Upgrades Create State-Of-The-Art EPMA Facility

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A series of upgrades of an Electron Microprobe X-ray Analyzer (EPMA) were made to expand capabilities, increase efficiency, and cut costs. High technology EPMA features were acquired with cost effective expenditures. The driving force for our upgrade was to meet the continuing needs of corporate customers as well as many small companies which come to us for low-cost/high quality EPMA services.

Background

Electron Microprobe X-ray Analyzers (EPMA) have been utilized commercially in metallurgical analysis applications since the late 1950's. Our company bought an ARL probe (EMX) in 1962, which was heavily used until its retirement in 1975. At that time, we purchased an ARL SEM-Q type EPMA. In the late 1980's, we again experienced business pressures to acquire faster, more efficient analytical instruments, but quickly realized that outright replacement of large capital equipment items such as EPMA were no longer possible. We found spiraling costs of new instruments juxtaposed with reduced availability of capital. Also, because we are a diversified metallurgical services group, we had similar needs to upgrade our other instruments, i.e., scanning electron microscope, energy dispersive spectrometer and Auger electron spectrometer.

Fortunately, other options had become available at our time of need. Small companies formed by individuals with considerable EPMA development and service experience began offering services and various instrument upgrades, including new software.

We implemented a continuing program of capital acquisitions to upgrade our ARL SEM-Q electron microprobe, with contract services from Advanced Microbeam, Inc. and their software development vendor. Each step in the upgrade process added capability, analytical quality, and cut costs. The upgraded EPMA facility is shown in Figure 1.

Our experience may be relevant to many laboratories feeling the pinch of spiraling prices, corporate downsizing, competition for service business. It should be noted that what we have done can be adapted to other older laboratory instruments as well. We have also made some highly effective upgrades to a PHI 545 Scanning Auger Spectrometer.

Hardware

Three additional spectrometers were added to the EPMA bringing the total to six, with two crystals per spectrometer. Since spectrometers operate simultaneously, more elements can be run per unit time with added spectrometers, resulting in faster overall runs. The various distributions of crystal types installed in our spectrometers were determined by customer needs for analyses of samples containing particular mixtures involving large numbers of elements. One spectrometer contains a synthetic crystal which provides high sensitivity concurrent carbon and oxygen analyses.

High speed optical-encoded drive motors were installed in all six spectrometers to replace original equipment. These are key elements in upgrading the speed of EPMA data gathering; this is because routines involving analysis of large numbers of elements require time be expended in changing crystal settings back and forth on those spectrometers used for more than one element during a run. High speed drive motors expedite this process.

Contemporary electronics were installed in the EPMA to replace original equipment in power supplies and control circuitry. Some of the original electronics of the EPMA are now actually internal to the computer (e.g. scalers/timers). These changes are important because they contribute to dependable performance of the instrument and reliability of results.

Computer

A new IBM-compatible Pentium processor (100 MHZ, 1.2 MByte hard drive, 32 Meg RAM, CD Drive) replaced the existing PDP-11 system computer. This change accomplished increased speed in EPMA data gathering functions, including beam and stage control and greater reliability in stage automation operations. Data writing capability to Compact Discs (CD) was incorporated to afford greater data storage capacity and enormous condensation of record storage. The computer has communications software and a modem for remote access and control.

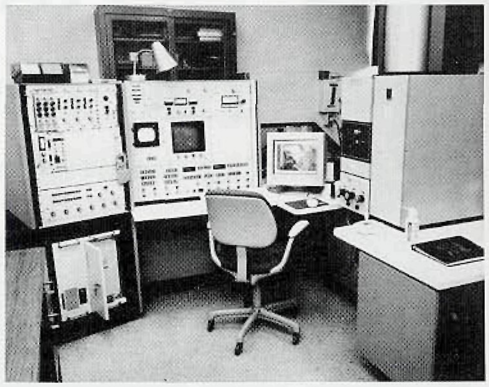

Figure 1: View of Upgraded EPMA Facility

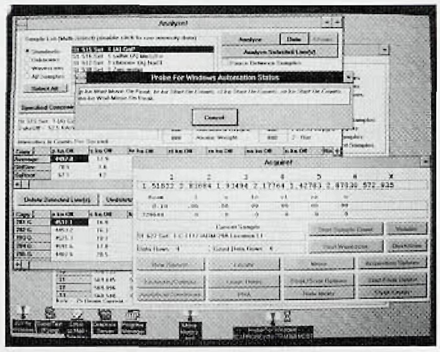

Figure 2: Windows Multitasking Environment

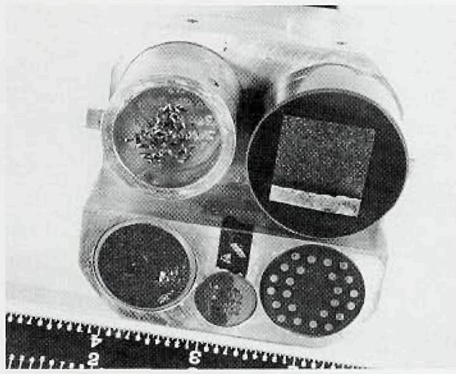

Figure 3: Special Specimen/Microanalysis Standard Mount for EPMA

Compact Disc (CD)

All EPMA archiving for long-term storage is done on CD. Approximately two years worth of work can be stored on one CD. CD stored data, and photographic images are likely to be involved in technical reports of the future. This type of information can be transmitted via local area networks and world-wide web, with excellent photo resolution and in color if required.

Software

Sophisticated windows based software is a key element of the EPMA upgrade. A screen showing multiple Windows functions can be seen in Figure 2. The software provides operational features for optimization of data gathering and highly accurate quantification of those data. It also provides enhanced data output in graphical displays and distribution maps. This software capitalizes on the computers high speed capabilities for frequent updates of system parameter values, such as beam current (which may drift with time). Beam current is measured for each line of data in each analysis. Pre-standardization and post-standardization data are examined to determine any drift or electronic errors; results of analysis of the unknown are then normalized accordingly. Detectability limits are calculated for each element in each line of data, based on measured peak to background ratios. The software permits precise definition of x-ray peak centers for optimized EPMA detectability of each of the chemical elements of interest. Peak overlap problems can be identified readily using other features of the software; a menu routine is used to test for any interferences in a particular run then calculations are made for compensating corrections. EPMA software works in conjunction with "surfer program" for routine plotting of concentration data versus X,Y coordinates.

Computer Beam Control Imaging

Digital beam control software works in conjunction with the existing CRT analog imaging of secondary electron and back scatter electron displays and x-ray mappings. The digital system can provide up to six-at-a-time x-ray maps with simultaneous output from the six spectrometers. Digitally-enchanted image quality greatly exceeds the analog image quality. Enhancement of the digital images is possible to optimize display of low intensity signals.

Reference Files

All set-up parameters and standardization data are retained on CD for later reference. This record keeping complies with quality assurance requirements and provides a convenient reference for quick set-up of subsequent analyses of similar materials.

EPMA Methodology and Standards

Unknowns and standards are loaded into the EPMA together in a special specimen holder. Therefore, pre-standardization, analysis, and post-standardization can be done at one time without opening the system. Figure 3 shows a photograph of the special specimen holder being installed in the EPMA. EPMA analysis routines can be fully pre-programmed for virtually unattended running. This frees the operator to perform other tasks.

Considerable strategic planning is needed to extract information from samples to be analyzed using EPMA; however, extremely detailed analysis routines can be pre-programmed for automated EPMA runs, with complete standardizations for quantitative analyses. A set-up begins examination of an image of the unknown, viewed in the EPMA; coordinates or locations of points to be chemically analyzed are entered into the computer (start and stop points of linear or area surveys could be entered). Set-up of the standard requires only specification of the coordinates of three fiducial orientation marks. No recalibration of standard locations is necessary, unless the standard mounts are unclamped from their fixed positions in the specimen holder. Standards consist of end mounted wires (24) in a metallographic mount, and each wire contains 20 standardization points (physical locations). The EPMA optical microscope cross hair is set on fiducial marks 1, 2, 3 in sequence. The computer then determines the exact position of all 24 standards and the associated standardization points.

Remote Control of EPMA

Remote access and control of the EPMA help us to realize more productive hours from the instrument each day. Early morning warm-up of the instrument and late evening shutdown after completion of unattended analytical runs and post-standardization routines are readily achieved by remote control.

Service

Service diagnosis of the EPMA computer and interfacing hardware is now possible remotely. Computer linkages are simply established with our service vendor via telephone lines through modems at our respective sites. Same day problem identification is usually possible.

Conclusion

Upgrades for EPMA have succeeded in enhancing our analytical capabilities while cutting the unit cost of services. We routinely provide EPMA services to outside firms via technical service agreements. More information including current rate can be obtained by contacting the authors at Babcock & Wilcox, R&D Division, 1562 Beeson Street, Alliance, OH 44601.