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THE ROLE OF LIGHT MICROSCOPY IN AEROSPACE ANALYTICAL LABORATORIES

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ABSTRACT

Light microscopy has greatly reduced analytical flow time and added new dimensions to laboratory capability. Aerospace Analytical Laboratories are often confronted with problems involving contamination, wear, or material inhomogeneity at levels that would not concern other industries. The detection of potential problems and the solution of those that develop necessitate the most sensitive and selective applications of sophisticated analytical techniques and instrumentation. Cost and often more important, time effective analyses that optimize a diverse laboratory's capabilities must be used. This inevitably involves light microscopy, no other single instrument is as versatile a starting point for solving Aerospace contamination problems. The microscope can characterize and often identify the cause of a problem in 5-15 minutes with confirmatory tests generally requiring less than one hour. When an identification cannot be made the characterization indicates which instruments in the laboratory can most efficiently identify the source of a problem with the aid of the microscopic data. Assemblage analysis, the identification of the percentage contribution of multiple contamination sources, is impracticle if not impossible using any other instrument or combination of instruments. Light microscopy has made and will continue to make a very significant contribution to the analytical capabilities of Aerospace Laboratories.

INTRODUCTION

The microscope does much more than magnify. It is a sophisticated optical instrument capable of working with very small samples and characterizing them by the effects their electron distributions have on the visible electromagnetic

spectrum. Electron orbital energy, spatial distribution, density, and electron freedom all result in optical effects measurable with the microscope. An experienced microscopist can identify a large number of compounds by these observed effects. Those not identified can be characterized for future identification. These effects are so sensitive that careful measurement of them in known materials can detect spatial or crystal faults, molecular weight changes, internal stress, degree of crystallinity, trace impurities and other conditions which alter electron configurations. All of this can be done nondestructively and within a relatively short period of time.

Even with the most modern laboratory wet chemical techniques involving the use of the microscope can be important. As few as 1000 atoms of an element can be detected using microscopic wet chemical methods. Microcrystal tests are among the most widely used techniques for the qualitative identification of organic functional groups. These tests are based on the characterization of the crystalline products from the reaction of an unknown and a known, specific, reagent. If the form or progress of a chemical reaction is of interest the microscope offers a method of approach not offered by any other instrument. Solubility studies, thermal phase transformations, rates of change and other physiochemical properties of compounds can be studied to great advantage microscopically.

Many industrial, as well as natural materials, have characteristic morphologies. Common biologicals, pollens, spores, diatoms, insect parts, hair, plant parts, plant ash (tobacco), and other materials are sometimes encountered in contamination samples. The recognition of them with some knowledge of their natural history can often help solve a contamination problem. Industrial artifacts from production, as well as the products themselves, often have important microscopic morphologies. The application of the microscope to metallurgy, ceramics, and microelectronics are examples of the importance of morphologies to products. Industrial artifacts as contaminates also exhibit helpful diagnostic morphologies. Wear particulate in a hydraulic system has a shape characteristic of the type of wear that produced it. The recognition of that shape can help identify the type and extent of wear. Airborne industrial artifacts such as paint spheres, metal grinding or cutting residue, weld residue, tire wear, etc., can help indicate the origin of contaminates in a controlled environment. These materials within a part can indicate a control or a cleaning problem during assembly.

Figure #1 is a graphic representation of some of the techniques in Light Microscopy. Most of these techniques are applicable to common Aerospace contamination problems. The theory and mechanics of using these techniques is much to large a task to be covered in this paper but the bibliography at the end will hopefully be of value to those interested in pursuing analytical microscopy. The purpose of this paper is to point out the added capabilities as well as the cost effectiveness that resulted from our increased utilization of the light microscope.

EQUIPMENT AND SETTING

The Light Microscopy Laboratory, also known as the Particle Identification Laboratory, is part of a much larger laboratory complex. Instruments available in the general laboratory include:

Liquid Chromatograph	Gas Chromatograph
GC/Mass Spectrograph	Scanning Electron Microscope
Emission Spectrograph	Transmitting Electron Microscope
Fourier Transform Spectroscopy I.R.	Electron Microprobe
Standard I.R. Spectrograph	Energy Dispersive X-ray
X-ray Diffraction Laboratory	Fast Neutron Activation
Differential Scanning Calorimetry	Atomic Absorption
Thermal Gravimetric Analysis	General Wet Chemistry Lab
	General Physical Test Lab
	Differential Thermal Analysis

The microscopy laboratory has a Bausch & Lomb and a Nikon Zoom Stereomicroscope, a Nikon Apophot Research Microscope, a Nikon LKE Research Microscope with a Mettler hotstage, a Carl Zeiss Episcopic Brightfield/Darkfield Research Microscope, two laminar flow benches, a large centrifuge, sedimentation and density gradient equipment, and various other support equipment for sample collection and preparation. The research microscopes have polarized light, phase contrast, and numerous interference technique capabilities as well as a wide range of illumination systems. A 2000 slide standards cabinet is an additional piece of equipment very important to light microscopy. The Microscopy Laboratory consists of two rooms: one 19 x 14 feet and one 7-1/2 x 13 feet. The Laboratory is located in the 2-01 Building of the Boeing Aerospace Plant II complex in South Seattle.

OPTICAL MICROSCOPY Optical Crystallography Photometry Interference Microscopy Dispersion Staining	CHEMICAL MICROSCOPY Hot or Cold Stage Microscopy Microcrystal Tests Ultramicro Analysis Environmental Cell Microscopy
MORPHOLOGICAL MICROSCOPY Optical Metallographics Biological Microscopy Wear Analysis Counting Analysis	SUPPORT TECHNIQUES Sample Preparation Particle Manipulation Density Analysis Sedimentation Analysis

FIGURE #1

The Field of Light Microscopy as Used in This Paper Includes the Body of Techniques Seen Above.

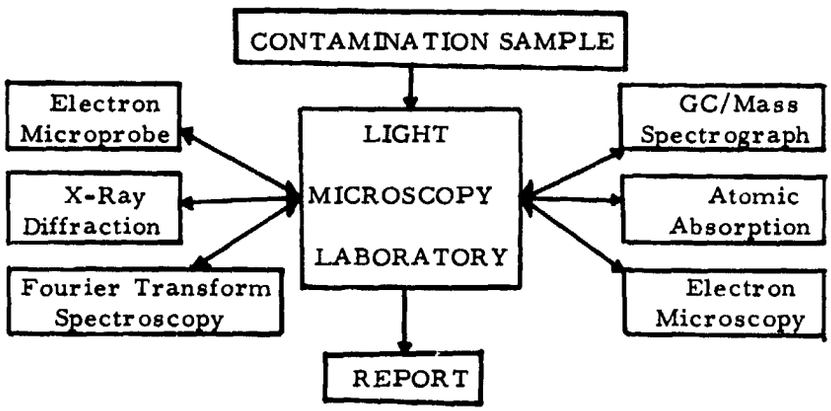

FIGURE #2

Flow Chart for the Analysis of a Contamination Sample.

APPLICATION

Three hypothetical case histories are presented here. They are based on actual case histories but developed to emphasize general techniques in contamination identification rather than specific results. The first is a typical hydraulic sample sequence, the second is a corrosion problem, and finally an airborne contaminate problem. Essentially these samples all follow the same flow diagram as represented in Figure #2.

PROBLEM 1 - Hydraulic Oil Particulate Contamination

One hundred milliliters of a MIL 5606 hydraulic oil were filtered through a cellulose ester membrane filter and the particulate was counted per ARP 589. The oil failed to meet the required particulate cleanliness level and was brought to the Light Microscopy Laboratory for evaluation. Oil remaining in the sample bottle was poured into graduated, clean, capped centrifuge tubes and centifuged. The clean oil was then poured into a clean sample bottle for later analysis as required. The particulate in the bottom of the tube was washed with clean kerosene and centrifuged twice more. A final wash was made with chlorobenzene and the particulate was entered into a density gradient tube. The filter used in the particle count was examined using a stereoscopic microscope for particle distribution and an estimate was made of the sample complexity, type of material, and quantity of each type. A clean cork bore, #8, was selected and a portion of the filter was punched out to be cleared and mounted. The filter was cleared using a 1:1:1 solution of hexane, 1,2 dichloroethane, and dioxane. This was then mounted in a permanent mounting media and covered with a coverglass. This sample was then examined under transmitted polarized light, brightfield illumination and oblique top light.

Common natural or industrial minerals were tentatively identified and their locations on the slide recorded. Morphologically identifiable materials such as biologicals, tooling residue, wear metal, or industrial artifacts were recorded along with their position. The density gradient column had equilibrated by this time and the particulate was removed by density to confirm the quantitative estimates and the identification of common materials. Wear metals were mounted for electron microprobe analysis to identify the alloy type wearing and crystalline materials requiring identification were mounted for X-ray diffraction or microprobe analysis depending on size. Elapsed time to this point was one hour. Generally 15-30 compounds have been identified or charac-

terized by this time and an assemblage analysis has been completed indicating a rough percentage distribution of particle types from system wear, airborne fallout, and industrial activities. Within another few hours, depending on the number of particles requiring additional analysis, the alloy types experiencing wear will be identified. In another 48 hours, X-ray data will identify the few remaining compounds.

A typical turn around time for a sample of this type is two to three hours with one or two wear metal alloy types identified. Total manhours expended, 4-6 hours including the typed report. The filter, particulate, and mounted slide are retained for future reference by program with new standards prepared as necessary. If a large number of samples are being examined from one program the analysis time can often be reduced to under one hour.

PROBLEM 2 - Corrosion

A magnesium part fabricated by a subcontractor was received in an airtight, clean, sealed drum. Upon opening the drum, water and oil was found and the part exhibited some corrosion. The part was photographed to document it's condition, then a small sample of the corrosion product was collected, as was a sample of the oil and water. The part was resealed after drying in a clean barrel with excess desiccant. The samples were delivered to the Light Microscopy Laboratory for evaluation. Two questions were asked of the laboratory: (1) What caused the corrosion? and (2) Is the corrosion product stable in its new environment? Paint chips, and other materials were found on initial examination as well as different colored corrosion materials. The corrosion material varied from a waxy to a hard tough substance. Under crossed polarized light most of the material was demonstrated to be isotropic (cubic or amorphous), or microcrystalline (crystals a micrometer or less in greatest dimension). The two main types of corrosion material were mounted for X-ray diffraction and electron microprobe analysis. The results from X-ray diffraction were inconclusive, no apparent crystallinity was indicated even in the material identified as microcrystalline by microscopic examination. Electron microprobe analysis identified carbon, magnesium, cadmium, potassium, iron, zinc, with traces of chlorine and sulfur in the microcrystalline material and magnesium, zinc, sodium, oxygen, sulfur, and chlorine in the other particle. The microcrystalline material charred in the electron beam, the other particle did not. Under the microscope an acetone extraction was made. The microcrystalline material was leached by the acetone and the evaporite was collected on the

tip of a fine tungsten needle. The needle with the sample was entered into the direct probe of the mass spectrograph for analysis of the organic material. With the microscopic identification of chemically altered paint films and the analysis of the oil found in the barrel a cause for the corrosion could be hypothesized. The water and oil was of a type that could have come from an unfiltered machine shop compressed airline. During shipping the trolley that held the part rubbed through some of the paint on the inside of the barrel exposing the metal to corrosion. Water vapor began setting up a galvanic attack between the magnesium part and a cadmium coated washer used on it. This initial corrosion product complexed with iron and zinc from the barrel and organics from the paint and the oil. The material as it then existed was somewhat unstable as indicated by the X-ray and mass spec. data. Stored in a dry environment with excess desiccant any additional corrosion possible would be negligible compared to that which had already occurred, though the corrosion product may change. Total hours charged: 10; turn around: 72.

PROBLEM 3 - Airborne Contamination

A class 200,000 particle/cubic foot room failed to meet that criteria. A volumetric sample collected on a cellulose ester membrane filter was brought to the Light Microscopy Laboratory. The filter was examined using the stereomicroscope and then cleared using the same technique mentioned earlier with the hydraulic oil example. This NO SMOKING area had a recirculating air system but cigarette ash was identified in the sample. The sample was also found to contain a large amount of opaque black particulate. Some of the black particulate was in the form of shiny spheres with very low density. The area surrounding the room was surveyed to characterize external sources of particulate. The blower for the air system was located outside the room about 20 feet above the floor on a wall that had no entrance into the controlled area. Below the blower and about 15 feet west was a copy station with Zerox and IBM copiers. These machines were in almost constant use with a line of people awaiting their turn. Many of the people were smoking cigarettes. A volumetric sample from that station exhibited a similar distribution of particulate types indicating this area as the major source of particulate. The melting point of the toner, as well as its appearance and density, matched that of the black spheres found in the controlled environment room. An examination of the air duct above indicated that during a recent overhaul of the blower

a taped joint in the duct had been broken and not repaired. Outside air was being drawn in through this crack in the duct and was causing the area to fail. The duct was repaired and the problem disappeared. Total hours charged: six including the report; turn around time: four hours.

The prior three examples demonstrate the relationship between the light microscope and other instruments available for use in the general laboratory complex. The majority of the work done in the microscopy laboratory does not require additional instrumental analysis. The identification of a diatomaceous sludge in the bottom of a cooling water reservoir pinpoints the source and suggest the solution to a rapid increase in suspended solid loads. A density separation followed by a magnetic separation of the dense material and a hardness separation of the light material determines whether the black oil suspended particulate coming from a joint is metal wear, rubber gasket wear, graphite lubricant, or molydisulfide lubricant. These last two examples required five minutes each from receipt of the sample to verbal communication of results. No other method of analysis is capable of that speed or sufficiently versatile to identify all five of the materials represented. It is this versatility that results in many thousands of dollars of cost avoidance and cost savings each year.

DISCUSSION:

On sight a mounted particle under the objective of a polarized light microscope exhibits the following characteristics:

Color	Homogeneity	Refractive Index Relative to the Mountant
Shape	Types of Inclusions	
Cleavage	Crystallinity	Size
Fracture Type	Luster	Relative Light Absorption

With a little more work the crystal type can be identified, as well as its index of refraction in given directions, its dispersion, density, solubilities, melting point, elemental composition, etc. The sample can then be quantified by count of each particle type, by area, or by volume and density. All of this can be done using microscopic techniques but for a few materials other instrumental techniques may be quicker. The effective use of the light microscope is the result of balancing its capabilities with those of available instruments. Many materials are sufficiently distinct to be identified by those features which are quickly recognizable by a trained microscopist. For those that are not so quickly

identified or that require further characterization it is important to balance microscopic and instrumental time with cost effectiveness. With the results of a quick microscopic overview an analytical approach for a specific particle type is often obvious. It is this quick overview by a trained microscopist that results in the greatly enhanced analytical efficiency.

Prior to our increased utilization of the microscope many problems resolved within minutes microscopically required many hours, even days using other methods. If a serious problem was encountered samples were sent to every instrument, tying up instruments and personnel for extended periods of time, often in an attempt to analyze materials not suitable to that piece of equipment. Light microscopy has resulted in a considerable improvement over the old approach.

Light microscopy as a general analytical tool is not taught on any educational level in this country with few exceptions (the training offered by McCrone Research Institute, 2820 S. Michigan Ave., Chicago, Ill. 60616, is one of the most notable exceptions). Most universities offer courses on the application of the microscope to biological sciences, geological sciences, etc., but none on the microscope itself as an analytical instrument. At Boeing we are now actively documenting, training and implementing microanalytical techniques for routine use. In this way we expect to have a broad base of experienced personnel within the Company and a thorough documentation of applicable techniques.

CONCLUSION:

The versatility of the microscope and the available non-instrumental techniques of micro and ultramicro analysis can supplement any laboratory responsible for the testing of a wide variety of materials. This is particularly true of Aerospace contamination problems. The greater utilization of light microscopy, with its related techniques and improvements in instrumental interfacing has resulted in decreased analytical flow time and significantly increased data collection.

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