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https://doi.org/10.1017/S1551929500066086 Published online by Cambridge University Press

Sample Preparation for X-Ray Microanalysis

Don Chernoff, Small World

Looking at samples in a scanning electron microscope often involves some type of sample preparation. The most common form of sample prep is coating the sample, usually with a heavy metal. The most common metal used for sample coating is gold. The need for coating samples is taken as a given by many people, but is it always necessary? To answer this question let us take a look at the reasons for coating a sample.

The most common reason for coating a sample is to eliminate the static charge which builds up on a non-conductive sample exposed to an electron beam. But it is fairly common to see people coat samples that are already conductive. The second and less obvious reason to coat a sample is to improve the secondary electron yield, which produces a better quality image with reduced noise. Remember that the image in a SEM is formed by secondary electrons and that these electrons come from around the top 100-200 Angstroms of a sample. Not coincidentally this is the typical coating thickness. Gold is the most common material for coating since it produces a good uniform coating and because it has a very high secondary electron yield. That is, it will produce many more secondary electrons under a given beam than carbon, for instance. The basic rule is that higher atomic number elements will have a higher secondary electron yield. Higher electron yield equals better signal to noise ratio equals prettier pictures.

The potential downside to coating is how it may affect x-ray microanalysis results. Coatings can interfere with x-ray analysis in three basic ways. First, if the coating is very thick, it can limit the beam penetration into the sample. This is not usually a problem. Second, peaks will appear in the spectrum corresponding to the film. Gold M lines can cause confusion and interfere with some low energy lines, namely the P and S K lines and the Mo L line. Third, and most significant, the gold coating can act to absorb x-rays as they leave the sample on their way to the x-ray detector. This tends to be a problem when looking for very low energy x-rays (Boron to Oxygen) and when looking for trace amounts of an element. This absorption can cause you to miss elements which are present in a sample or cause erroneous quantitative results.

The simplest way to avoid these problems is not to coat your sample. In many cases this is the preferred technique for x-ray analysis. One downside may be poor image resolution and/or charging. In modern SEM's image resolution is typically good enough on un-coated samples to allow you to find the area in need of x-ray analysis.

The bigger problem can be charging. If a sample charges severely it may be impossible to image it well enough to do analysis. It is also possible for a static charge on the sample to exert an opposing force to the beam resulting in a lower net accelerating voltage beam hitting the sample. For example, if a 5 kV worth of static charge builds up on a sample and you are using an accelerating voltage of 20 kV, the actual net accelerating voltage reaching the sample will be 15 kV. You can verify this by looking at where the background of your spectra reaches zero counts on the energy scale. In many cases the spectrum will go to zero at a lower kV than the accelerating voltage of the microscope. This phenomena is most important if you are trying to do quantitative analysis, and will give rise to ZAF correction errors. In these cases a coating with a light element such as carbon can provide a conductive surface with minimum interference with generated x-rays. A little bit of charging may have little affect on x-ray production for qualitative analysis. Charging can also give rise to beam drift. If you are analyzing a sample in spot mode you may find your beam far removed from the area where you placed it at the beginning of the analysis. In fact there is a simple test you can do to look for beam drift. If you can place the beam on a spot and it does not drift after about 30 seconds your sample is most likely conductive enough that it does not need to be coated to perform x-ray analysis. ■

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COMING EVENTS

✓ June 19/23 '95: **MICRO ONE - Intensive Course in Light Microscopy** (Gordon Grau Scientific). Kissimmee, FL. Barry Fookes: Tel./Fax: (407)931-1975

✓ June 26/30 '95: **Congres "Trinoculaire" de Microscopies Electroniques.** Joint Meeting SBM, SFME, SGOEM. Details: P.A. Buffat, EPFL-CIME. Fax: +41 21 693 44 01, eMail: philippe.buffat@cime.epfl.ch

✓ June 26/30 '95: **Computer Simulation and Processing of HRTEM Images.** NCEM, Lawrence Berkeley Lab., Berkeley, CA. Michael A. O'Keefe, eMail: MAOK@LBL.GOV.

✓ June 26/30 '95: **11th Annual Short Course on Molecular Microspectroscopy.** Oxford, OH. (513)529-2873

✓ July 3/6 '95: **CYTO 95 - The Application of the Microscope in Life Sciences** (RMS) Univ. of Southhampton 0865 248768, Fax: 0865 791237

✓ July 10/13 '95: **INTER/MICRO '95.** McCrone Research Institute. Chicago, IL. Nancy Daerr, Tel: (312)842-7100, Fax: (312)842-1078

✓ July 10/Aug 4 '95: **Estuarine Fish Ecology** (U. of S. Carolina short course). Georgetown, SC. Kitty Harper: (803)777-2692.

✓ August 6/11 '95: **Microbeam Analysis Society (MAS) National Meeting.** Breckenridge, CO. Gregory Meeker, Tel.: (303)236-1081, Fax: (303)236-1414.

✓ August 6/11 '95: **XIVth International Pfefferkorn Conference on the Science of Biological Specimen Preparation for Microscopy and Microanalysis.** Belleville, IL. Marek Malecki: (608)263-8481, Fax: (608)233-2400.

✓ August 13/17 '95: **Microscopy Society of America/Histochemical Society Annual Meeting.** Kansas City, MO. (800)538-3672 or (508)540-5594, Fax: (508)548-9053, mmaser@mbl.edu

✓ August 29/Sept 2 '95: **14th International Congress on X-ray Optics & Microanalysis.** GuangZhou, China. Tel.: 8620-777-5213, Fax: 8620-777-5791.

✓ Sept '95: **1st International Conference of Electron Microscopy and Advances in Research in Different Fields of Science.** Ismailia - Egypt. Dr. Khalifa Ibrahim Khalifa: Phone/fax: (20)64-329478.

✓ Sept 2/6 '95: **3rd Interamerican Congress of Electron Microscopy.** Caxambú MG, Brazil. Elliot Kitajima, Tel.: 55-61-348-2424, Fax: 55-61-349-9094, kitajima@guarany.cpd.unb.br

✓ Sept 4/8 '95: **New Zealand Microscopy Conference '95.** (NZSEM). Dunedin, New Zealand. Allan Mitchell, 64 3 479 7301, Fax: 64 3 479 7254, allan.mitchell@stonebow.otago.ac.nz

✓ Sept 12/15 '95: **EMAG 95.** Univ of Birmingham, UK. 44-171-235-6111, Fax: 44-171-823-1051, iopconf@ulcc.ac.uk

✓ Sept 26/Oct 2 '95: **14th International EM Congress.** Cancun, Mexico. Miguel Jose Yacaman: 525-570-85-03 Fax: 525-570-85-03

✓ Sept 26/30 '95: **OIM Academy - EBSP & Orientation Imaging Microscopy.** (TSL, Inc.). Provo, Utah. David Dingley: Tel: (801)344-8990, Fax: (801)344-8997.

✓ Sept 29/Oct 1 '95: **Symposium on Integrated Microscopy.** (Integrated Microscopy Resource, U of WI). Madison, WI. IMR, Univ. of WI, 1675 Observatory Drive, Madison, WI 53706. imradmin@calshp.cals.wisc.edu

✓ Sept 29/Oct 1 '95: **14th Annual Advances in Microscopy Symposium "Microscopy Outreach: Conveying its Science, Art & Technology"** (NCSMMA). Wrightsville, NC. Peter Ingram: (919)541-6598, Fax: (919)681-8419, ingram@rti.org

✓ Sept 29/Oct 1 '95: **First Annual Symposium on Integrated Microscopy.** Univ. of Wisconsin, Madison. imradmin@calshp.cals.wisc.edu

✓ Oct 4/5 '95: **International Seminar on Quantitative Microscopy.** Braunschweig, Germany. H. Geuther: Fax: 49 531 592 4015, heinrich.geuther @ptb.de

✓ Oct 9/13 '95: **Scanning Electron Microscopy and X-Ray Microanalysis for the Materials Scientist.** (SUNY - Inst. of Materials Science). New Paltz, NY. Dr. A.V. Patsis: Tel.: (914)257-3800, Fax: (914)255-0978.

✓ Oct 12/14 '95: **Great Lakes Microscopy Conference '95.** (GLeMA) Toledo, OH. Dr. Carol Heckman: (419)372-8218

✓ Oct 16/20 '95: **AVS Annual National Symposium.** Minneapolis, MN. Tel.: (212)248-0200, Fax: (212)248-0245.

✓ Oct 24/27 '95: **Ultramicrotomy in Materials Science** (RMC and Univ. of Arizona). Tucson, AZ. Bob Chiovetti: (520)889-7900, Fax: (520)741-2200.

✓ Nov 15 '95: **23rd Scottish Microscopy Symposium.** Edinburgh, Scotland. Stephan Helfer: 0131 552-7171, Fax: 0131 552-0382, Stephan@rbge.org.uk

✓ Feb 5/9 '96: **14th Australian Conference on Electron Microscopy (ACEM-14) & 1st Meeting of the International Union of Microbream Analysis Societies (IUMAS).** Sydney, Australia. Maret Vesk: 61-2-351-2351, Fax: 61-2-552-1967

✓ July 2/4 '95: **MICRO '96** (RMS), London, U.K. 44 1865 248768, Fax: 44 1865 791237

✓ Aug '96: **6th Asia-Pacific Conference on Electron Microscopy, APEM 6.** Hong Kong. Dr. E.C. Chew: 852 609 6845, Fax: 852 603 5031.

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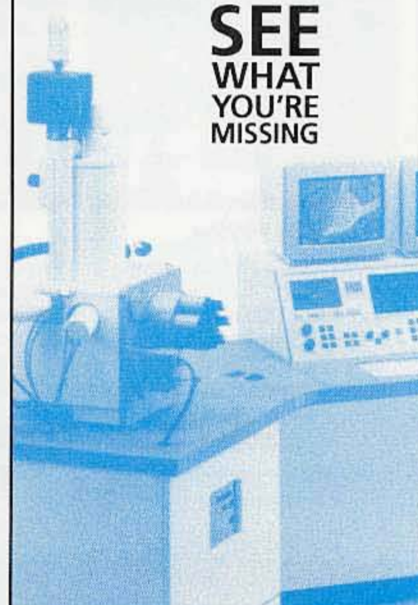


Circle Reader Inquiry #11

OOPS!

As pointed out by several of our readers, the cover micrograph on our last issue was from a TEM - not a SEM.

I wish to note that this was my misteak, not that of the authors of Cell and Tissue Ultrastructure.

--- Don Grimes, Editor

Biology For Nonbiologists And Support People

Sterling P. Newberry

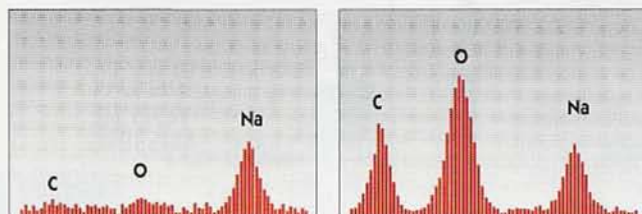
The fact that about half of our readers are not biologists does not mean that they are disinterested in the fascinating current developments in the field of biology any more than the biologist are disinterested in the equally fascinating progress of materials science or physics. Popular press and even trade journals often over simplify or at best give small pieces of information. It is nice when a refereed journal gives a state of the art review. Science Magazine recently gave such a review under the title "Development Frontiers in Biology"[1]. As is customary with Science, this issue also contains editorial summaries of many of the contributions in less technically obscure language.

I highly recommend this issue to all our readers. One should note that some of the new findings would have been very difficult or impossible without the use of microscopy and that the unsolved problems which are listed will need analytical microscopy to move hand in hand with the genetic testing methods which are becoming so powerful. ■

1. Science vol. 266, pps. 561 to 614, Oct.28 (1994)

Light Element Peaks Revealed!

Oil Film on EDX Windows Removed:



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The objective of this newsletter remains, perhaps unlike many others, to publish material and information of interest to the working microscopist.

And, as advertising thankfully increases, so does the challenge of obtaining an appropriate proportion of material and information. With this challenge in mind, I would like to sincerely thank the following individuals who have made contributions to the newsletter during the past year:

Gib Ahlstand, Univ. of Minnesota
Jessica Bailey, Handmade Software, Inc.
John Bozzola, Southern Illinois University
Charles E. Bryson III, Surface/Interface, Inc.
Anthony D. Buonaquisit, AMT
Stephen W. Carmichael, Mayo Clinic
Don Chernoff, Small World
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Donald P. Cox, Goldmark Biologicals
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Leonard Polizzotto, Polaroid Corporation
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Jean-Paul Revel, CALTECH
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Kenneth Smith, McCrone Associates
Robert Summers, SUNY Buffalo
A.J. Terry, ORTECH Corporation
Scott D. Walck, Wright-Patterson AFB
Daphna R. Yaniv, Arizona State Univ.

Speaking of "thanks", should you find the publication of some value, you might thank your sales representative for the advertising support of his/her company.

Circle Reader Inquiry #16

https://doi.org/10.1017/S1551929500066086 Published online by Cambridge University Press