Methods To Identify Contaminants On Photoresists

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Contaminants on photoresist can be difficult to identify, especially if they are very thin, *e.g.*, less than 0.5 µm. It is very doubtful at the 0.2 µm realm that histological or optical microscopy methods will work. There are several methods available to identify such contaminants, each giving different degrees or type of information about contaminants.

1) The "simplest" method is Fourier Transform Infrared Spectroscopy (FTIR). However, "simple" is perhaps not the best choice of words. Depending on contaminant film thickness, using FTIR with Attenuated Total Reflection (ATR) multi-reflection, it may be possible to "see" the film, but the photoresist background will need to be dealt with (a non-trivial matter). FTIR would require a substantial lateral size of the contaminant, say 100 µm or more for this technique to work. The FTIR would not identify a specific organic contaminant *per se*, and would not identify a biological one.

2) Electron Spectroscopy for Chemical Analysis (ESCA), also called X-ray Photoelectron Spectroscopy (XPS), is very good at analyzing very thin films. Depth of information for XPS is about 100 Angstroms (0.01 µm). The lateral information area is at best about 10 µm (50-75 µm is more typical). It can detect all elements greater than He at concentrations of about 0.1-1.0%. and give quantitative results (the accuracy depends on the standards used, etc.). ESCA/XPS can also give some chemical state information, *e.g.*, nitrogen as azide versus nitride, carbon as CF_x versus carbide, etc. This can be extremely useful, but chemical state information is not completely unambiguous. ESCA/XPS requires ultra-high vacuum ($<10^{-9}$ torr) and samples must be compatible. Photoresist should not be a problem, but if a contaminant has high volatility or is very hydrated (such as a biological), then this method may not work as is.

3) Time-Of-Flight Secondary Ion Mass Spectroscopy (TOF SIMS) is a mass spectrometry method with extreme surface sensitivity. TOF information comes from the top 2-3 monolayers of a sample and can easily see films of one monolayer or mono-atomic thickness. Mass resolution is good (typically $M/\triangle M$ = ~10,000) and spatial (lateral) resolution is reasonable (about 0.2 µm) but not both simultaneously.

TOF also requires ultra-high vacuum, but a cold stage (offered by one or two of the TOF manufacturers) can work with volatile and somewhat hydrated samples. Specific identification *may* be possible with TOF. Information from TOF represents molecular/elemental mass fragments from the surface. Complex organics can often be identified (with standard) and "reverse assembly" of molecular mass fragments can sometimes be done to yield the parent molecule. TOF has excellent elemental/molecular sensitivity, with some elements detected at the parts per billion range or lower.

Caveats: The FTIR method, although more commonly available, is the least likely to work, especially if the film is very thin and in small spots. FTIR for this application requires a very skilled analyst (A routine lab guy is not likely to be successful, even if there is enough material to do the job). You also need a high quality machine (a really good FTIR with an IR microscope, multi-pass ATR cell, etc.) to match your highly skilled analysis.

ESCA/XPS is an expensive technique (Instruments usually cost about $400,000) and requires an experienced operator. Commercial analytical laboratories are probably the best bet if this needs to be done. Cost for this analysis would probably run from $450-$1,500, depending on what is needed from the analysis. TOF instruments are even more expensive (typically $600,000) and require very skilled, experienced analysts. There are not very many of these machines around the country. Commercial analytical labs are your only real choice here. Analysis would probably run around $750-$1,000.

COMING EVENTS

Marine Biological Laboratory Courses: Woods Hole, MA
May 7/15 '98: **Analytical & Quantitative Light Microscopy**
May 19/26 '98: **Microinjection Techniques in Cell Biology**
Oct 7/15 '98: **Optical Microscopy & Imaging in the Biomed Sciences**
Carol Hamel: (508)289-7401, admissions@mbl.edu

✓ **Practical Aspects Series of Short Courses at Univ of Maryland**
May 18/22 '98 Scanning Electron Microscopy - Session I
May 19/22 '98 Image Analysis
May 25/29 '98 Scanning Electron Microscopy - Session II
May 26/29 '98 X-ray Microanalysis
Tim Maugel: (301)405-6898, eMail: maugel@zool.umd.edu

✓ May 19/21 '98: **Ultramicrotomy of Materials Workshop & Seminar** Leica, Diatome US & EM Sciences) University of Colorado, Voice mail: (800)248-0665 X5010, Diatome: (215)646-1478, www.leica.com eMail: Mike_Broykin@leicana.com

✓ May 21/23 & 25/27 '98: **Quantitative Image Analysis Workshops.** (North Carolina State University) Raleigh, NC. Alice Warren: (919)515-4195, Fax: (919)515-7614, email: alice_warren@ncsu.edu

✓ May 22 & 23 '98: **SPM Master Class (Workskhop).** (Univ. of Minnesota). Minneapolis, MN. Characterization Facility: (612)626-7594

✓ May 26/June 25 '98: **Biological Transmission Electron Microscopy** (Nassau Community College). Garden City, NY, Stephen Beck: (516)572-7829

✓ May 27/29 '98: **Microscopical Society of Canada Annual Meeting:** Montreal, Canada. www site: http://nucleus.rsvs.ulaval.ca/MSC

✓ June 7/10 '98: **SCANDEM '98** (Helsinki Univ. of Technology), Espoo, Finland. http://scandem.hut.fi, eMail: scandem-98@hut.fi

✓ **LEHIGH MICROSCOPY SHORT COURSES - 1998**
June 8/12 '98: SEM and X-ray Microanalysis
June 7 '98: Introduction to SEM and EDS for the new SEM Operator
June 15/19 '98: Advanced Scanning Electron Microscopy with Digital Image Processing.
June 15/19 '98: Quantitative X-ray Microanalysis of Bulk Specimens and Particles
June 15/18 '98: Analytical Electron Microscopy: Analysis of TEM Specimens
June 16/19 '98: Atomic Force Microscopy and Other Scanned Probe Microscopies
For information, contact Sharon Coe at phone: (610)758-5133, Fax: (610)758-4244, eMail: slc6@lehigh.edu

✓ June 17/28 '98: **3D Microscopy of Living Cells** (Univ. of British Columbia) Vancouver, BC, Canada. Prof. James Pawley: (608)265-5315, email: JBPAWLEY@FACSTAFF.WISC.EDU

✓ June 22/26 '98: **14th Annual Short Course on Molecular Microspectroscopy** (Miami Univ) Oxford, OH (513)529-2874, Fax: (513)529-7284

✓ June 22/28 '98: **OIM Academy - OIM Theory and Practice** (TexSEM Lab) Provo, UT (801)495-2758, Fax: (801)495-2758

✓ June 30 - July 2 '98: **3D Image Processing** (Univ. of British Columbia) Vancouver, BC, Canada. Prof. James Pawley: (608)265-5315, email: JBPAWLEY@FACSTAFF.WISC.EDU

✓ July 7/9 '98: **MICRO 98** (Royal Microscopical Society) London, UK, +44 (0) 1865 248768, Fax: +44 (0) 1865 791237, info@rms.org.uk

✓ July 11/12 '98: **New Developments in Multi-photon Excitation Microscopy** (prior to MSA/MAS '98) Atlanta, GA Annamarie Dowling: (708)361-6000, Fax: (708)361-6166

✓ July 12/16 '98: **Microscopy & Microanalysis '98.** (Microscopy Society of America) Atlanta, Ga. http://www.msa.microscopy.com

✓ July 24/26 '98: **5th Joint Meeting of The Histochemical Society and The Japan Society for Histochemistry and Cytochemistry,** San Diego, CA, http://www.hcs.microscopy.com

✓ July 26/29 '98: **31st Annual International Metallographic Society Convention** (ASM) Ottawa, Canada. http://www.asm-intl.org

✓ July 27/31 '98: **3rd Annual Fundamentals and Applications of Light Microscopy** Waltham, MA Mary McCann, (617)484-7865, Fax: 617)484-2490, mccanns@tiac.net

✓ August 2/7 '98: **UltraPath IX Meeting** (Society for Ultrastructural Pathology), Asheville, NC, Dr. J. Allan Tucker: (334)471-7473, Fax: (334)471-7884

✓ August 10/13 '98: **INTER/MICRO-98** (McCrone Research Institute) Chicago, Il. Nancy Daerr: (312)842-7100, Fax: (312)842-1078, email: ndaerr@mcri.org

✓ August 17/21 '98: **Summer School on Computing in Electron Microscopy** (Lawrence Berkeley National Lab) Berkeley, CA. (510)486-6036, Fax: (510)486-5888 eMail: JLCavlina@lbl.gov

✓ Aug 23/27 '98: **Microstructure and Microtribology of Polymers** (ACS) Boston, MA ACS: (202)872-4396, Fax: (202)872-6128

✓ Aug 31 - Sept 4 '98: **ICEM XIV/International Congress on Electron Microscopy.** Cancun, Mexico. (525)553-4507, Fax: (525)553-4500, email: icem@icem.inin.mx WWW: http://icem.inin.mx

✓ Sept 19/23 '98: **100th Anniversity Golgi Conference** Pravia, Italy, Dr. A. Mironov +39872 570 323, Fax: +39 578 240

✓ Sept 28 - Oct 2 '98 (Tentative): **5th Annual Materials Science Course** (RMC) Tucson, AZ Ann at (520)903-9366, Fax: (520)903-0132

✓ Sept 28/Oct 2 '98: **OIM Academy - Advanced OIM Theory and Application** (TEXSEM Labs) Provo, UT. (801)495-2758, Fax: (801)495-2758

✓ Sept 29/Oct 2 '98: **5th Annual Materials Sciences Course** (RMC) Tucson, AZ. Ann: (520)903-9366 X251, Fax: (520)903-0132, eMail: rmc@rmc-scientific.com

✓ Oct 5/9 '98: **6th International Conference and Workshop on Molecular Morphology.** Salzburg, Austria Prof. Gerhard W. Hacker: 43 662 4482 4730, Fax: 43 662 4482 882, eMail: g.hacker@lkasbg.gv.at www: http://kongress.at/IMMC/

HELP!!!

In our efforts to improve the quality of this publication, we continue to appreciate contributions of articles/materials as well as cover images.

The only criteria for articles/materials is that they be of interest to a reasonable number of working microscopists. They can be of any reasonable length and color images are usually acceptable.

Cover images should have "technical value" as well as being interesting to view. While color is preferred, black/white is acceptable.

Reflected/Transmitted Nomarsky DIC Lighting

Ely Silk, Cryolume Scientific Company

What could be better than reflected or transmitted Nomarski differential interference contrast? Why. combining the best features of both and for very little cost. My intended use of the technique was for Nomarski reflection DIC microscopy. It will work, of course, with other types of reflection microscopy.

What this embarrassingly simple artifice accomplishes is to *simultaneously* add transmission capabilities to reflection observations with the result being improved viewing of delicate details. And, yes, because of the reflective front surface layer, the observer can study details on the bottom side of the specimen which is usually hidden from view. This requires focusing *through* the specimen and below the point of normal focus. Usually we take pains to avoid this bonus!

The procedure is as follows:

1) Place one drop of immersion oil on the aluminized front surface of a small, front surface mirror (e.g., Edmund Scientific p/n 30286).
2) Carefully place a clean glass cover slip on the oil drop.
3) Place a drop of the liquid containing specimens on the cover slip from step 2.
4) Carefully place a clean glass cover slip on top of the drop with the specimens.
5) Arrange the *optical sandwich* on the microscope stage and adjust the reflection optics as usual.

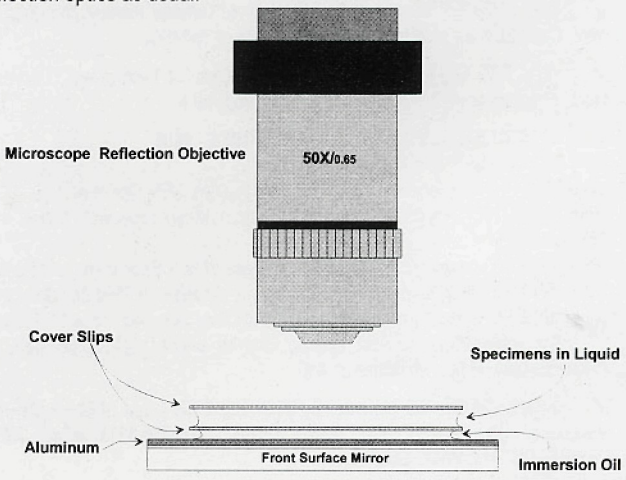

Figure 1: REFLECTION/TRANSMISSION TECHNIQUE

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The cover slip resting on the oil drop performs the following functions:

1) Protects the aluminum layer from liquids containing the specimens.
2) Serves as a shim to keep the specimens far enough from the aluminum layer to prevent mirror reflections from showing up in the field of view. This works because of the limited depth of field of typical microscope objectives.

The method will work with previously produced microscope slides which have specimens embedded in standard mounting media. The microscope slide is placed directly on the oil droplet resting on the front surface mirror, eliminating the need for the two cover slips and specimens. However, my experience indicates it is not quite as good as the cover slip technique. Also, reflection objectives are usually not corrected for cover slips. Use #0 thickness cover glasses and avoid very high power objectives.

Instead of using ready-made front surface mirrors, you can vacuum coat a reflective aluminum layer on microslides or cover slips. The combined lighting method is synergistic. I find that the results are superior to either reflected or transmitted DIC. Images are very bright and crisp. Try the technique while viewing various microorganisms. Witnessing ameboid movement across a slide with the special lighting is an experience. You will wonder how you ever got along without it! ■