FILTER DEBRIS ANALYSIS TOOL

by

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ABSTRACT: The use of polarized light microscopy (PLM) as an analysis tool for lube oil filter debris examination is investigated. Reflectance PLM is shown to be able to discriminate between metallic wear particles and nonmetallic particles present in filter debris by differentiating optically isotropic versus anisotropic materials. The utility of transmittance PLM in identifying common nonmetallic materials in filter debris is also demonstrated. The identification is accomplished by estimation of the particle's refractive indices through a technique known as dispersion staining. That information, coupled with knowledge of the material's morphology, permits identification by comparison with literature references.

INTRODUCTION: Polarized light microscopy (PLM) has been used for decades; in the reflectance mode as a metallographic tool (1) and in the transmittance mode as a means of characterizing mineralogical species (2, 3). These uses suggest that the technique may be of use in the examination of lube oil filter wear debris, which contains both metallic wear particles and mineral particulates (usually resulting from ingress of atmospheric or other contaminants).

Light emitted from a source consists of transverse waves vibrating symmetrically at right angles around the direction of propagation. A polarizing filter placed in the light path allows the transmission of light vibrating in only one plane along the propagation direction. Such light is considered to be "plane polarized".

Optical microscopes that utilize polarized light are typically used in one of two modes: transmittance or reflectance. Regardless of the mode, polarized light microscopes consist of a polarizing filter, usually called the "polarizer", situated, as shown in Figure 1, between the light source and the sample. A second polarizing filter is situated between the sample and

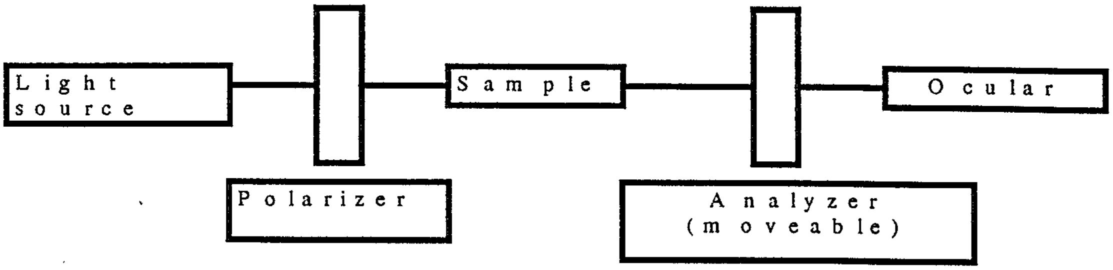

Figure 1: Schematic of light path in polarized light microscopes.

objective/ocular. Detailed discussion of standard microscope components and operation is beyond the intent of this report. A working familiarity with optical microscopes is therefore assumed. Interested readers are referred to the many excellent texts on these topics. (4, 5, 6, 7)

The second polarizing filter, usually called the "analyzer", is constructed so that its plane of polarization is perpendicular to that of the polarizer. It is also usually moveable, that is, it can be placed in or out of the light path thereby permitting sample analysis using one or two polarizing filters. Analysis using two polarizing filters with mutually perpendicular polarizing planes is termed "analysis under crossed polars". Under crossed polars the plane polarized light transmitted through the polarizer is blocked by the analyzer and the area of illumination appears dark.

In its simplest sense, material examination under crossed polars permits distinction between materials (or regions of a material) that are optically isotropic versus optically anisotropic. Optically isotropic materials interact with plane polarized light so that the transmitted or reflected light remains in the plane established by the polarizing filter. Thus, optically isotropic materials appear dark under crossed polars as the light transmitted or reflected from the sample is prevented by the analyzer from reaching the eyepiece. Optically anisotropic materials, on the other hand, rotate the plane of polarized light established by the polarizer by some material-dependent angle. Therefore imaging of optically anisotropic materials is possible as the polarized light can now traverse the analyzer.

This paper investigates the use of PLM in the study of filter debris particulates. Discussion of the utility of the technique is divided into two parts: reflectance PLM and transmittance PLM.

Figure 2: Reflectance PLM image of steel wear particle, taken using 1 polar.

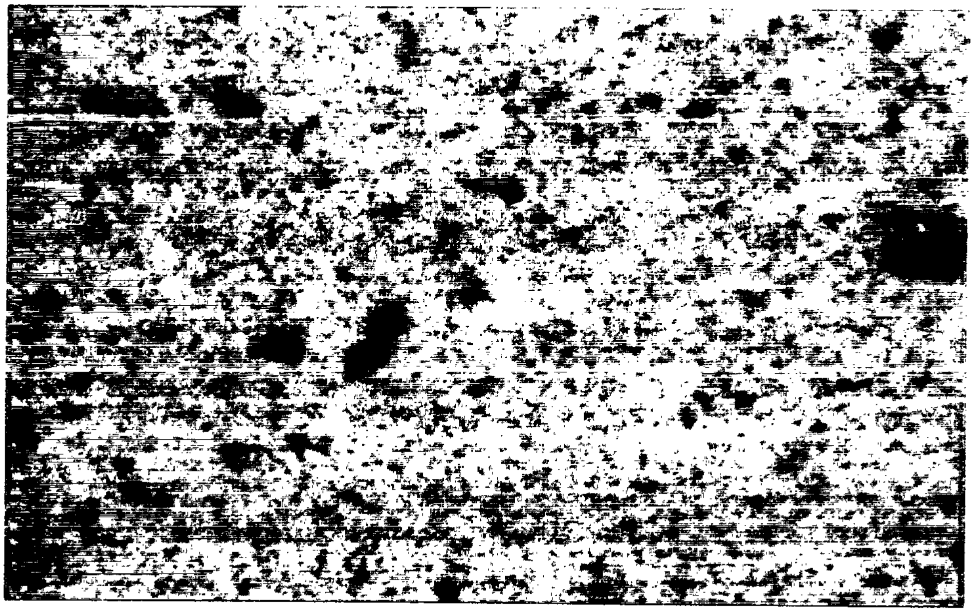

Figure 3: Reflectance PLM of steel wear particle, taken using crossed polars.

An example of the use of reflectance PLM on actual filter debris samples is shown in Figures 4 and 5. Figure 4 shows helicopter main transmission filter debris taken with 1 polar. The metal wear previously established terminology, materials with 1 refractive index are optically isotropic whereas those with greater than 1 refractive index are optically anisotropic.

Transmittance polarized light microscopy has long been used to characterize and identify particulates. It is apparent that knowledge of a particle's morphology and refractive indices could be used, via comparison to literature values, as a means to identify the particle. It is often not necessary however to quantitatively determine refractive index to permit material identification. Usually it is sufficient to estimate refractive index and the remainder of this paper focuses on just such a qualitative method.

The exact value of a material's refractive index varies slightly with the wavelength of the radiation with which it was determined, a phenomenon known as dispersion. Thus, refractive indices are correctly reported according to wavelength. The most commonly used wavelength is the D line of radiation emitted from sodium. Refractive indices determined with this radiation are reported using the familiar n_D symbol.

The angle (θ_R) by which light is refracted as it passes from one medium to another varies according to Snell's Law:

$$\sin\theta_R = (n_I/n_R)\sin\theta_I \qquad (2)$$

where n_I is refractive index of the first or incident medium, n_R is the refractive index of the refracting medium and θ_I is the incident angle of the light. Standard transmittance PLM techniques require immersion of the particulate under evaluation in a liquid of known refractive index The light used to illuminate the immersion liquid and the sample of course consists of the many wavelengths of visible light. As n_I and n_R vary with wavelength, it can be seen from Snell's Law that the refracted angle will also vary with wavelength. The magnitude of this variation will depend on the relative dispersion characteristics of the immersion liquid and particle. In general practice, the immersion liquid is selected so as to have a high dispersion, thereby ensuring a relatively significant variation in refracted angles.

In effect then, the immersion liquid and particle act as a sort of prism separating the illuminating light according to wavelength. Given the small distance that the objective is from the sample this

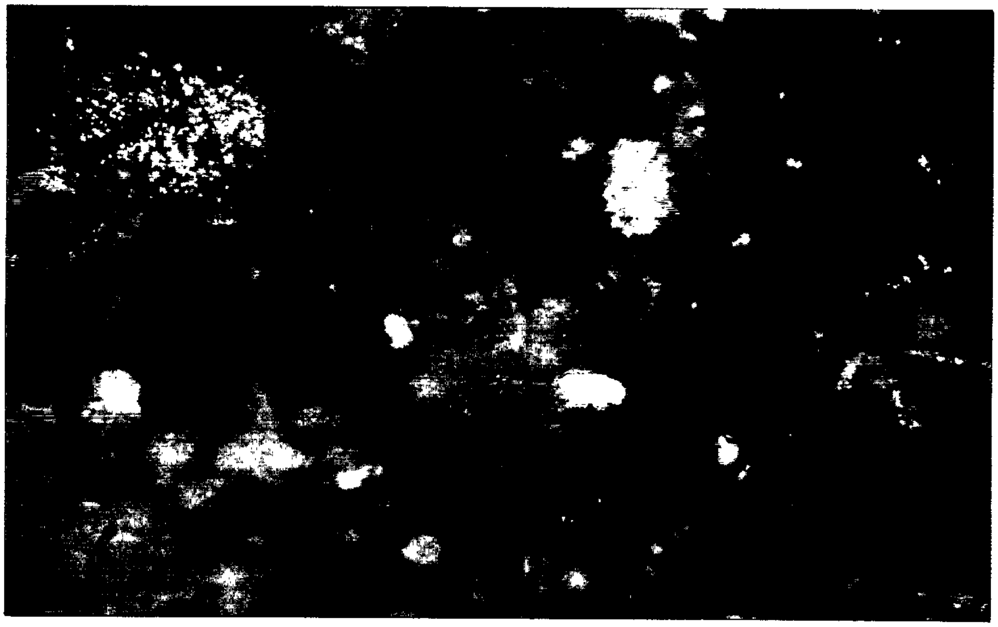

Figure 4: Helicopter main transmission filter debris, taken with 1 polar.

Figure 5: Helicopter main transmission filter debris, taken with crossed polars.

particles are readily apparent as the bright silver and gold-colored areas. Figure 5 shows the same area taken under crossed polars. The metal particles are considerably darker in this image. Note also the dramatically improved imaging of the non-metallic particles.

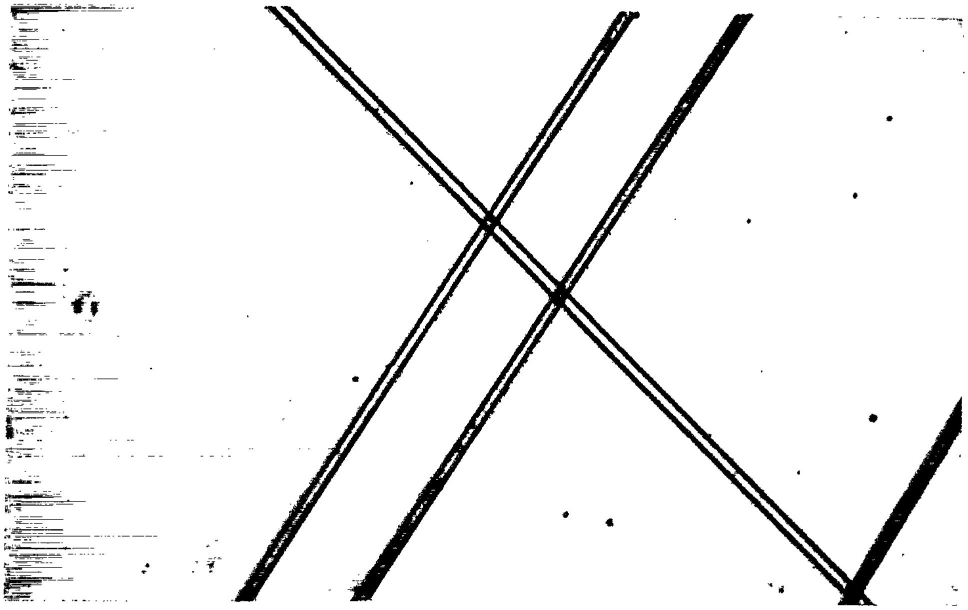

Figure 6: Glass fibers imaged using 1 polar.

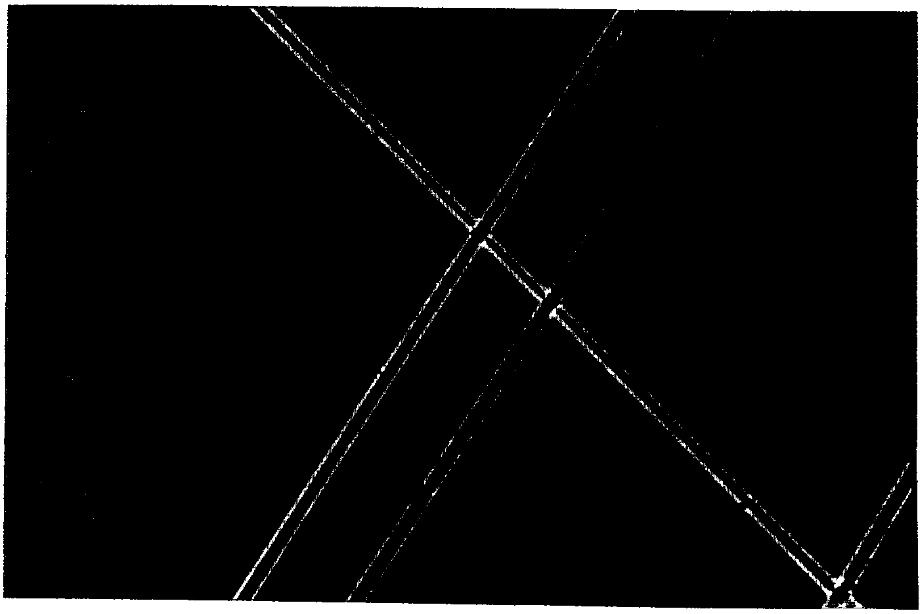

Figure 7: Dispersion staining colors of glass fibers.

Anisotropic fibers generally have two refractive indices that are oriented at right angles. One is oriented parallel to fiber length and the other perpendicular to fiber length. These are typically termed $n_{||}$ and $n_{\perp}$ respectively. Most transmission polarized light microscopes are oriented so that $n_{||}$ is determined by orienting the fiber so that it is parallel to the x-axis of the ocular. Dispersion separation effect is not normally observed. However, placement of a central stop in the objective has the effect of permitting the passage of only the most refracted wavelengths. If a relatively small band of visible light is sufficiently refracted, the edges of the material will appear colored when viewed with an objective containing a central stop. Dispersion staining colors are enhanced by removing the substage condenser and closing the substage aperture to a minimal opening, so this has become standard practice for this technique.

The width of the band of visible light that can by-pass the objective central stop is a function primarily of how close the material's refractive index is to that of the liquid. In general, the closer the respective refractive indices the narrower the band of light and the better the production of dispersion staining colors. If the indices are too far apart, significant bands of light can pass and the particle simply appears white. In practice then it is important to select the refractive index of the immersion liquid to be close to the material's expected refractive indices.

Common practice in the transmission PLM of atmospheric pollutants utilizes an immersion liquid having an n_D of 1.550. As the materials typically encountered in this type of work (silica, natural and synthetic fibers, etc.) are similar to those encountered in filter debris analysis, use of this liquid is proposed. A further advantage of using this liquid is that tables of material dispersion staining colors for this liquid exist and can be utilized for filter debris work.

An example of the use of dispersion staining colors is shown in Figure 6 and 7. Figure 6 shows glass fibers taken in 1.550 immersion liquid under typical 1 polar operating conditions. Figure 7 shows the blue dispersion colors obtained for the same glass fibers by using a central stop objective with the substage condenser removed and the aperture ramped down.

As a further example, Figures 8 and 9 show paper fibers and their dispersion staining colors taken under identical conditions. The technique can also be used for identification of fibrous materials. Note that two dispersion staining colors are distinguishable in the paper fibers. This is an indication that the fiber has two refractive indices, that is, the fiber is anisotropic. The glass fibers on the other hand have a single dispersion staining color and are therefore isotropic.

These examples indicate that reflectance PLM is able to discriminate between metallic and non-metallic particulates in lube oil filter debris. The optically isotropic metal particles appear bright when imaged using 1 polar and dark when imaged under crossed polars. Thus, an image analysis protocol whereby point by point comparison of brightness between 1 polar and crossed polar images of filter debris should permit discrimination of metallic wear particles.

It should be remembered that such an approach does not actually detect metallic wear particles. The process really discriminates between optically isotropic and anisotropic materials. Any optically isotropic particulate, such as glass particles, would respond in the same manner as metal particles and could therefore be potentially misidentified as metals. Conversely, any optically anisotropic metal, such as titanium, would not be identified by this method.

As well, it should be noted that metal oxides are typically anisotropic. Therefore, metal particles with considerable surface oxidation will not appear totally dark under crossed polars. Areas of oxidation will be imaged. Any image analysis routine developed would have to account for this possibility.

B. Transmittance Mode: Transmittance PLM is the study of optically transparent or translucent materials immersed in a fluid of known properties. In effect, it is an examination of the affect of the material on the light transmitted through it.

An important factor in this analysis is the refractive index, n. The refractive index of any material is the ratio of the speed of light in a vacuum (C_v) to the speed of the light in the material (C_m). Hence

$$n = C_v/C_m \qquad (1)$$

In general, the speed of light through any material is proportional to the atomic number of the atoms from which it is comprised. As a general rule, the higher the average atomic number, the slower the speed of light in the material and, correspondingly, the higher the material's refractive index.

Highly symmetric and amorphous materials have the same composition in all spatial dimensions and, hence, have a single refractive index. Less symmetric materials have different atomic arrangements in one or more dimension and therefore have multiple refractive indices. Using the

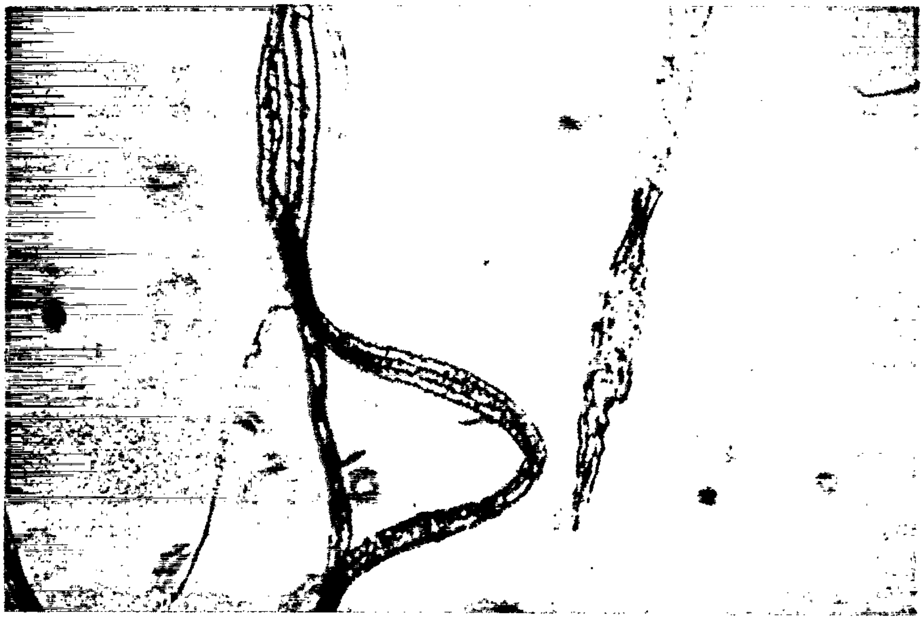

Figure 8: Paper fibers imaged using 1 polar.

Figure 9: Dispersion staining colors of paper fibers. Yellow color corresponds to $n_{||}$ while blue color corresponds to $n_{\perp}$.

staining colors are typically referred against these indices. Thus, for paper, the color for $n_{||}$ is yellow while that for $n_{\perp}$ is blue.

PROCEDURES AND EQUIPMENT: A Zeiss Axioplan polarized light microscope was used in the reflectance mode with a 5X 0.15NA strain free objective to image filter debris samples. The debris samples were from military helicopter transmissions and turbines.

A Nikon Labophot2-POL transmittance polarized light microscope was used to identify fibrous and particulate species. The employed objective was a 10X 0.25NA strain free objective with a central dispersion staining stop. All particulate or fibrous species used in the report were either reagent grade chemicals or microscopy standards.

RESULTS AND DISCUSSION:

A. Reflectance Mode: Effective use of filter debris examination as a condition monitoring technique requires accurate assessment of the quantity, size and shape of metallic wear particles. Given the large number of wear particles which can be present, determination of these parameters can be a tedious task. Image analysis routines exist which could be employed to perform these tasks. (8, 9) However, metallic wear particles often comprise only a minor portion of lube oil filter debris. (10) Therefore, utilization of image analysis requires an associated method to distinguish or separate metallic wear particles from other debris particulates.

Most metals of engineering significance, titanium alloys being an important exception, are optically isotropic. The non-metallic particulates present in filter debris typically consist of materials such as paper fibers from filter elements, synthetic organic fibers from cleaning rags, metal oxides, sand, and other similar materials. These materials tend to be optically anisotropic. Thus, PLM may be a method by which metals and non-metals could be distinguished in filter debris.

Figures 2 and 3 illustrate the affect of crossed polars on the appearance of metal wear particles. Figure 2 shows a reflectance PLM image of a steel wear particle using one polar. Note the characteristic lustrous, reflective appearance of the metal. Figure 3 shows the same particle taken under crossed polar conditions. Note the now dark appearance of the metal particle and the enhanced appearance of the background particulates.

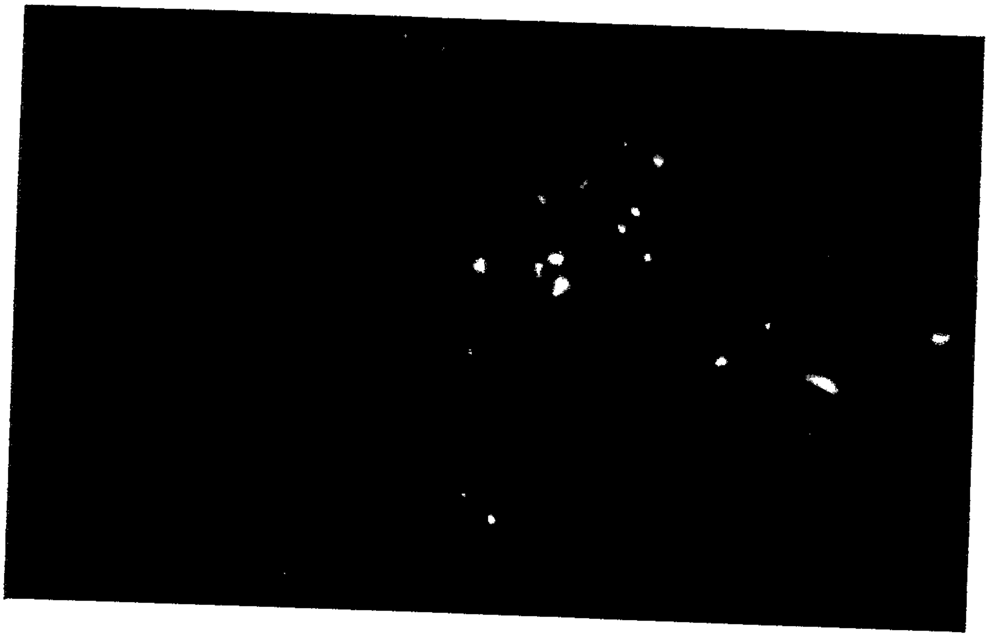

Figure 10: Dispersion staining colors of silica.

Dispersion staining colors can also be determined for particulates. Figure 10 shows the blue colors characteristic of silica particles.

It should be noted that the glass fibers and silica particles have similar dispersion staining colors. This is not too surprising as both materials contain SiO_2 backbones. Thus, dispersion staining colors by themselves are not sufficient to identify a material. Positive identification must rely on determination of morphological characteristics. Fortunately, tables exist that list these and other factors that can be used in material identification. An example of such a table is given in Table I and contains morphological characteristics and dispersion staining colors for some materials commonly found in lube oil filter debris.

CONCLUSIONS

Filter debris examination is a relatively recent field in which metals, minerals and atmospheric pollutants are commonly encountered. It stands to reason that techniques long successfully employed in metallurgical, mineralogical and atmospheric pollutant analysis could be employed in

TABLE I
PARTICULATE IDENTIFICATION

PARTICULATE	MORPHOLOGY	DISPERSION STAINING
Salt (NaCl)	cubic crystals	pale blue
Silica (quartz)	glassy flakes	blue to blue-magenta
paper fibers	flat ribbons	$n_{\|\|}$ - yellow $n_{\perp}$ - blue
glass fibers	straight rods	blue
cotton fibers	cylindrical fibers with twists	$n_{\|\|}$ - yellow $n_{\perp}$ - pale blue-
nylon	any shape, but generally rod-like	$n_{\|\|}$ - blue to magenta
polyester	any shape, but generally rod-like	$n_{\|\|}$ - white $n_{\perp}$ - pale blue
polyolefin (carpet fibers)	shredded ribbons	$n_{\|\|}$ - yellow to pale blue
gypsum	monoclinic rhombs	pale blues
calcite	jagged rhombs	pale yellows to white

this field. This paper attempted to introduce one such technique, PLM, to the filter debris examination community.

Reflectance PLM was demonstrated to be useful in discriminating metallic particles from nonmetallics. The benefit of such discrimination may not be immediately obvious as metal particles are usually readily identifiable visually by a human operator. However, the tediousness of such tasks have raised the possibility that artificial intelligence methods could (or should) be employed. Discrimination of metal particles has proven to be anything but trivial via these methods. Reflectance PLM is a potential method to overcome this problem.

Transmission PLM was demonstrated to be useful in the identification of mineralogical particulates and atmospheric pollutants. While filter debris examination is usually thought of as an examination of metal wear particles, occasionally the nonmetallic components of filter debris can provide clues towards the operating wear mechanisms. The presence of hard, brittle species, such as sand, glass, etc., may indicate excessive abrasive wear. The presence of salt may indicate component corrosion. The presence of fibrous materials may indicate filter media breakdown or sloppy maintenance practices. The ability to identify such nonmetallics would therefore be of benefit, and transmission PLM provides a means to accomplish this.

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POLARIZED LIGHT MICROSCOPY AS A FILTER DEBRIS ANALYSIS TOOL

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