The History of the Electron Microbeam Analysis Laboratory at The University of Michigan

I. The First Twenty Years

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Founder of the Electron Microbeam Analysis Laboratory
And Director Until 1987

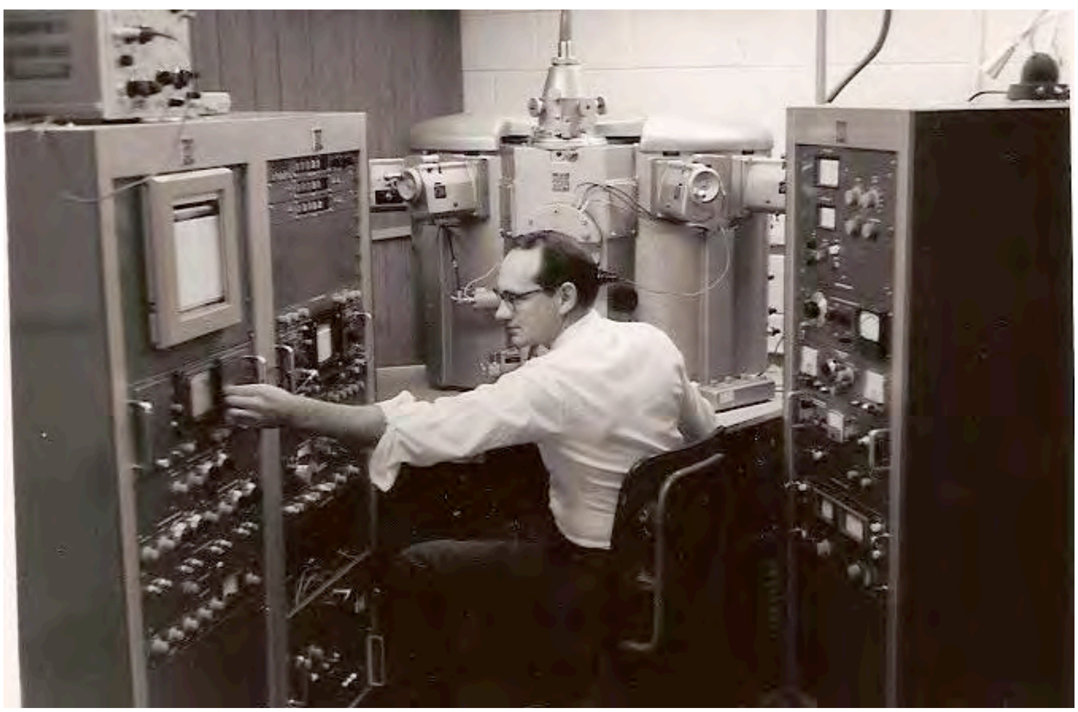
In 1951 Raimond Castaing presented a Doctoral Thesis at the University of Paris in which he described the development of an instrument in which an electron beam was focused to a fine spot on the surface of a solid sample, and the x-rays generated by the interaction of the electrons with the atoms in the sample were analyzed by a crystal spectrometer to identify the elements present and to determine their concentrations. Metallurgists and geologists around the world showed an immediate interest in this instrument, which was capable of providing qualitative and quantitative elemental analyses of regions of solid materials only a few micrometers in diameter. By the middle of the 1960s several companies had developed refined commercial versions of this instrument, and I became convinced that the University of Michigan, one of the world's leading research universities, should have one of these instruments. In collaboration with Professors Reynolds Denning of the Geology Department and Kamal Asgar of the Department of Dental Materials, proposals were submitted, unsuccessfully, to the National Science Foundation and other granting agencies requesting funds to purchase one. When Gordon Van Wylen became Dean of the College of Engineering in 1965 he received a sizeable discretionary fund, and Associate Dean M. J. Sinnott persuaded him to use part of it to assist in the purchase of one of these instruments.

Dean G. J. Van Wylen

Assoc. Dean M. J. Sinnott

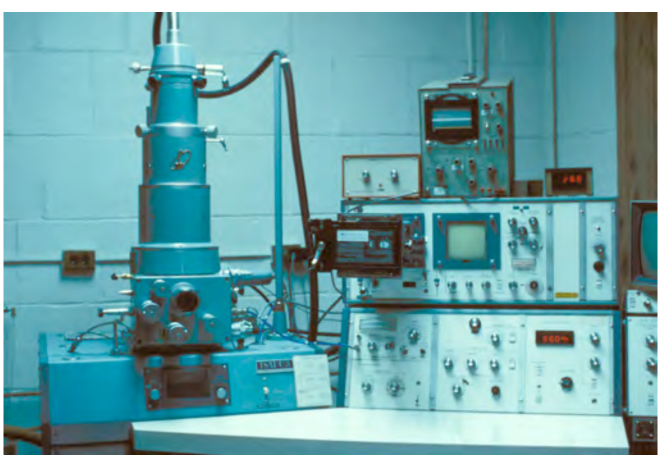
. With additional funding provided by A. G. Norman, Vice President for Research, a Model EMX-SM Electron Microbeam X-ray Spectrochemical Analyzer was purchased from The Applied Research Laboratories in November of 1967 for $95K. This instrument was equipped with three crystal spectrometers. Since each spectrometer could be set to analyze x-rays emitted by a different element, it was possible to perform micro-analyses of mineral and alloy specimens containing as many as ten elements in a matter of one or two hours. This capability led to the discovery of several new minerals and contributed important data for a large number of Geology students' theses.

A. G. Norman, V.P. for Research



Frank Drogosz and The ARL EMX-SM Electron Microprobe

One advanced feature of this instrument was its capability to produce scanning electron micrograph images. These provided resolution as much as 100 times better, and a depth of field more than ten times greater, than ordinary light microscopes. This capability made it possible to study ‘rough’ objects such as fracture surfaces of metals and ceramics, pollen grains, insects, bacteria, fibers, etc. in much greater detail than had been previously possible. Ultimately, the demand to use the instrument for scanning microscopy became so great that it interfered with Its use for analytical purposes. In response to this demand, Vice President Norman purchased a JEOL Model JSM-U3 scanning electron microscope ($76K) in 1969, This instrument soon also became overloaded, and so in 1975 Vice President Norman generously purchased a second JSM-U3 (used, $35K). It was particularly advantageous that both microscopes were the same, because then users trained on one instrument could work on the other with equal facility,

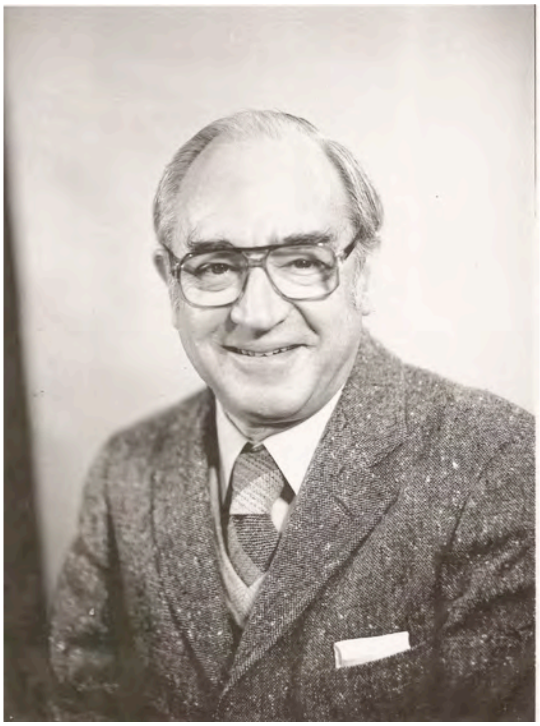


A JEOL JSM-U3 Scanning EM

By 1976 both of the SEMs, and the microprobe, were equipped with energy dispersive x-ray spectrometers (EDXS). These devices use a specially processed silicon diode to collect the x-ray photons emitted from the specimen. The photons interacted with the diode producing pulses of free electrons which are proportional in magnitude to the energies of the x-ray photons. These pulses are then processed by a dedicated computer to identify the elements that produced them. Whereas crystal spectrometers, such as those on the microprobe, can process photons from a single element at a time, these devices can collect and analyze photons from all elements in the specimen simultaneously. This makes it possible to identify the major elements present in the sample in a few minutes, and to obtain a rough quantitative analysis in about fifteen minutes. This is much faster than analyses can be performed by the crystal spectrometers, although the quantitative results are not as accurate. Since all three instruments in the laboratory now had the capability for performing microanalyses, the laboratory was named “The Electron Microbeam Analysis Laboratory” (EMAL), distinguishing it from other laboratories on campus that had ordinary electron microscopes.

In the mid-1970s the Philips Company introduced a transmission electron microscope (TEM) in which the electron beam could be focused to a fine point and scanned over the specimen to produce scanning transmission electron micrographs. This immediately provided the possibility of stopping the focused beam on a spot in the specimen and doing an analysis with an energy dispersive spectrometer. Soon other manufacturers produced similar instruments, and STEMs became very popular because of their great versatility. In the standard transmission mode they could produce conventional images from thin specimens with a resolution of less than 5 Å. In the scanning transmission mode they provided images with enhanced contrast and comparable resolution, plus the capability for performing microanalyses. It immediately became apparent that such an instrument would be a valuable addition to our laboratory, and so I prepared a proposal to the National Science Foundation requesting funding for the purchase of one. Professor Charles Overberger, who had become Vice President for Research by that time, accompanied me to present this proposal to NSF, telling the contract administrator there that "This is a very good proposal. You ought to fund it!" The proposal was indeed funded, and in 1978 we purchased a JEOL Model JEM-100CX STEM ($300K). Together with the microscope we were able to purchase an EDXS system from the Nuclear Data Co. that included a computer with 80 kilobytes of internal memory and a hard disk drive that provided 2 megabytes of external memory. Most EDXS systems at that time ran on PDP-11 computers with 28 KB of memory, so we had an EDXS system with the largest computer system in the country. Hal Estry, our electronics engineer, wired it up so that it served not only the STEM but also the EMPA and one of the SEMs.

By 1980 it became clear that the microprobe and scanning electron microscopes were outdated and needed to be replaced. In 1984, through an effort spearheaded by Professor Eric Essene of the Geology Department, funds were obtained from the Earth Sciences Division of NSF that enabled us to purchase an up-to-date microprobe from the Cameca Company. On this instrument the spectrometers were computer operated, as were the stage drive and the collection and processing of the spectral data. This made it possible program the instrument to perform



C.G. Overberger, V. P. for Research

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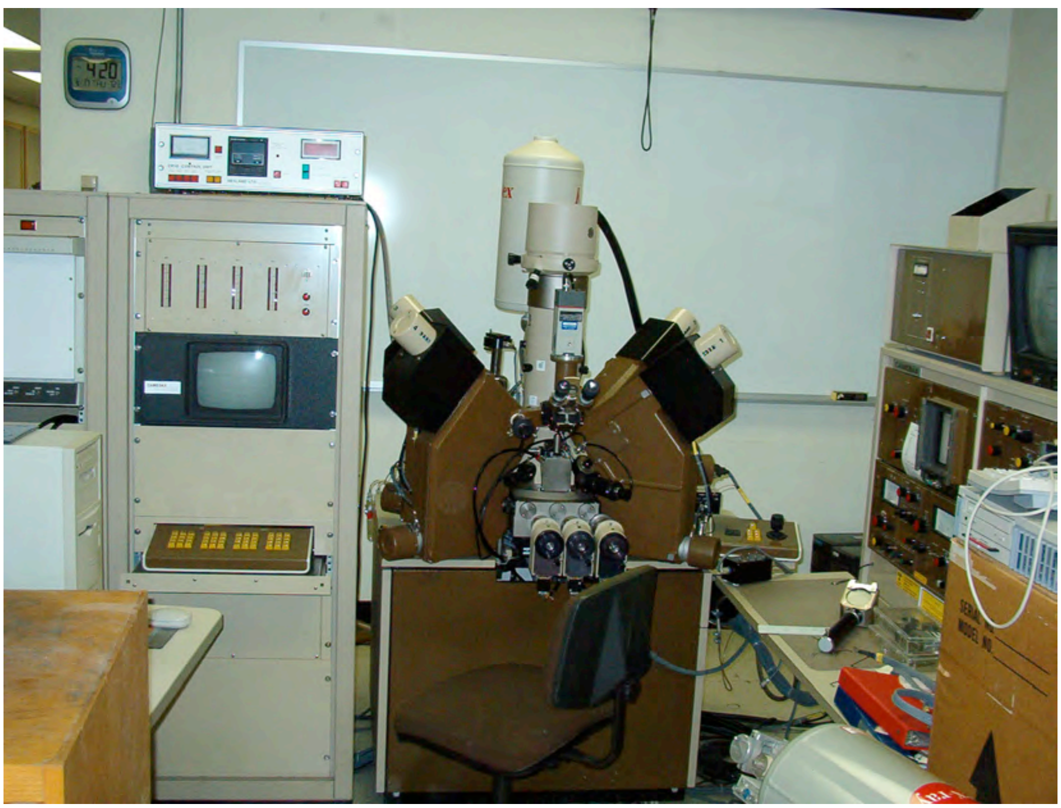
L. F. Allard Operating the JEOL JEM-100CX STEM multi-element analyses on a large number of pre-selected spots in a very short time. Furthermore, the programs used in processing the data were highly refined, and produced results that were accurate and reliable. It was also equipped with an EDXS.

At the same time Professor Kaufman of the Botany Department and Prof. Wilkinson of the Geology Department, obtained funds from NSF that made it possible to purchase an Hitachi Model S-570 scanning electron microscope. This instrument produced images of much better quality than the older SEMs. In addition, it contained software that would automatically adjust the focus, contrast, and brightness of the image.

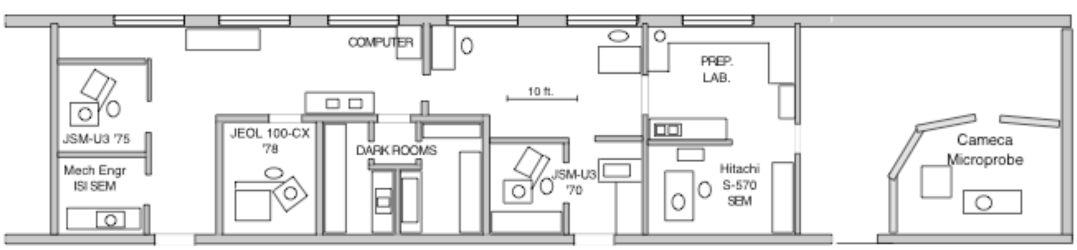


The Cameca Camebax Electron Microprobe

The Hitachi S-570 Scanning Electron Microscope

This made it easy for even inexperienced users to obtain good quality micrographs, increased operating efficiency, and reduced film consumption markedly. It was also equipped with an EDXS system.

Finding space to house such a large number of electron optical instruments is normally a big problem; however, I was exceptionally fortunate in this respect. Because research in the Department of Chemical and Metallurgical Engineering had shifted emphasis, a group of laboratory rooms on the northwest corner of the fourth floor of the East Engineering Building were virtually no longer being used, and I was able to take over these rooms for the EMAL instruments. This process centered around the Department's mass spectrometry laboratory. To accommodate the ARL EMPA we walled off a portion of a large unused room on the north side of this laboratory, producing a room for the ARL microprobe, and a nice preparation and service laboratory. By the time the first SEM was ordered the mass spectrometer had fallen into disuse, and so we got rid of it, walled off a part of that room to hold the SEM, and used the rest of it as office space for laboratory personnel. On the south side of the original mass spectrometer laboratory was a very large room that had once been heavily used as a metallography laboratory for sponsored research projects. When the second SEM was ordered I was able to move into this room, build two small enclosed rooms at the south end of it, and use one to house the second JEOL SEM and the other to hold an SEM owned by the Department of Mechanical Engineering. Later on the STEM was installed in a sub-room that once housed the research metallographic microscope. The suite also included two darkrooms that we took over and used for processing electron micrographs. When the Cameca microprobe arrived we expanded northward into an unused room there. Then we disposed of the ARL microprobe and used that room for the Hitachi S570 SEM. In the end EMAL occupied a substantial part of the northwest wing of the fourth floor of the East Engineering Building.

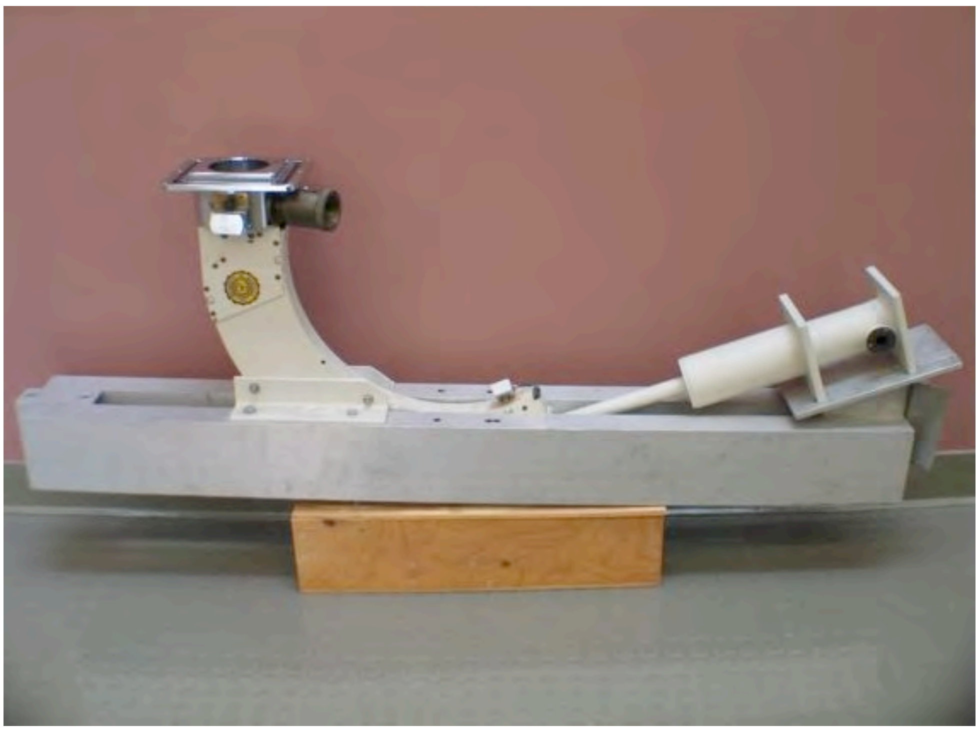


EMAL LABORATORY IN THE EAST ENGINEERING BLDG.
Final Configuration

Over the years the laboratory was run by a group of very talented and highly dedicates people, including: Larry Allard, John Mardinly, and Steve

Krause, who were graduate students in Materials Engineering, Peggie Hollingsworth, a graduate student in the Medical School, Hal Estry, a professional electronics engineer, and a number of part-time student assistants. Larry Allard, John Mardinly and Steve Krause became highly proficient experts in the operation of all instruments in the laboratory, and thus were able to provide assistance to users with all kinds of specimens. Peggie Hollingsworth assisted with the preparation and observation of biological specimens. Hal Estry provided the expertise in electronics that is so essential in keeping a group of instruments such as ours in good operating condition. Collectively we were able to provide the routine maintenance and special service necessary to keep all the instruments in the laboratory functioning at full capability. This made it unnecessary to have service contracts with the instruments' manufacturers, saving many thousands of dollars in operating expenses.

In addition to providing routine service and maintenance we made a number of modifications to the instruments to improve their performance and upgrade their capabilities. For example, Hal Estry modified the scanning circuits of the SEMs to incorporate a "slow scan" mode of operation that was featured on newer instruments, he built a filament heating current controller that allowed us to use high intensity lanthanum hexaboride filaments in the STEM, and he built a system that allowed us to manually control the vacuum valves, bypassing the built-in automatic system, on the STEM to facilitate certain special maintenance operations. We modified a standard micromanipulator and installed it on one of the SEMs where it was used for such delicate operations as dissecting fruit flies. On the electron microprobe, implementation of mainframe computer reduction of analytical data and tape deck readouts to eliminate the use of computer cards, kept it current and functioning. New spectrometer crystals improved greatly the capability for analysis of the light elements Na, Mg, Al and Si, and allowed Michigan become the one of the first laboratories to undertake routine analysis for the ultra-light element fluorine. However, our most ambitious project was undoubtedly the design, with the help of Professors Roy Clark and John Bardwick of the Physics Department, and construction of an electron energy-loss spectrometer for the STEM.

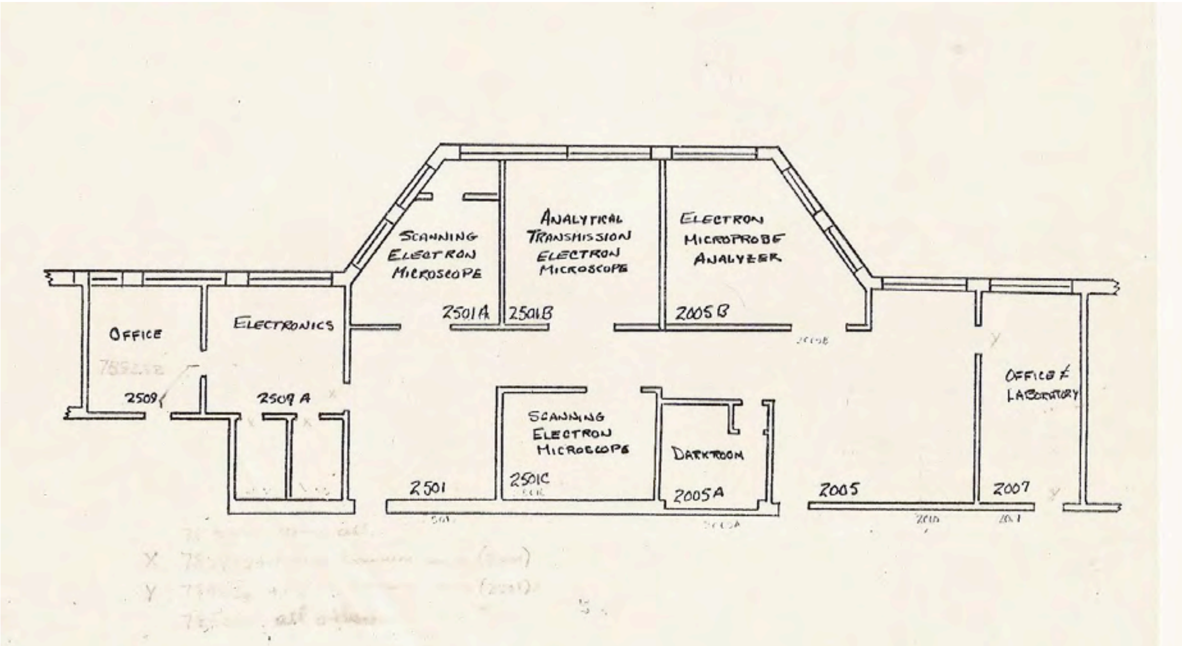


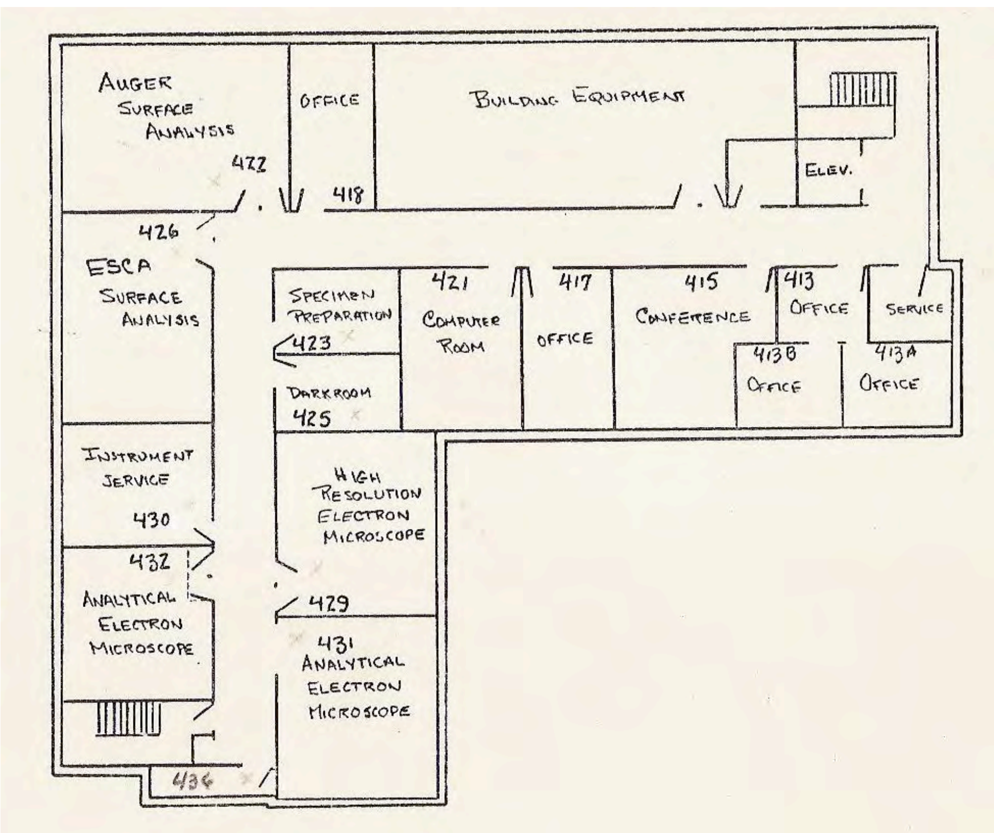
The High Resolution Electron Energy-Loss Spectrometer

This device had a magnet with a larger radius, and thus provided spectra with better resolution, than any other EELS in the country at the time,

In the early 1980s a very serious problem arose. For nearly thirty years the College of Engineering had been planning to move from its two buildings on East University Avenue to the North Campus. In 1981 James J. Duderstadt became Dean of the College, and one of his first official acts was to declare that the move was to be made as promptly as possible. By 1985 the move was essentially complete, and EMAL became virtually the sole occupant of the East Engineering Building, with the threat of eviction when the building was renovated to meets the needs of the College of Literature, Science and the Arts. Fortunately, the importance of the laboratory was recognized by the Deans of the several Schools and Colleges whose faculty had been making use of its facilities, and a widespread search was launched to find new quarters for it that were centrally located and easily accessible to the entire University, and yet were large enough to hold all its instruments so that they could easily be supervised and maintained. Sites in the School of Public Health, the Medical School, the Dental School and the Chemistry Building, were considered, but none was found that was satisfactory. After extensive deliberation and debate, typical of the academic decision-making process, I finally recommended to the Vice President of Research that the Laboratory be divided into two units, one to remain on the Central Campus, equipped particularly to serve the needs of the natural and physical sciences groups located there, and the other to be located on the North Campus and equipped towards the needs of the engineering Departments located there.

The response to this proposal was immediately favorable. The Geological Sciences Department remodeled a little-used lecture room in the C C Little Building to provide excellent space for the Central Camp[us branch, containing the Cameca microprobe, the Hitachi S-570 SEM, and one of the JSM-U3 SEMs. Carl Henderson was hired to manage this laboratory. The College of Engineering added a basement under an addition that was being made to the Space Research Building and made





Top. The EMAL Laboratory in the C C Little Building on Central Campus. Bottom. The North Campus Laboratory in the Space Research Building that available for the North Campus branch. The STEM and the second JSM-U3 were moved into it. This turned out to be an exceptionally favorable arrangement. Members of the Geological Sciences Department were the major users of the microprobe, and this instrument was now located in the building their department occupied. Members of the Biological Sciences Division were the predominant users of the SEM, and it was now located in close proximity to their building. Most research workers doing high resolution transmission electron microscopy were on the North Campus. In addition both laboratories had space to accommodate new instruments.

In 1986, through a grant from NSF plus matching funds from the College of LS&A and the Vice President for Research, Professor Donald R. Peacor of the Geological Sciences Department obtained a Philips CM-12 STEM which was installed in the Central Campus laboratory, and which has been used extensively in studies of the microstructures and compositions of minerals. Shortly after he became Chairman of the Department of Materials and Metallurgical Engineering Professor Ronald Gibala obtained funding from the College of Engineering to purchase of two advanced TEMs. One instrument, a JEOL 2000FX with a scanning transmission system (STEM), an EDXS unit and an EELS spectrometer, was designed for analytical studies. The second instrument, a JEOL 4000EX, high resolution TEM that operated at a 200 kV electron accelerating voltage and had a TV camera system attached, was reserved for high resolution work, Because Larry Allard, John Mardinly and Steve Krause had all obtained their Ph. D. degrees by this time and had left the University, Professor David Van Aken took charge of the installation and management of these two instruments.

In 1985 the Perkin Elmer Company offered several universities an opportunity to purchase one of their surface analysis instruments for half price. Such instruments are capable of obtaining analytical data on the outermost one or two atom layers of a solid specimen, and although such an instrument would be highly useful in a number of areas of research, none was available in the entire University. I therefore pointed out to Linda Wilson, who was then our newly appointed Vice President for Research, the fact that this would be too much of a bargain to pass up, and she obligingly purchased a PHI Model 5400 X-ray Photoelectron Spectrometer (XPS) which was placed in the North Campus facility.

Linda Wilson, V. P. for Research

Hal Estry, & George Brooks at the Phi 5400 XPS

One unique feature of EMAL early on was the practice of making its instruments widely available to faculty and students throughout the University. In the 1950s the general policy of the National Science Foundation was that a sophisticated instrument such as a transmission electron microscope should be restricted to use by a single faculty research group. I, on the other hand, believed that this was not a very efficient use such expensive and 'hard-to-obtain' instruments, because under such circumstances they were often not used to their full capability and often stood idle for a good fraction of the time. Therefore, when the ARL microprobe was obtained I openly advertised its availability throughout the University. With the capable assistance of Frank Drogosz, our department's instrumental laboratory technician at that time, this approach turned out to be very successful, and so was continued when the later instruments were acquired. Most years we had over one hundred regular users. Initially, funding for operation of the laboratory was generously provided by contributions from the Deans of the College of Engineering, the College of Literature, Science and the Arts, the Dental School, the Medical School, and the School of Public Health. Eventually, as the number of instruments increased operating costs rose to the point where it became necessary to add a modest hourly charge. I was assisted in setting Laboratory policy by an Executive Committee which consisted of faculty members from several departments; long term members included Professors Donald Peacor and Eric Essene of the Geological Sciences Department, Johannes Schwank of the Department of Chemical Engineering, Kamal Asgar of the Department of Dental Materials, and Roy Clark of the Physics Department.

Wilbur C. Bigelow

In 1987 Professor Gibala wisely hired Dr. John Mansfield to take over management of the North Campus Branch. With both branches of the laboratory now firmly established and well managed, I resigned as Director, turning that position over to Professor Donald Peacor of the Geological Sciences Department. During the twenty years I had served as its Director the Laboratory had been instrumental in bringing the *first* electron microprobe analyzer, the *first* scanning electron microscope, the *first* analytical scanning transmission electron microscope, and the *first* surface analysis instrument to the University. In addition, it had made these powerful research tools available for use by hundreds of scientists throughout the University. All of this was, of course, made possible by the exceptional cooperation and support I had received from faculty members and from administrators at all levels throughout the University.