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TECHNICAL PROGRESS REPORT
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Covering
Electron Microscope Studies
For the period June 1, 1967 through May 31, 1968

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TECHNICAL PROGRESS REPORT*

(June 1, 1967 to date)

On the first of June, 1967, the electron microscope was still at Argonne National Laboratory. Since that time new laboratory space has been provided for the microscope in the Enrico Fermi Institute at the University of Chicago, and the microscope has been relocated on the campus. Two new facilities have been put into operation. One of them is a field emission electron gun designed to operate at 100 KV and to form the basis for a new microscope. The other is a simple scanning microscope constructed from spare parts from our original machine, and whose purpose is to allow independent studies of energy loss phenomena which may be of value in obtaining picture contrast.

In addition to these changes in location and equipment, we have added some new personnel to the project in the form of Research Assistants, Engineers, Students and a Research Associate.

Finally, the original microscope has been improved with the addition of a new lens in place of the old RCA lens; a move which has increased the resolving power so that it is now between $10 \text{ \AA}$ and $20 \text{ \AA}$ (a figure which had been thought to be impossible for a scanning microscope).

*The research described in the following pages was supported by AEC Contract AT (11-1)-1721.

A. LABORATORY FACILITIES

The University of Chicago made available approximately 2000 square feet of laboratory space in the ground floor of the Enrico Fermi Institute. (This space was previously utilized by Professor Allison.) The space was completely refurbished in order to provide an adequate environment for microscope work. The rooms were tiled with ceramic tile from floor to ceiling, provided with new lighting and completely air conditioned with highly filtered air. The net effect has been that this laboratory space is very clean indeed, and has provided us with excellent working conditions. For those occasions when absolute cleanliness is required, we find that portable clean air hoods are perfectly adequate. The space is divided into three laboratory modules: one we are using to house the microscopes, the second is primarily a specimen preparation and tip-making area, and the third is a general purpose laboratory.

In addition to these three laboratories, we have now been allocated a fourth laboratory on the same floor of the Enrico Fermi Institute, into which we will be able to move much of our general purpose laboratory work insofar as this new space is not as clean as the other space and is more suitable for general laboratory work.

Refurbishing of this laboratory space was provided at no cost to the Atomic Energy Commission, but was supported by a National Science Foundation Institutional grant in the amount of nearly \$60,000.

B. 30 kV MICROSCOPE

This microscope is the original one which was first constructed a few years ago at Argonne National Laboratory and subsequently brought to the University of Chicago. We have chosen the name "30 kV Microscope" because its normal operating range of voltages is from 15 to 30 kilovolts. While we are quite sure that we can ultimately increase the voltage to about 50 kV, we have not tried this so far because of a tendency for a high voltage breakdown to occur; an event which can easily cause a 24-hour delay in our program due to the destruction of the field emitting tip. As our other instruments come into operation we will more easily be able to afford the luxury of experimenting with ways to increase the voltage range of the microscope.

This microscope is the one which has been described in our recent publications and in last year's proposal. It consists basically of an electron gun with a field emission tip, followed by a magnetic lens to focus an image of the field emission tip on the specimen. After passing through the specimen, the electrons may be subjected to energy analysis.

1. Resolution Studies with the RCA Lens

For the last year or so our studies have concentrated on trying to understand the imaging processes of the electron gun when used in conjunction with a particular magnetic lens, which was an RCA intermediate lens. This lens does not have a very high performance, but nevertheless it was a sufficiently good lens that

we were able to measure the resolution of the microscope as a function of various parameters, and at the same time we could calculate the performance based upon our knowledge of the lens and the electron gun.

This particular phase of the work has now been completed and a report on this will be submitted shortly for publication.

In summary, we believe that we now understand the imaging process in the sense that we can calculate the expected resolution for any lens and gun combination. As an example, the best resolution that we could expect with the RCA lens was $20 \text{ \AA}$, and experimentally the best resolution obtained was $40\text{--}50 \text{ \AA}$.

2. Energy Loss Studies

The experimental work on energy loss studies for the last few months has been confined to measurements on the electrostatic spectrometer itself. These measurements were difficult to perform because of the various perturbing aspects, such as stray magnetic fields, the movement of elevators, etc., but nevertheless we have been able to confirm that the spectrometer is behaving properly and is as good as one could expect such a spectrometer to be. The resolution we have been able to get with the spectrometer is still the same value that we quoted before; namely about 0.3 volts in 20,000 volts.

3. Installation of the New Lens

We have very recently installed in the microscope our new lens in place of the RCA lens. This new lens was designed by us and the bulk of the fabrication was done in the Argonne Central Shops. The most difficult part of the fabrication was the machining of the pole pieces. This work was given to an outside fabricator because of the nature of the task, but when it was returned to us at the University of Chicago it was decided that an attempt should be made to improve these pole pieces. This task was undertaken by Mr. Walter Mankawich, our Development Engineer, with great diligence and patience, and he ultimately produced a lens which is almost certainly the best ever made. The final measured tolerance on the essential dimensions of the pole pieces was no greater than .02 mils.

This lens was designed to work in an ultra-high vacuum system so that the excitation coil is completely shrouded in water-cooled copper and the lens was so designed that the specimen is located at the point of maximum magnetic field. This is the optimum location from the point of view of spherical aberration but is not commonly done commercially because of the difficulty of inserting the specimen and providing the usual specimen motions. We appear to have been able to solve these engineering problems satisfactorily, although as we shall point out later, there may be better solutions.

A new electron gun was designed so that it would fit accurately on top of the new lens and so that the two, together, form an essentially monolithic

structure which is the beam forming system. In this way the essential components of the microscope are much less subject to vibrations and relative displacements.

This system was so accurately made that it worked on virtually the first attempt and has shown few if any signs of misalignment.

The lens has only been in operation for a week or so, but nevertheless it would appear to have already demonstrated a resolution which is probably better than $20 \text{ \AA}$.

C. 100 kV GUN

This is the system which was under construction last year. This gun has been very carefully designed for high voltage and ultra-high vacuum operation. It has now been completely assembled and leak-tested, and has successfully withstood 80 kV. More recently a field emission tip has been successfully operated in the gun and work is underway in order to provide all the necessary voltages for the complete operation of a high voltage electron source.

It is this electron gun which we ultimately intend to provide with a cylindrical magnetic lens similar to the one which is now in the 30 kV Microscope, and also provide with a quadrupole-octupole correction system for the purpose of investigating the possibility of eliminating spherical aberration.

D. GUN MICROSCOPE

Due to the changes which were made on the 30 kV Microscope, a significant amount of internal microscope hardware became available, such as a complete field emission electron gun, the specimen motions, specimen block, etc. As we also had available the necessary high vacuum pump, all that was necessary to provide another working microscope was a vacuum box which was a relatively minor matter. In addition it was felt that if we could provide this additional medium performance instrument we would be able to increase the availability of our new technology and begin work more rapidly on studies of energy loss phenomena.

This new microscope is of great simplicity and could ultimately be the source of significant cost savings in scanning microscopes. As is well known the normal commercial scanning microscope, such as the Cambridge Stereoscan, can provide a resolution of between $100 \text{ \AA}$ and $500 \text{ \AA}$ ($500 \text{ \AA}$ guaranteed, $100 \text{ \AA}$ with patience), and consists of an electron gun with 4 or 5 auxiliary magnetic lenses. Our new gun microscope consists simply of one of our field emission guns with no other lenses, and has a calculated best resolution of about $150 \text{ \AA}$. The potential cost savings are obvious.

The gun and specimen motion were placed in a versatile vacuum box which has numerous auxiliary ports for the installation of whatever detection equipment one chooses. The graduate students who are beginning to work with this instrument

are concerned primarily with energy loss mechanisms and will therefore be interested in the energy analysis of transmitted electrons. There are other interested parties, however, such as Dr. Uretz of the Department of Biophysics at the University of Chicago, who would be interested in the use of such an instrument to study cathode luminescence from selectively stained samples. It may also be that secondary emission pictures would be useful.

The display system which is being used for this microscope originally formed part of the display system for the 30 kV Microscope.

E. AUTOMATIC CAMERA

This camera has now been completed and tested. It is in routine daily operation and is now arranged so that either of the two working microscopes mentioned above can be switched into it when it is desired to take a photograph. The pictures are of very high quality, as can be seen from the enclosed samples. This camera was constructed for us by the Electronics Division at Argonne National Laboratory, and we are pressing them to prepare a report for publication because we feel this type of camera could have wider applications.

F. THEORETICAL STUDIES

Our theoretical studies of multipole lens systems have continued and we now believe that we have a design for such a system which at least in principle can

eliminate spherical aberration and therefore improve the resolution of our microscope. The basis for our design was discussed at the Argonne Conference on High Voltage Electron Microscopes in August of 1966. We have applied these ideas to the case of a 50 to 100 kV microscope, and the result seems to be that such a system is indeed feasible. It consists of a spherical aberration correcting device which is to be inserted between the electron gun and a conventional lens. The correcting system consists of four quadrupole elements and three octupole elements. We found that by carefully choosing the parameters of the cylindrical lens and the sizes of the quadrupoles and octupoles, it is theoretically possible to introduce into the cylindrical lens an electron beam which has exactly the opposite spherical aberration as the lens itself. If this is true then the only aberrations which would be left would be chromatic aberrations and aperture aberrations of higher order, and they would presumably have less effect than the third order spherical aberrations.

This system has been completely designed and fabrication of it has begun. The fabrication will be extremely difficult because of the large number of magnetic pole pieces involved and because of the very tight mechanical tolerances which will have to be placed on them.

Other theoretical studies are in progress not only on other possible multipole systems but also on possible improvements in our electron gun technology.

Only a minor amount of work has been done on superconducting lenses because the successful exploitation of superconducting technology awaits a bright idea rather than straight-forward design. It is easy to demonstrate that the superconducting lens of today has no advantage over the conventional lens, other than the stability of its magnetic field. In our case, stability is not yet a great problem - and in any case when it does become a problem it might be wiser and more economical to spend money on stabilizers rather than on the expensive superconducting technology.

Calculations have also been performed on new types of spectrometers with improved performance which could be used for energy analysis of the transmitted beam of electrons. Calculations so far indicate that a magnetic spectrometer can be built with a performance which exceeds that of our current electrostatic spectrometer. We believe it may be possible to achieve a working resolution of about one part in a million. At 100,000 volts the resolution would then match the energy spread of electrons from a field emission tip, and would therefore be the optimum performance.

PERSONNEL

1. ALBERT V. CREWE, Principal Investigator

Born: February 18, 1927, in Bradford, Yorkshire, England.

Education: B.S., Physics, University of Liverpool, 1947
Ph.D., Physics, University of Liverpool, 1951

Appointments:

Assistant Lecturer, University of Liverpool, 1950-52;
Lecturer, University of Liverpool, 1952-55;
Research Associate, The University of Chicago, 1955-56;
Assistant Professor of Physics, The University of Chicago, 1956-59;
Associate Professor of Physics, The University of Chicago, 1959-63;
Technical Director of the Synchrocyclotron at the University of Chicago,
1956-58;
Director, Particle Accelerator Division, Argonne National Laboratory,
1958-61;
Director, Argonne National Laboratory, Argonne, Illinois, 1961-67;
Professor, The University of Chicago, 1963 to present.

Societies: Fellow of the American Physical Society;
Fellow of the American Nuclear Society;
The Electron Microscope Society of America (EMSA);
The Scientific Research Society of America (RESA).

2. MICHAEL G. R. THOMSON, Research Associate

Born: March 7, 1941, in Portsmouth, Hampshire, England.

Education: B.A., Natural Sciences, University of Cambridge, 1962
Ph.D., Physics, University of Cambridge, 1967

Dr. Thomson has recently joined us from the Cavendish Laboratory in Cambridge where he recently completed his thesis on quadrupole systems. His thesis supervisor at the Cavendish Laboratory was Dr. Peter Hawks, who is perhaps the world's expert on the theoretical aspects of quadrupole systems. Dr. Thomson is particularly interested in the use of computers to solve electron optical problems, and is at the moment engaged in studying our quadrupole-octupole corrector system and also our electron gun design.

3. GRADUATE STUDENTS

David Glockner	graduate student in Physics
*Michael Isaacson	Physics
*Dale E. Johnson	Physics
Paul Lin	Physics
Gerald I. Saxon	Physics
*Joseph Wall	Biophysics

*Supported in part or in total by sources other than AEC.

4. ENGINEERING SUPPORT

a. Walter Mankawich, Development Engineer

Mr. Mankawich has been at the University of Chicago for a number of years and we are very fortunate to be able to acquire him for this project. He has long experience in the design and fabrication of high precision equipment. He performed the final machining on our new microscope lens and the final product was a lens which is almost certainly the most precise lens ever fabricated. Currently he is performing the mechanical design and fabrication of the quadrupole-octupole corrector. One of the principal problems of this system is the need for very high precision in fabrication.

b. Sollie Lucero, Design Engineer

Mr. Lucero is a Design Engineer who has been at the Institute for some time. He was previously involved in the design of bubble chambers and related equipment, and has been loaned to us for the next year by Dr. Hildebrand. He is engaged in the design of a new specimen changer which, if successful, will be of great value not only to us but to other microscope users.

c. John Erwood, Electronics Engineer, and Walter Edwards, Associate Electronics Engineer

Mr. Erwood and Mr. Edwards worked with Professor Allison before his untimely death. Both men are skilled electronic engineers and have been at the University for several years (Erwood since 1957 and Edwards since 1963). They were extremely valuable during the past year as they shouldered the major burden of overseeing the refurbishment of the laboratory space at the University, and in getting the microscopic equipment reassembled after the move from Argonne. At the present they are working in their capacity as Electronic Engineers in support of the fabrication of the microscopes, quadrupole-octupole lens, electron gun, and related efforts.

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4. J. W. Butler, A. V. Crewe and L. M. Welter; A Computer-Controlled Electron Microscope System; abstract of paper presented at MICRO-67, Churchill College, Cambridge, England (1967).
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6. A. V. Crewe, D. N. Eggenberger, L. M. Welter and J. Wall; Experiments with Quadrupole Lenses in a Scanning Microscope, Journ. of Applied Phys., 38, 4257 (1967).
7. A. V. Crewe, D. N. Eggenberger, J. Wall and L. M. Welter; An Electron Gun Using a Field Emission Source; to be published in The Review of Scientific Instruments (1968).

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9. A. V. Crewe, M. Isaacson and D. Johnson; A Simple Scanning Microscope of Medium Performance; abstract of paper to be presented at the Fourth European Regional Conference on Electron Microscopy, September 1968, in Rome.
10. A. V. Crewe, D. Cohen and P. Meads; A Multipole Element for the Correcting of Spherical Aberration; abstract of paper to be presented at the Fourth European Regional Conference on Electron Microscopy, September 1968, in Rome.

SAMPLE PHOTOGRAPHS
TAKEN WITH AUTOMATIC CAMERA

These pictures were taken very recently just after we installed our new lens.

The pictures are of sperm tails and the specimen was given to us by Dr. Tahmisian of Argonne National Laboratory. They are taken with different magnifications which are indicated on the pictures. Perhaps the most interesting are the last two pictures.

These pictures were taken several minutes apart and are of the same area of the specimen. In the areas marked on the two pictures, one can discern objects which are $20 \text{ \AA}$ or less in size. The fact that these small objects appear on the two separate pictures is the best indication we have at the moment that our resolution is indeed better than $20 \text{ \AA}$.

INDEX TO PHOTOGRAPHS

SPECIMEN: TK(3) Helix Aspersa Sperm Tails.
Fixed with Osmium, sectioned and embedded
in EPON EPOXY resin, and lead stained for
30 minutes.
(Made 12-18-64)

1. Electron Energy = $V_o = 20.0 \text{ KeV}$ (for all pictures).
 2. All pictures taken on 10 second scan.
 3. Pictures #4 and #5 are identical but taken 1 minute apart.
-

<u>PICTURE</u>	<u>SCALE</u>
1	1200 $\overset{\circ}{\text{A}}/\text{cm}$
2	400 $\overset{\circ}{\text{A}}/\text{cm}$
3	120 $\overset{\circ}{\text{A}}/\text{cm}$
4	40 $\overset{\circ}{\text{A}}/\text{cm}$
5	40 $\overset{\circ}{\text{A}}/\text{cm}$

2400 Å
|—|

#1

800 Å

240⁰Å

#3

80 Å

#4

80 Å