

## Strategy and Tactics of Microprobe Analysis

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### Abstract

This talk is designed as a tutorial that I might give to a new technician working in my electron microprobe laboratory, part of a centralized analytical facility in a large research company. Most of what I would teach this new person is applicable to every microprobe lab. It is a compilation of things to think about before accepting the analytical job, while planning the work, while doing the work, and while summarizing the results.

Scientists and technicians arrive in my lab with samples to be analyzed. These people are intelligent and highly trained but have widely variable knowledge about available analytical techniques. We start by interrogating the customer and jointly examining the samples with light microscopy to determine the customer's needs and the best ways to get the right answers. This always includes the consideration of other types of analyses. Even if it is a probe job, there are many types of techniques to choose from - quals/EDS, photos/maps, semiquants, spot quant, analytical traces, et hoc genus omne. Choosing the right one(s) is an important part of our job.

Survey the sample in the probe to determine if there are problems to overcome. The first part of the survey is to check (EDS) that the sample you've been given matches the description. Be sure that approximate composition/geometry/plating sequence/configuration/and whatever make sense. Know what every part of the sample is composed of. When working with a new, to you, combination of elements, carefully check for interferences and overlaps of all kinds. Use good standards such as certified pure elements and simple compounds whenever possible to check these problems. If you lack a standard, there are ways to buy, beg, borrow, steal, or cheat.

In order to perform good analyses of any kind, you need to know enough to choose the right standards from your supply and probe techniques available on your instrument. You might check interferences on one set of standards but use others for the actual analyses. If you have 2 or 3 correction procedures available, you need to

know which works best for this application. Photomicrographs may be the major data form needed or may just be taken to help guide other analyses. Don't do a casual job on these; they may be the item that is most important and/or most publicized. Avail yourself of every opportunity to learn more about probe techniques through lectures such as this, local and national meetings, short courses, discussions with knowledgeable colleagues as well as books and the web.

When the analyses are done, it is time to transfer the information into the requester's hands. Make sure that you also transfer the information into his mind; he has to understand what the quant summaries, photos, plots, line traces, etc. mean and what they don't mean. Again, the degree of detail needed depends on the expertise of the customer. A hint – If at all possible, put the data in graphical forms because these are so easy to interpret. After the customer leaves, critique yourself. What mistakes/missteps did you make? Could you have done better under the time/money/quality constraints put on you? How about with no constraints? When you start saying no to most of these questions, you have become an accomplished analyst.

### Biography

Since 1974, Paul Hlava has been in the electron microprobe laboratory (as staff member in charge of the lab since 1980) at Sandia National Laboratories in Albuquerque, New Mexico. Because the EMP lab is part of the Materials Characterization Department, a centralized analytical facility for Sandia, Paul gets to work on a wide variety of (prosaic to exotic) materials and projects. He normally analyzes many alloys and joins (welds, brazes, solders, metal to ceramic, glass/metal seals, etc.) but also does work on high tech ceramics, low-temperature superconductors, electronic materials, phosphors, contamination, corrosion, and failure analyses. Paul graduated from the University of New Mexico with a geology MS in 1974. At UNM he worked as a research graduate doing probe research under Klaus Keil. Paul occasionally uses his geological and mineralogical expertise on Sandia projects but also does some personal research on minerals. He has been co-discoverer and co-author on the descriptions of several new mineral species.

Paul has been a member of the Microbeam Analysis Society and the local affiliate, the New Mexico Microbeam Users Group, since 1975. He has been a director of MAS and president of NMMBUG. He has occupied the positions of MAS Director of the Affiliated Regional Societies and Tour Speaker coordinator for the past 8 years or so and he continues in those positions. He received the MAS Distinguished Service Award in 1996.