

Optical Microscopy

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Optical (light) microscopy is a powerful aid in defect and problem identification and root cause analysis. The author considers it the best single tool in problem solving. This article describes the main types of magnifying devices used in the coatings industry, including low magnification lenses, stereomicroscopes, and compound microscopes. It discusses reflected and transmitted light microscopy and the use of different kinds of illumination, particularly bright field and dark field. It reviews specific applications, including the examination of surfaces, fibers and other particles, cross sections, and liquids. Ancillary equipment such as cameras, hot and cold stages, and reticules (reticles) are mentioned. The final word is that as useful as the microscope is, users should be careful in drawing conclusions strictly from what they see. You can be fooled!

INTRODUCTION

Coatings chemists, technologists, and service people face many problems in their day-to-day work—defects and dirt on freshly applied paint, color that is not the correct shade, field failures, and unexpected corrosion. Although I am convinced that the most important problem-solving tools still are eyes and minds, there are limitations to what we can see. We need to be able to extend our vision. That is where optical (light) microscopy comes into play.

Microscopes and related devices enable us to magnify and resolve what we are seeing. Is that speck of color on the part or panel rust, dirt, or a pigment particle? Is that defect a crater or a pop? Does it originate in the substrate, primer, or topcoat? Has the part been painted more than once? Is the pigment in the off-color paint flocculated or only partially dispersed? What is the particle size of a pigment dispersion or latex? These and other questions can be answered using optical microscopy. Light microscopy does not usually provide chemical information (exceptions

being the identification of fibers and minerals), but it can be used to select areas for analytical techniques such as x-ray, FTIR, and SEM. I need to point out that I am not a microscopist, but a paint chemist who has used microscopes in the definition and solving of problems for 30 years. I consider a light microscope the most valuable single tool for solving coatings problems. In addition, I firmly believe that all paint chemists and technologists should become familiar with microscopes and learn how to use them. More information on microscopes and microscopy can be found in the literature¹⁻⁶ and in web sites on the Internet.⁷⁻⁹

THE TOOLS

Low Magnification Devices

Magnifying devices range from simple, inexpensive hand lenses to large compound microscopes with video cameras and image analysis software. Each has its place and each can be very useful. Low power magnifying glasses

and hand lenses (2–10×) are highly portable and can be carried in a pocket or briefcase for use in the field, shop, or plant. I often have used a 7× hand lens for looking at surface defects, field failures, weathering effects, and corrosion. However, such devices are limited in their magnification and resolution (resolution or resolving power is the ability to see two points as separate points). They are better than the naked eye, but not by much. The next step up is a 40–60× shop microscope, a relatively inexpensive (~\$300) device that can be very useful in the field. Higher power shop microscopes are available, but I find them too difficult to focus, particularly in a plant situation where the ob-

Figure 1—Common Main Objective (CMO) type stereomicroscope.



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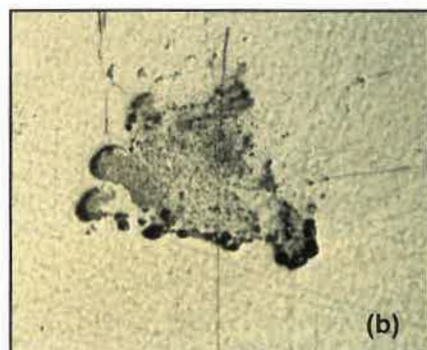
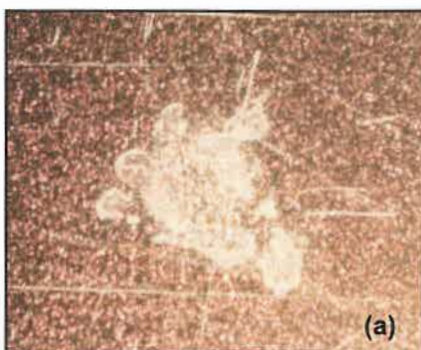
jects of interest may be moving. Besides that, their poor resolution does not justify using a higher magnification. There is no sense in magnifying an image that is not sharp. In addition to low magnification and poor resolution, low magnification devices rarely allow documentation of images. A shop microscope-digital camera combination can be rigged, but usually takes some ingenious design and machine shop work. However, I have seen surprisingly good pictures produced by such a set-up.

Stereomicroscopes

Most stereomicroscopes also are relatively low magnification instruments (roughly in the range of 2–100×), but high magnification is available. Regardless of magnification, they give much higher resolution than the devices described in the previous section. Stereomicroscopes are very useful for the examination and documentation of surface defects, dirt, fibers and other material trapped on filters, weathering effects, etc. The images produced may be all that is needed or may lead to further investigation with a higher power optical microscope or other techniques. The original stereomicroscope design (still in use) consists of two parallel compound microscopes side by side and often is called a Greenough microscope named after its inventor. One eye looks through one microscope, the other eye looks through the other. If the instrument is adjusted properly, the viewer sees a three-dimensional image. A newer design is the common main objective (CMO) type (Figure 1), which uses a single large objective lens. The coatings industry and its customers use both types, but the CMO instruments dominate in analytical and R&D laboratories. Although more expensive, the high resolution of these microscopes and the wide range of optical and lighting accessories available for them make them the choice for research and problem solving.

Older stereomicroscopes may have one or more fixed sets of objectives, but modern ones have zoom lenses with click stops or continuous adjustment knobs. Photographs are taken through one of the two eyepieces or a third, trinocular, tube. Sample preparation is minimal, although parts or panels may be cut for easier handling. Stereomicroscopes have long working distances (the distance from the object

Figure 2—(a) Acid etch on a clearcoat; (b)—Same defect with polarized light.



to the specimen), up to 15 cm with some instruments. Extended-height stands enable microscopes to be raised to allow examination of large specimens. In addition, stands with cantilever booms are available that make it possible for the microscope to be swung out over parts. Because of these accessories, relatively bulky parts and assemblies can be examined. This is particularly useful in a plant situation where parts will not be sacrificed and cut up to be examined, but will be sanded and repainted.

As with all observations, the illumination of the specimen is critical. It is common to use side lighting with a fiber optic light source, particularly at a low angle, to better see the topography of a surface. Many stereomicroscopes have internal coaxial lighting such that light passes through the objective to illuminate the specimen. This light may be polarized internally with another (movable) polarizer being attached to the objective. The light usually comes from above and is reflected from the specimen. However, some stereomicroscopes also are set up for transmitted light. If not, it is possible to rig lighting from underneath so as to look at a film, filter, or other specimen with transmitted light. The movable polarizer attached to the objective can be turned to change the polarization of the light that strikes the specimen. Without this ability, defects may be difficult to see, particularly on clears over basecoats, or it may be difficult to decide whether the defect is in the clear or the basecoat. Proper use of the polarizer can prevent or reduce reflection from aluminum flakes and other pigments in basecoats and allow the production of good images of the surfaces of clearcoats. Figure

2 shows images of acid etch on an automotive base/clear. Figure 2a is shown without the polarization effect; Figure 2b is shown with polarized light. The etch damage is very obvious in both images, but 2b proves that the defect is on the surface. In addition, 2b shows texture that is not visible in 2a.

Compound (Conventional) Microscope

Nearly all microscopes are compound (have more than one lens) these days, but the term compound microscope tends to be reserved for higher power, non-stereomicroscopes such as the one shown in Figure 3. A compound microscope is an instrument that can

Figure 3—A typical transmitted light compound microscope with a bright field condenser.³

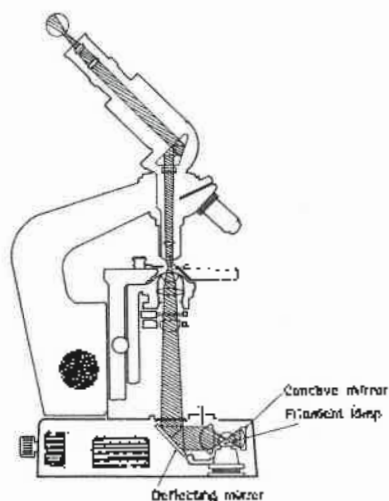
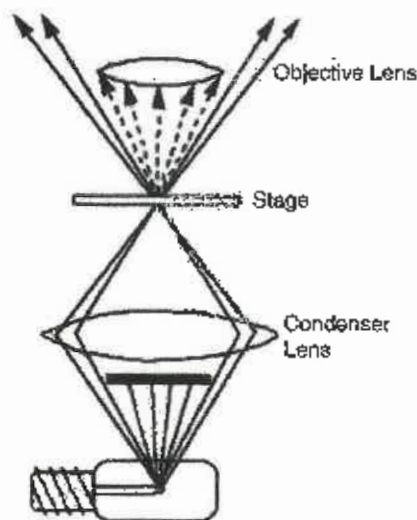


Figure 4—Dark field condenser arrangement.



produce images at high power (100–1000 \times) and at high resolution. Many of them have the ability to polarize the light that strikes the specimen. The illumination can come from above so that the specimen is observed via light reflected from it or from below (as in Figure 3), so that the image is a result of the light that is transmitted through the specimen. Light passes from the specimen through an objective lens and then to the eye through a second magnifying lens called an ocular or eyepiece. In focusing, the microscope or stage is moved up and down using coarse and fine focus knobs. Nearly all compound microscopes have revolving turrets with three or four objectives that can be moved

into position as needed. The eyepieces usually are 10 \times and a useful set of objectives is 10 \times , 20 \times , 50 \times , and 100 \times . For the nominal magnification, multiply the objective magnification by that of the eyepiece. However, internal lenses, particularly those in camera connectors/converters, can affect the number.

Compound microscopes are available in a bewildering range of models with numerous accessories. Fortunately, many components and accessories can be added on, so a relatively basic instrument can be purchased and upgraded when needed. The main initial choices are whether to buy a metallurgical (reflected light) microscope, a biological (transmitted light) microscope, or one that combines both capabilities. I recommend the third choice because the combination microscope can look at just about anything—surfaces, particles, cross sections, wet paints, latexes, etc.

Reflected light images are useful in the examination of coatings defects, weathering effects, field failures, damage from impacts and abrasion, fibers and dirt particles, etc. The usual way is to simply view the image reflected from the surface. However, lighting and contrast can be improved by using modifications such as dark field, phase contrast, and interference contrast. For example, zinc phosphate crystals on steel are transparent and essentially invisible under normal lighting, but dark field and interference optics allow them to be seen.

Transmitted light microscopy is well known as a technique for studying biological specimens, but also is useful for the examination of paints, latexes, free

films, and fibers. The light source for transmission microscopy usually is a moderate-to-high intensity lamp, but the light must be focused via a lens beneath the stage called a condenser. Some condensers are fixed in position, but most can be raised or lowered to improve the focus of the light. In bright field microscopy, the light is directed through the specimen. Particles in the specimen block light, so they are dark and the background (the field) is bright. A bright field condenser usually includes an aperture diaphragm, a device that controls the brightness and contrast by adjusting the diameter of the light beam coming up through the condenser. Usually the best position for a bright field condenser is very close to the stage. The diaphragm is adjusted to give a bright image without blinding the observer. Initial examination often is done with a lower power objective (such as 10 \times) for easier focusing and an overall view of the specimen, and then a higher power objective is moved into place.

A transmitted light microscope also can employ a type of illumination called dark field, which was briefly mentioned before. To produce the dark field effect, light is blocked from passing directly through the condenser by an opaque disk in the center (Figure 4). This may be done with a special condenser or by modification of a conventional bright field condenser. With the center blocked, light is directed toward the edges of the condenser and an inverted hollow cone of light is focused onto the specimen. Only light that is scattered by the specimen can reach the eye. The result is that the background or field is

Figure 5—Wrinkling.

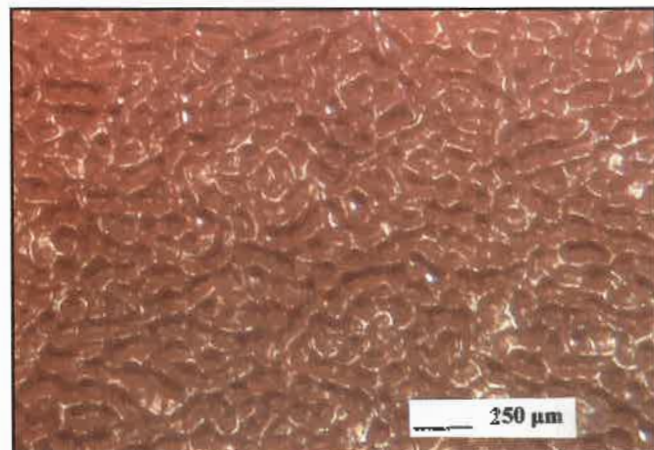
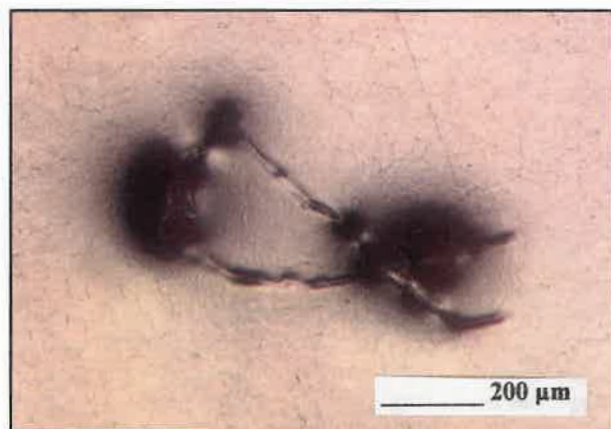


Figure 6—Gassing from a plastic substrate.



Figure 7—Fiber on the surface of a primer.



dark and many particles show up as bright white. Visibility is enhanced by the contrast between the bright specimen and the dark surroundings. Pigment particles also are brightly lit, but the color may not be accurate. Dark field microscopy is very useful for looking at paints, latexes, and waterborne resin dispersions. In dark field, positioning of the condenser is more critical than in bright field, however, and it must be moved up and down to find the best image. Focusing with dilute dispersions sometimes is difficult because there is so little material present and because dust particles on cover slips can cause confusion because they resemble latex or dispersion particles.

Polarized light is a valuable addition to compound microscopes. It greatly helps the viewer in the identification

of fibers, pigments, and other crystalline materials. It cuts down on glare and bright reflections from surfaces and enables the viewer to see defects more clearly.

ANCILLARY TOOLS

Cameras and Image Analysis

It is very important to be able to record microscopic images for future reference, to show others, and to in-

sert into reports, presentations, and other documents. Records can be made via photographs (Polaroid, conventional film, or digital), videotapes, and computer files. Image analysis software can be used to edit and upgrade images, count and size particles and make other measurements, such as film thickness, as well as aiding in the archiving of the images.

Hot and Cold Stages

A number of devices for heating or cooling specimens being examined under the microscope are available or can be built. Depending on the cost and complexity, temperature control may be approximate or very exact. Hot stages are useful for measuring melting points of crystalline raw materials and contain-

inants, degradation temperatures of materials, etc. Wet coated coupons baked on a hot stage or very small hot plate provide a means to simulate bakes and reproduce defects or other problems that occur during a bake. A video camera should be used to record the process. Cooling can be employed to force crystallization or coagulation, look at the effect of freezing, etc.

Reticules

A reticule (also reticle), or eyepiece micrometer, is a clear glass insert with a scale or other lines or shapes inscribed on it. The reticule sits right at the focal plane inside the eyepiece lens of the microscope and allows the investigator to make accurate measurements and/or positioning of specimens. Reticules come in many varieties and sizes. The most common type, built into one eyepiece on most microscopes, is a simple cross line (crosshair) arrangement. The most common measuring reticule is a straight line scale, a ruler in miniature. Other types have square grids, concentric circles, or circles of different sizes (useful for sizing pigment and latex particles). The distance between any two marks on a scale reticule or the diameter of a circle depends only on the objective. Reticules must be calibrated, usually with a stage micrometer, a microscope slide that has very small marks of known dimensions inscribed on it. The slide is viewed with the microscope and the true distance factor noted for the reticule with the given objective. This should be done for each objective

Figure 8—Crater.

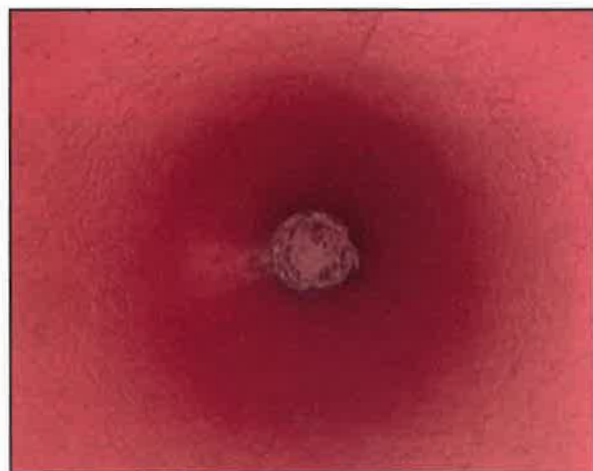


Figure 9—Cross section of the crater.



Figure 10—Cross section of an automotive coating showing overspray particles in the clearcoat.



Figure 11—Cross section of a four-layer automotive coating on steel.



lens and a sheet with the factors should be posted where users can see it. A cruder, but less expensive method useful at lower powers is to use a plastic ruler, some of which have accurate scales in 0.3 mm divisions.

SPECIFIC APPLICATIONS

Examination of Surfaces

When specimens can be brought into the lab, surface defects and field failures should first be examined and documented with a stereomicroscope. Experience is the best teacher, but it is useful to begin the examination at low power where focusing is easy and a fairly large area can be seen. The investigator can then carefully zoom up to higher powers. Lighting angles and intensities should be varied and observations made with and without coaxial light and a polarizer so as to produce the best images of the defect or problem. Figures 5 and 6 show examples of surface defects as they are seen with a microscope.

Examination of Fibers, Dirt, and Other Particles

Occasionally, a particle in or on a surface can be identified directly. Examples include weld balls, rust particles, overspray, and determining whether dirt is a fiber or a chunk of material, etc. However, in many cases, and particularly if a more exact identification is desired, the particle must be removed (usually with a sharpened needle and tweezers) and observed on its own. Through the use of a polarizing microscope, fiber types can be identified and compared to possible sources of such contamination, such as tack

cloths, wipe rags, filters, paper products, clothing, etc.¹⁻² Figure 7 shows a fiber in a primer. This fiber is well covered by paint, but many are not, and even covered fibers often can be separated from the coating. References 9 and 10 provide useful information on the examination and manipulation of defects, particularly dirt.

Cross Sections

With some defects and problems, viewing of the surface does not allow definition of the problem. For example, cratering, solvent popping, and gassing from substrates all can produce very similar holes in coatings. It is important to see where the hole originates, whether the hole is at the top of a bubble, etc. This is best done by cross-sectioning the specimen. In the simplest method, the part or panel is cut at right an-

gles to the surface, the cut face sanded with finer and finer paper until the center of the defect is reached (a stereomicroscope is useful in keeping track of where you are). Depending on the situation, the edge may or may not be polished. For coatings on metal, the cutting is best done with a diamond saw, and the sanding and polishing with papers and cloths on a rotating wheel. However, in auto plants, I have cut parts and panels with hacksaws and jeweler's saws and sanded the cut edge on sheets of sandpaper using a figure-eight motion and produced adequate cross sections. Painted plastic and paint chips can be cut with a sharp knife or scissors and the edge may not need further treatment. Painted wood is easy to cut, but also easy to damage. Sanding of edges must be done carefully to avoid smearing of details such as interfaces between the coating and the wood, whether a stain or other coating has penetrated into the wood, etc. Simple cross sections are viewed at 100–200× with a compound microscope, often with both coaxial lighting and side lighting or alternating them to find the best image. Figure 8 shows a crater in an industrial coating that looks deep and ugly (and appears even worse to the eye). Figure 9 provides a cross-sectional view at the same magnification and shows that, unlike crater diagrams in many publications (including my own), the sides of this crater slope very gently and the crater is quite shallow.

Metallurgists and most microscopists consider the cut/sand/polish method as too crude for their use. They prefer to embed specimens. This involves cutting a small coupon that includes the defect and placing the coupon at right angles

Figure 12—Waterborne resin dispersion after aging.

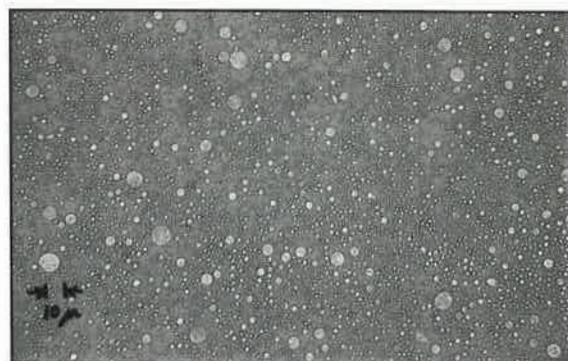


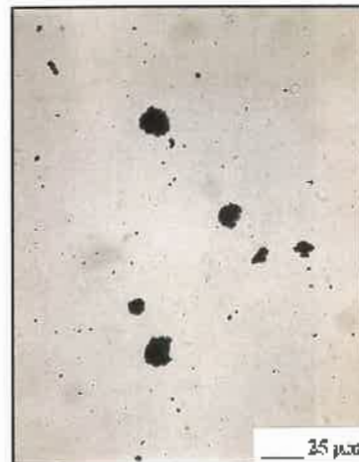
Figure 13—Bright field images of an electrodeposition paint: (a) initial fill, (b) flocculated, (c) badly flocculated.



(a)



(b)



(c)

on the bottom of a small cup, to which is added an epoxy, polyester, or acrylic casting compound." After cure, the specimen, now embedded in hard plastic, is removed from the cup and the face that was at the bottom of the cup is sanded and polished until it is very smooth and an excellent image can be seen in the microscope. Because the surface is smooth and everything is in the same plane, images are sharp even at 500× or higher powers. Embedding is necessary for the observation of metal microstructure and to obtain the best photographs, but is not needed for many, if not most, coating applications. It does take practice to prepare good specimens and to get good images with the cut/sand/polish method, but it can be fast and effective. None of the cross sections discussed in this article were embedded.

In addition to the analysis of defects, cross sections are useful in the identification of dirt. When viewed from the surface, most dirt in paint looks like a bump, nothing more. If the bump is cross-sectioned, then the dirt is uncovered, can be seen, and often can be identified. Figure 10 shows basecoat overspray droplets that had found their way into an automotive clearcoat.

Cross-sectioning, particularly of embedded specimens, also is a good, but tedious, technique for measuring film thickness. It has been used as a referee method for settling disputes between paint producers and users and in the evaluation and calibration of thickness

gauges. However, be aware that resins used in embedding compounds can swell paint films and change the thickness. Film thickness by cross-sectioning has been codified in at least one ASTM method. ASTM D 5235, "Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products." Figure 11 shows a cross section that was used to measure thicknesses of all four layers of an automotive coating. Although the top layer is purple in the picture, it actually is a clear coating. False colors are not unusual in microscope images.

Examination of Free Films

Occasionally, coatings films can be examined because they have delaminated or could be stripped from a substrate. In addition, free films can be made by drawing paint down or spraying onto a difficult to adhere to material such as Teflon®, polyethylene or propylene, or a steel panel wrapped with Tedlar® film. Free films often are brittle and difficult to handle, but they can be examined using transmitted light, which enables the viewer to evaluate pigment dispersion quality and detect the presence of crystalline components or contaminants (polarized light) in the final film. Paint chips and de-

laminated films can be cut with a craft knife or scissors and viewed in cross sections.

Microtoming

An increasingly important technique is the horizontal cutting of coatings into thin (5–20 μm) slices with a microtome. This may be done to allow the use of transmitted light for microscopic examination of a paint film, to separate a topcoat from a primer, or otherwise isolate one layer of the coating for examination or analysis. Microtoming is absolutely necessary in the investigation of component migration from one layer to another (as in the migration of a melanine from a primer to a basecoat or even a clearcoat).

Figure 14—Dark field image of blue automotive basecoat.

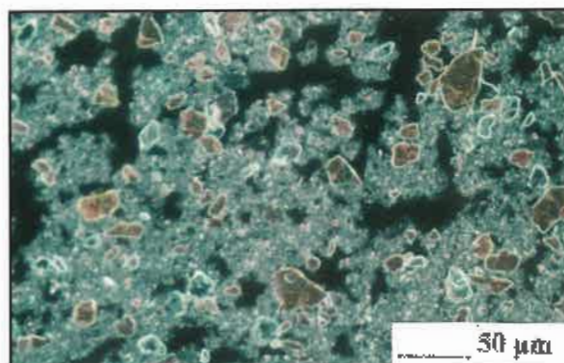
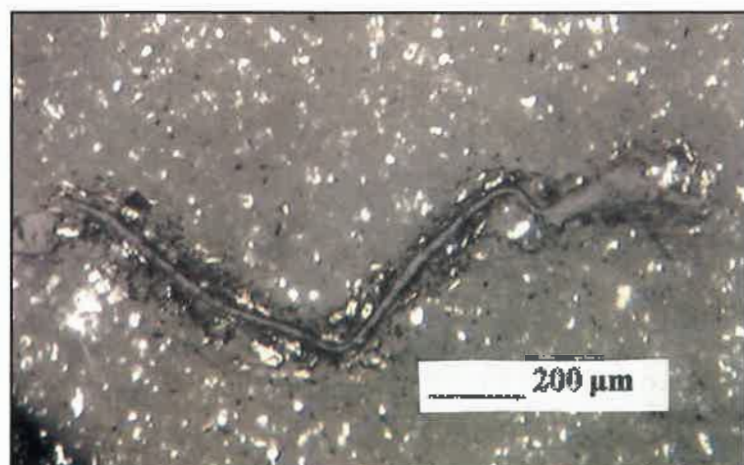


Figure 15—Automotive coating defect composed of a ribbon of cured paint on edge.



Examination of Liquids

Many paint problems lead to questions about the quality and stability of the wet paint, the pigment dispersion that went into it, or the resin or latex. Transmitted light microscopy at 200–500× is an excellent way to look at these liquids. A small drop of liquid is placed on a clean glass slide, and then gently covered with a glass cover slip. Dark field illumination usually is a good way for observing such specimens and is particularly good for monitoring the stability of paints, dispersions, and latexes with heat or other aging. For example, it is easy to see whether Brownian motion (a sign of a stable latex or dispersion) continues to exist or has stopped or whether phase separa-

tion is occurring. A combination of dark field imaging and photography or other image archiving can be used to follow changes in particle size and note increases in the degree of flocculation. Figure 12 shows a waterborne dispersion resin that has begun to coalesce into droplets. The micelles should be the size of the smallest particles in the picture.

Pigment dispersions often are better viewed with bright field illumination. Figure 13 shows the appearance with bright field illumination of three different time periods in the life of a troublesome electrodeposition bath: (a) initial fill, (b) flocculation after several weeks, and (c) severe flocculation after several more weeks. The resin dispersion and other components are essentially invisible, but the pigment and its flocculation are easily seen.

Flooding and floating, rub-up, or other color problems often lead to the examination of the pigments in a paint or paste. The investigator usually is looking for flocculation and/or determining whether the pigment was dispersed completely. Unfortunately, in some multi-pigment grinds, both problems co-exist. One pigment will be over-dispersed to the point of flocculation, whereas another pigment will be far from completely dispersed. This is not a good situation, but at least the viewer has found out what the problem is. Figure 14 shows flocculation in a blue automotive basecoat. The large plate-like particles are aluminum flakes. The flakes are not flocculated, but exhibit a considerable variation in

Figure 16—"Creature" that turned out to be plastic.



size, possibly due to degradation during circulation of the paint in the auto plant.

A difficulty in looking at the quality of a pigment dispersion either in a paint or paste is that any highly filled system (including most pastes) will look good because there is so much pigment jammed together that no flocculation can be seen. The obvious solution is to dilute the sample. However, if after dilution you see that the resultant dispersion is flocculated, you do not know whether it was flocculated in the first place or you just flocculated it when diluting it. Dilution must be done very carefully, ideally with the paste vehicle or a clear version of the paint.

FINAL COMMENTS

Microscopes are very useful tools for the identification and solving of problems, but the investigator must always be aware of the possibility of being fooled by what is seen or is thought to have been seen. The great rheologist and microscopist, Henry Green, once commented, "Some of the things that you see under the microscope are not artifacts." Today, we have the advantages of digital cameras and image analysis software, but mistakes still are possible. It is not wise to take a quick look and jump to a conclusion. The material in the defect in Figure 15 looks very much like a fiber and it would be easy to conclude that this was so. However, the "fiber" was teased out of the coating and it turned out to be a strip or ribbon of cured paint on edge. So, instead of searching for fiber sources, we looked for sources of paint chips, flakes, or ribbons (which often come from tools that hold parts or assemblies or, in an auto plant, hold the hoods and deck lids open).

I once looked at peculiar particles that were being trapped by a filter in a paint plant. What I saw is shown in Figure 16. I thought that they looked like little shrimp or other creatures, perhaps diatoms from diatomaceous earth. However, after several of the "creatures" were examined by Scanning Electron Microscopy (SEM) and IR microscopy, it was clear that they were composed of polyolefin plastic and appeared to be turnings or scrapings from a beading on a stirrer or disperser. So, be careful what you conclude from just your observations with a microscope. You can be fooled! □