

# Minnesota Microscopy Society

Local affiliate of the *Microscopy Society of America*  
and the *Microbeam Analysis Society*



Newsletter

September 2011

## “SDD-EDS Can Eat WDS’ Lunch!”

### Dale E. Newbury

NIST Fellow  
Surface and Microanalysis Science Division

### Date

Thursday, September 29, 2011

### Schedule

- 5:30 p.m. Registration
- 6:00 p.m. Dinner
- 7:00 p.m. Speaker

### Location

[Earle Brown Heritage Center](#), north of  
downtown Minneapolis in Brooklyn Center  
Google map: <http://bit.ly/q3Qa5r>



nist.gov



### Menu

- Minnesota Chicken Wild Rice Soup
- Salad, choice of dressing
- Local Wild Rice Pilaf
- Raised Beef Brisket with Guinness Brown Sauce or  
Turkey Tenderloins with Rosemary Jus Apple
- Cobbler or Tiramisu Trifle

### Reservations

Cost for the fall program is \$20 for members, \$10 for students. Reservations are being taken through **11:59 p.m. on Sunday, September 25**. Please make your reservation either by:

- email (preferred): [reservations@mnmicroscopy.org](mailto:reservations@mnmicroscopy.org) or
- phone: Bede Willenbring, H.B. Fuller Company; 651-236-5470 (9a – 5p)

## MMS Fall Kickoff Event – SDD-EDS as alternative for WDS

*continued*

### Speaker Bio

Dale E. Newbury obtained a B.S. in metallurgy and materials science from Lehigh University in 1969 and attended the University of Oxford as a Marshall Scholar, receiving a D. Phil. In Metallurgy and Materials Science in 1972. He has spent his career as a researcher at NBS/NIST, arriving as a National Research Council Postdoctoral Fellow in 1972, becoming a member of the research staff in 1973, serving as group leader for Microanalysis Research from 1979-1994, and being appointed as a NIST Fellow in 1994. His research interests include scanning electron microscopy, x-ray microanalysis, materials characterization, and materials science. He has taught for 38 years at the Lehigh University Summer Microscopy School.



### Abstract

**SDD-EDS Can Eat WDS' Lunch! (SDD-EDS Matches WDS for Quantitative Accuracy and Precision for Major, Minor, and Upper Trace ( $C > 1000$  ppm) Constituents, and at Lower Electron Dose; Elemental Mapping Too!)**

DALE E. NEWBURY AND NICHOLAS W. M. RITCHIE  
Surface and Microanalysis Science Division  
National Institute of Standards and Technology, Gaithersburg, MD 20899

Quantitative electron beam excited x-ray microanalysis was invented by Raimond Castaing in France 60 years ago with x-ray intensity measurements by wavelength dispersive spectrometry (WDS) and the “k-value” protocol, where the same peak is measured in the unknown and a standard for each constituent. The emergence of the energy dispersive spectrometer (EDS) based upon Si(Li) semiconductor detector technology in the late 1960s provided an important advance over WDS: the entire excited x-ray spectrum was recorded in every EDS measurement, enabling a full qualitative analysis at every analyzed location. Quantitative EDS analysis with the k-value protocol was quickly demonstrated through the efforts of Reed, Ware, Schamber, Statham, Fiori, Heinrich, Myklebust and others, but EDS has generally been considered a poor cousin to WDS when highly reliable concentration results are needed. While EDS may have been considered adequate for measuring major constituents (concentration  $C > 0.1$  mass fraction, 10 weight percent) in the absence of peak interferences, WDS has always been the choice when spectral interferences occur (e.g., S K, MoL and PbM) and for measurements of minor ( $0.01 < C < 0.1$ ) and especially trace ( $C < 0.01$ ) constituents. Confidence in EDS quantitative analysis performance has been further diminished with the widespread (dominant?) use of “standardless analysis,” for which the analytical errors are broad and poorly defined.

*continued ...*

## MMS Fall Kickoff Event – SDD-EDS as alternative for WDS *continued*

### **Abstract** *continued*

The rapid emergence of the high throughput silicon drift detector (SDD) EDS is causing a paradigm shift in x-ray measurement capabilities. By measuring extremely high count SDD-EDS spectra and deconvolving with spectral filtering and multiple linear least squares (MLLS) fitting, SDD can match WDS for the precision and accuracy of x-ray intensity measurements (k-values), with an electron dose lower by a factor of 10 or more. This performance is possible even when severe interferences occur and when there is a large dynamic concentration range for the mutually interfering elements, e.g., TiK $\alpha$  and BaL $\alpha$  with a separation of 41 eV and a Ba/Ti concentration ratio of 24:1. Because SDD-EDS can match WDS k-values, the matrix correction procedures [e.g., ZAF,  $\rho(\rho z)$ ] will yield similar concentration error distributions with SDD-EDS.

SDD-EDS can also attack elemental mapping problems that have traditionally been considered only amenable to WDS measurement: e.g., quantitatively mapping trace levels in the range 0.001 – 0.01 mass fraction and mapping when severe interferences occur, e.g., solders involving interfering elements Cd-In-Sn.



## 2011:

**October 25**      **What:** **Bethel Meeting | Community Life Center**

**Location:** Bethel University  
Arden Hills, MN

**November 5**      **What:** **Microscope Day**

**Time:** 1:00 – 4:00 pm

**Location:** Minnesota Science Museum  
St. Paul, MN



Anyone interested in volunteering or to suggest activity ideas, please contact Ann Palmer – [palme003@umn.edu](mailto:palme003@umn.edu)

## 2012:

**March 15**      **What:** **MinnTS**

**Location:** to be determined

**May 4**      **What:** **Spring Symposium**

**Location:** Minnesota Science Museum  
St. Paul, MN



### **Plans are being made for the Spring Symposium!**

If you have ideas for topics you would like to hear about or have heard an interesting speaker you think would make a good Spring Symposium speaker, send your suggestions to any of the MMS board members or to [treasurer@mnmicroscopy.org](mailto:treasurer@mnmicroscopy.org).

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Corporate Sponsors are the backbone of financial support for the Society. These members make it possible for the Society to support Project Micro and to cover many expenses of the regular meetings and the Spring Symposium. MMS gratefully acknowledges the Corporate sponsorships provided by the following companies in 2009-2010. To become a Corporate Sponsor, complete and return the MMS membership form at the end of the newsletter.

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**Reminder:** Membership in MMS runs from  
 January through December.  
*Have you paid your 2011 dues?*

Please visit the MMS website, <http://www.mnmicroscopy.org/MMSform.pdf>, for a fill-and-print pdf version of the membership form.

## Minnesota Microscopy Society – Membership Form

All microscopists are urged to support their Society at one of the membership levels offered below. Often, supervisors will support MMS memberships out of their project budget because they recognize that it is a very inexpensive way to maintain and increase the skills of their microscopists. If you have been a member over the years and recognize the value of MMS to the community of microscopists it serves, consider upgrading your membership this year to the patron or sustaining level. Thank you.

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