

Secondary fluorescent excitation in the scanning electron microscope: Improved sensitivity of energy dispersive analysis

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The use of secondary fluorescence for x-ray energy spectroscopy in the scanning electron microscope has greatly enhanced both resolution and the lower limit of detection. This note describes a simple secondary fluorescence system. The x-ray energy spectra taken from a stainless steel sample illustrate the advantages of this method.

The use of secondary excitation sources in x-ray spectroscopy is not uncommon. The advantages of this technique can also be utilized with a scanning electron microscope (SEM) equipped with an x-ray energy spectrometer (XES) system to improve resolution and detection sensitivity (LLD). A sample holder and secondary target has been constructed as shown schematically in Fig. 1. The holder consists of SEM "toad stool" with a channel cut as shown in the figure. Samples are mounted by insertion in this channel. The secondary x-ray target shown in Fig. 1 is machined in the form of a truncated cylinder from a 6.35 mm diam 99.999% copper rod vapor coated with 99.999% gold. The electron beam is focused near the base of the target into a shallow depression approximately 1 mm deep. This configuration effectively shields the detector from bremsstrahlung and characteristic radiation produced by interaction of the electron beam on the secondary target surface. Note that the sample need not be conductive since the electron beam never strikes the sample surface. Multiple pure element targets can be vapor deposited on the secondary fluorescer surface in order to selectively excite a wide range of specific elements.

Figure 2 shows a comparison of x-ray energy spectra (obtained from a sample of stainless steel) made with

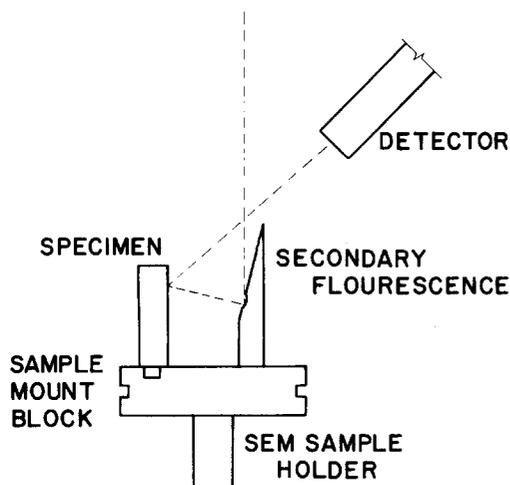


FIG. 1. Secondary fluorescence sample holder.

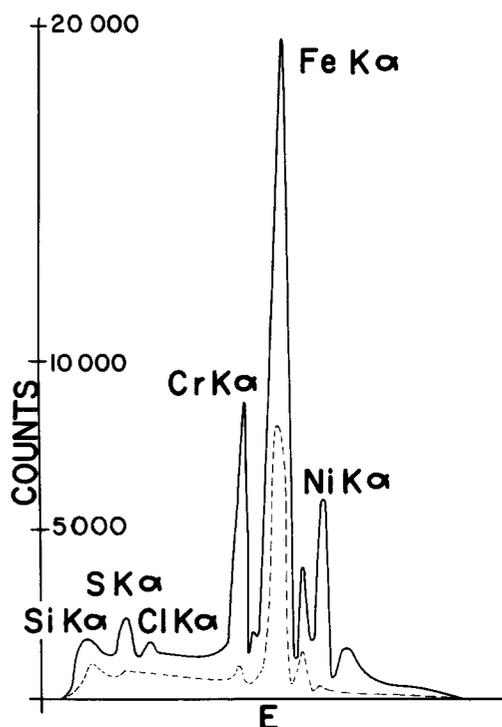


FIG. 2. Energy dispersive spectrogram from a stainless steel sample. Dotted line is direct excitation (20 kV-100 μ A). Solid line is excitation with secondary fluorescer (20 kV-180 μ A).

direct electron excitation and with the gold secondary target excitation both at 20 keV. Note that nickel, which is not visually evident in the direct excitation spectrum, is easily seen using the secondary fluorescence method. The detectability of all other elements has also obviously been enhanced. Similar analyses have been performed utilizing this technique on nonconductive glass samples, and impurities in the 50-100 ppm range have been detected.

Gold lines are absent from the spectral output due to the shielding effect provided by the geometry of the secondary target. The increased sensitivity of this system is due to (1) the high efficiency of selective excitation of Cr, Fe, and Ni K x-rays by gold $L\alpha$ ($E=9172$ eV); (2) the large reduction in bremsstrahlung background; and (3) the absence of intense scattered target radiation. Note that

scattered target radiation will be present when the sample is composed of low atomic number elements.

This present system is applicable only to bulk analysis.

The authors are presently working on a secondary x-ray fluorescence system having a spacial resolution of the order of 20–40 μ .

PUBLICATIONS

All books received in the office of the editor will be acknowledged by listing in the Books Received column of this section. Books pertaining directly to instrumentation or closely allied subjects will be selected for review as space permits. Reviews of books on physics or engineering, other than instrumentation, will be carried in Physics Today or other journals published by the American Institute of Physics.

Books Received

Practical Scanning Electron Microscopy. Edited by Joseph I. Goldstein and Harvey Yakowitz. Pp. 582+xvii. Illus. Plenum, 227 West 17th Street, New York, NY 10011, 1975. Price: \$35.00.

Elements of Electrical and Electronic Instrumentation. Kurt S. Lion, Pp. 400+xi. Illus. McGraw-Hill, New York, NY 10020, 1975. Price: \$22.50.

Electron Microprobe Analysis. S. J. B. Reed. Pp. 400+xiii. Cambridge University, 32 East 57th Street, New York, NY 10022, 1975. Price: \$34.50.

Mammalian Cells: Probes and Problems. Proceedings of the First Los Alamos Life Sciences Symposium held at Los Alamos, NM, 17–19 October 1973. edited by C. R. Richmond, D. F. Petersen,

P. F. Mullaney, and E. C. Anderson. Pp. 309+vii. Illus. National Technical Information Service, U. S. Department of Commerce, Springfield, VA 22161, 1975. Price: \$7.60 (foreign \$10.10) (paperbound).

Semiconducting Temperature Sensors and Their Applications. Herbert B. Sachse. Pp. 380+x. Illus. Wiley, 605 Third Avenue, New York, NY 10016, 1975. Price: \$22.95.

Proceedings of the ICO Conference on Optical Methods in Scientific and Industrial Measurements. Edited by Shun-ichi Tanaka. Pp. 544+xii. Illus. Publication Office, Japanese Journal of Applied Physics, 2nd Toyo Kaiji Building, 4024-8 Shibashi, Minato-ku, Tokyo 105, Japan. No price listed.