MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS:1963-A
The program is a collaboration between the University of Chicago and Hughes Research Labs. The major goal is to produce two 60 KeV high resolution (10-100 A), high current density (1 A/cm²) ion microscopes. The project is partly funded by the NSF. During the first year of the program, the basic optical and overall engineering design of the two ion microscopes was consolidated, with the procurement and construction phase being completed near the end of the second year.
Research Objectives for the Second Year

The University of Chicago (UC) and Hughes Research Laboratories (HRL) collaborative program aims at the development and construction of two 60 keV high resolution (10-100 Å), high current density (1A/cm²) ion microscopes/microprobes over a three year period. A pictorial summary of the organization of the project and its funding is contained in Fig. 1. Two sets of coordinated technical and scientific tasks form an integral part of the UC-HRL program. A phasing diagram for these tasks is given in Fig. 2, also showing the repartitioning of DOD (through AFOSR) and NSF support among the various tasks.

The research objectives for the second year of the program, comprehensive of both the UC and HRL effort, are incorporated in the following main tasks:

1. New Ion Microscope Development

After having consolidated the basic optical and overall engineering design of the two ion microscopes during the first year of the program, the procurement and construction phase was to be carried out to completion during the second year.

2. Study of Physical Processes Yielding Image Contrast in Ion Microscopy

This continuing task was to be pursued using the existing UC-SIM, modified during the first year to operate with a Ga⁺ liquid metal source. Objectives were the study of secondary electron and secondary ion emission under gallium ion bombardment of solid targets. This exploratory program was meant to provide a better understanding of the phenomena to be exploited with the new ion microscopes in various research applications. Furthermore this part of the program fulfills the very important role of training graduate physics students in research, while providing suitable topics for their Ph.D. theses.

Status of the Research Effort

Since last year's report (July 1981) the program has progressed most satisfactorily. The accomplishments to date within the scope of each of the above tasks are described in the following summaries.
1. Progress on New Ion Microscope Development

a) Ion Microscope Design. Since last year's report, the design of the entire microscope system has undergone close scrutiny, and several modifications and improvements have been introduced. Updated layouts, corresponding to those contained in last year's report, are shown in Fig. 3 and 4. Details of the microprobe specimen chamber are shown in Fig. 5. Further aspects of the design have been consolidated. These include:

-- specimen inserter and stage motions
-- SIMS system
-- control and data acquisition electronics
-- data processing techniques.

A versatile specimen inserter system has been added to the microprobe design. It will allow the exchange of 12 specimens in one loading (pump-down of the air-lock chamber), eliminating the need to wait while new specimens are pumped down. In addition, the specimen motions (Perkin-Elmer x-y-z + 12 positions) will be modified to provide x-y motions plus tilts about two axes and 12 specimen positions. The specimen height will have a range from 1.6 cm (only secondary electrons) down to 3.1 cm (SIMS and secondary electrons).

Between the final (einzel) lens and the specimen will be located the SIMS energy analyzer and 90° deflector which will collect ions emitted upwards from the specimen and direct them into the quadrupole mass spectrometer (Fig. 6). The geometrical collection efficiency of the energy analyzer determines the minimum detectable amount of material ionized from the specimen. Therefore we are still attempting to optimize the analyzer design, aiming for overall efficiencies of at least 50%, comparable to commercial microprobes.

The most important element in achieving an easily usable analytical instrument is making the controls "friendly". This means that the various adjustments to the optics and data acquisition must be straightforward and reliable. To accomplish this, the control and data acquisition electronics have been designed using state-of-the-art microprocessor-based digital and analog circuits. All control and data variables will be displayed on the computer monitor screen and most adjustments will be set by an x-y joystick. In addition, various data processing techniques will be available through the microprocessor (a Z-80 connected to an S-100 bus) such as Fourier transforms, spatial frequency analysis and filtering, differentiation and
integration and contrast enhancement. The microprocessors will control the specimen position (d.c. raster deflection), stigmators, alignment position, channeltron voltages, raster size, raster elements (16 x 16 up to 1024 x 1024), scan speed (0.5 μsec/pixel up to 16 μsec/pixel), type of scan mode, the parameters involved in image processing (bias, gain, bandwidth, differentiation), the SIMS energy analyzer and SIMS mass selection.

b) Parts Fabrication and Procurement. A substantial amount of the total parts fabrication and procurement has been completed as of May 31 1982 as shown in "Program Schedule of 30 June 1982" (Fig. 7). The high voltage rack is awaiting only the installation of two 500 l/s ion pump controllers. Tests have been completed for the high voltage connectors between the Bertan supplies and the optical column. The vacuum system, comprising the vacuum chamber, support table, 500 l/s ion pumps, specimen inserter and foreline with three sorption pumps, is now being built by Torr Vacuum Products and will be delivered to HRL by the end of July. The various vacuum flanges have been manufactured by MDC Manufacturing, Inc. and delivered.

The optical columns have been machined by the HRL shop and were completed as of 31 May 1982. After this, a period of about eight weeks has been allotted for cleaning, assembly and alignment at which time the two optical columns will be ready for insertion into the vacuum system.

The ground-based electronics is being fabricated largely by MT Systems, Inc., after close consultations with HRL and UC. Purchases have been divided into those which are off-the-shelf commercially available digital electronics and the custom digital and analog circuitry for controlling the optical column and data acquisition electronics. The first subsystem (the commercially available components) is now running and was delivered to HRL in early April. The control program (written in BASIC for easy modification and expansion) has already been written and is operational. The remaining electronics will arrive after extensive testing at MT Systems and should be ready about the time the optical column is together by the end of July.

The schedule shows system integration to be complete by mid-September, with a 10-100 Å probe demonstrated by mid-October. The final design of the SIMS energy analyzer is still being discussed, to take advantage of the latest advances in the field, of which we became recently aware. The quadrupole mass filter and
the SIMS control electronics have been purchased. After preliminary vacuum, high-voltage and control electronic tests at HRL, one microprobe will be delivered to UC in November.

c) UC-HRL Contacts. Beyond last-year's report, the UC and HRL teams have met on several occasions, in April 1981, July 1981, and in April 1982. Furthermore, Mr. N. W. Parker, a graduate student of Prof. A. V. Crewe at Chicago, joined HRL in the Fall of 1981 and splits his time between UC and HRL. This has provided a most effective liaison between the two collaborating groups.

2. Progress in the Study of Physical Processes Yielding Image Contrast in Ion Microscopy

This aspect of the UC program is yielding important and exciting results. We have investigated various contrast mechanisms based on secondary electron and secondary ion emission and performed sputtering tests with the prototype 60 keV Ga⁺ UC-SIM. In addition to contrast due to the surface topography, the SIM was found to yield strong elemental contrast (Z-contrast) and dramatic crystallographic contrast due to ion-channelling effects.

a) Topographic Contrast. We have established that the SIM can give remarkably good contrast due to the surface topography of any kind of material, by collecting the secondary electron signal. We have extended the exploration reported in last year's report to include also biological objects and integrated circuits. We observe that the gallium implant often makes insulators conducting enough to eliminate charging effects. Thus it is generally not necessary to coat insulating specimens with heavy metals, like is done in the SEM, to avoid the detrimental effects of local fields. As examples, Fig. 8 shows details of an uncoated specimen of fruit fly (Drosophila melanogaster); Fig. 9 shows the progressive disappearance of charging effects in the observation of uncoated human red blood cells; Fig. 10 shows that, after SIM scans, red blood cells became conducting enough to be observable also in the SEM. Furthermore, Fig. 11 shows details of an integrated circuit, covered by passivation layers, which can be imaged without any difficulty in the SIM. Fig. 12 shows that in the SEM, similar focusing cannot be achieved due to charging effects. However, after gallium implant in the SIM, SEM focusing becomes possible.

b) Z-Contrast. We have determined that the contrast due to the Z-dependence of the secondary electron emission yields is very pronounced in our Ga⁺ SIM and differs from that provided by the SEM. An example is shown in Fig. 13. This investigation
will be extended to survey systematically a much greater spectrum of elements. The results obtained thus far confirm our expectation that Z-contrast in ion microscopy may become a very useful tool for the visual discrimination of surface microstructures due to elemental segregation.

c) Crystallographic Contrast. SIM imaging of polished samples of metals and alloys was found to be dominated by ion-channelling effects, yielding very pronounced crystallographic contrast in both secondary electron and secondary ion images. We have concentrated on this novel aspect of scanning ion microscopy which opens up a vast horizon of immediate practical applications in material science, metallurgy and semiconductor technology. A summary of relevant results is as follows:

1) The ion beam scan cleans the specimen surface of contaminants, absorbed layers and oxides, while imaging. The image contrast is observed to evolve until a stable configuration is reached in a few minutes. This is particularly noticeable on polished surfaces of non-noble metals (see Fig. 14).

2) Secondary electron images of crystalline but chemically uniform samples (such as, e.g., recrystallized, polished, etched α-brass and Cu) show dramatic evidence of crystallographic contrast, when only surface topography is the source of contrast in SEM images (see Fig. 15, 16). Upon rotating a sample by, e.g., 10°, the contrast between crystallite pairs may completely reverse (see Fig. 17, 18). These effects have been understood in terms of the dependence of the secondary electron yields on lattice orientation with respect to the incident beam due to primary ion channelling. The contrast effects observed are large, the signal level changing up to a factor of three when comparing channelling with non-channelling conditions. Similar observations have been performed on a sample of recrystallized, polished die steel (Fig. 19) and are in progress on Si samples.

3) We have shown that ion-channelling contrast can be applied to the detection of shock-induced defects in single crystals of meteoric iron (structures known as Neumann bands, see Fig. 20).

4) Crystallographic contrast is present also in secondary ion images in definite relationships to that observed in secondary electron images.
Comparisons of this kind are valuable for a better understanding of the mechanism of secondary ion emission (see Fig. 21, 22) to be exploited for SIMS analysis.

v) Ion-channelling effects on the sputtering rates have been probed by ion beam writing across crystallite boundaries (see Fig. 23). Quantitative results have been obtained on the crystal orientation dependence of writing speeds.

From the above it appears that the SIM may be a very valuable tool in material sciences and metallurgy, providing bulk sample information even through the immediate secondary electron imaging. The addition of analytical capabilities, such as being implemented in the new instruments under construction, will clearly open up a much broader range of applications. The above results have been presented at several international meetings (see list of publications) and have attracted the attention of the news media. Reports have appeared in Science News, Vol. 121, NO. 8, February 20, 1982, p. 118, Science 82, May 1982, p. 10, Industrial Research and Development, March 1982, p. 91. A cover story in Physics Today is scheduled for the July 1982 issue.

Research Plans for the Coming Year

We plan to follow our schedule of tasks described in our original proposal and outlined in Fig. 2. Clearly the first priority will be that of bringing to completion the construction of the two new ion microscopes/microprobes and to demonstrate and evaluate their performance. This task will be pursued in parallel at UC and HRL. In essence this will consist in:

a) establishing the resolution limits of the instruments;

b) measuring the probe current vs. probe size for both modes of ion source operation (field ionization and liquid metal);

c) bringing into operation the SIMS systems and

d) at UC, bringing into operation also the magnetic sector spectrometer for transmission measurements, to be transferred to the new instrument from the existing UC prototype.

This phase will merge with the task of initiating research applications of the new microprobes. The activity which is presently being carried out will greatly facilitate the choice of immediate fruitful applications. At UC, we want to pursue
and reexamine, at improved spatial resolution, much of the exploratory work presently in progress. The most promising areas of applications of the image contrast features of the SIM are in the study of metals and semiconductors, aimed at the detection of lattice defects, dislocations, impurities and micro-
structures which might be detectable due to ion-channelling effects. We also wish to complement this study with elemental microanalysis by SIMS. We have made definite collaborative plans with Prof. Hellmut Fritsche toward a joint study of the characterization of microstructures in amorphous semiconductors, using our new SIM/SIMS. We are involved in a preliminary survey of applications to the study of meteorites in collaboration with Drs. Edward J. Olsen and Lawrence Grossman of our Department of Geophysical Sciences. Arrangements for the study of biological specimens have been made with a number of scientists at UC. Jointly with HRL we will also pursue studies of channelling effects relevant to problems in semiconductor microfabrication.

Substantial progress has also been made at HRL toward the choice of research applications to be implemented with the new instrument. A specific topic involves the problems in the manufacture of Ga-As FET devices. This will be the initial test of the usefulness of the microprobe to an actual problem of great current interest. The SIMS capability of the microprobe will be used to search for the causes of the substantial non-uniformity of Fa-As FET characteristics, unexplainable by other techniques.

Within the scope of the current program, only an initial exploration along the above lines of research will be permitted. These topics and other ones will be taken up again in more detail in a new proposal for continuation of our work with the new high-resolution microprobes, presently being prepared.
Personnel

1. Research Associates

Dr. Timothy R. Fox, who obtained his Ph.D. in 1980 and has participated in our program until March 1982, has joined an industrial concern. He was replaced by Dr. Paul H. LaMarche, a graduate of Yale University, who joined our group on May 1, 1982. Dr. LaMarche has excellent credentials in our field of specialization.

2. Graduate Students

Mr. Kin Lam is making excellent progress toward his Ph.D. degree. His thesis topic relates significant results obtained with the STIM on the observation of anomalous energy losses in the traversal of thin carbon foils by molecular hydrogen ions. His results, presented by R. Levi-Setti in an invited paper at the US-Japan seminar on Ion Penetration Phenomena in Solids, Honolulu, Hawaii, January 1982, have attracted much interest.

A new graduate student, Mr. Thomas H. Shields, joined our group in October 1981.

3. Undergraduate Students

During the summer of 1981, Mr. David Cousins worked in our group as an NSF Undergraduate Research Participant.

Mr. Daniel J. Welsh and Mr. Larry Whitlow are employed as undergraduate student technicians.
Cumulative Chronological List of Written Publications in the Technical Journals

(a) Published and in press


5. Scanning Microscopy with Gallium Ions from a Liquid Metal Source.


7. Ion-Channelling Effects in Scanning Ion Microscopy with a Ga<sup>+</sup> Probe.

8. Ion-Channelling Effects in Scanning Ion Microscopy and Ion Beam Writing with a 60 keV Ga<sup>+</sup> Probe.

10. Ion-Channelling Effects in Scanning Ion Microscopy with a 60 keV Ga+ Probe.
    R. Levi-Setti, T. R. Fox and K. Lam; to be published in Nuclear Instruments and Methods.

(b) In course of preparation

11. \( \text{H}_2^+ \) Traversing Ultra-Thin Carbon Foils: The Energy Loss of Two Correlated Protons Below the Fermi Velocity.
    K. Lam; to be published in Nuclear Instruments and Methods.

(c) Theses

1. Hydrogen Ion-Solid Interactions Near the Bohr Velocity.

Interactions (Coupling Activities)

(a) Spoken papers presented at meetings, conferences, seminars.


2. Information and Dose in the Scanning Transmission Ion Microscope.

3. Same title as Publications (a) 2. Paper presented by R. Levi-Setti at the Bat-Sheva Seminar on Molecular Ions, Molecular Structure and
Interaction with Matter; Israel, January 1981.


7. Same title as Publication (a) 6. Paper presented by T. R. Fox at the 28th Int'l Field Emission Symposium, the Oregon Graduate Center, July 1981.


12. Imaging and Crystallographic Contrast with a 60 keV Ga⁺ Scanning Ion Microscope.

### UC Tasks

- **Task 1.** Design studies using the existing STM
- **Task 2.** New ion microscope development
- **Task 3.** Study of physical processes yielding image contrast in ion microscopy
- **Task 4.** Performance evaluation of UC STM-IMMA
- **Task 5.** Research applications of UC STM-IMMA
- **Task 6.** Reports
- **Task 7.** Travel

### NML Tasks

- **Task 1.** Focusing limit of existing column IMAO programs
  1. Theoretical analysis
  2. System modification
  3. Experiment evaluation
  Milestone 1: 10Å resolution or end source size demonstrated

- **Task 2.** New ion microscope development
  1. Preliminary design
  2. Engineering design
  3. Parts fabrication and procurement
  4. Assembly and initial checkout
  5. Microscope delivery to UC
  Milestone 2: New microscopes available at UC and NML

- **Task 3.** Performance demonstration
  1. Ion source
  2. Column alignment
  3. Beam focusing
  Milestone 2: Minimum spot size demonstrated (ODAL 10Å radius)

- **Task 4.** Research application of new microscope
  1. Choice of applications
  2. Microscope investigation
  Milestone 4: Novel ion microscope results

- **Task 5.** Reports
  1. Annual reports
  2. Final report

- **Task 6.** Travel

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**Fig. 2**
Fig. 6. Schematics of the detection and analysis system
Fig. 7 Schedule for the completion of the two high-resolution ion microprobes
GA\textsuperscript{+} FOCUSED-ION-BEAM IMAGES

DROSOPHILA MELANOGASTER, UNCOATED
SECONDARY ELECTRON SIGNAL, 60 keV UC-SIM

Fig. 8
Ga⁺ focused-ion-beam images

Uncoated human erythrocytes, critical point dried
Disappearance of charging effects under Ga⁺ implant
55 kV UC-SIM

Initial image

After 7.3 x 10¹⁵ ions/cm²
36 μm full scale

After 1.7 x 10¹⁶ ions/cm²
After 2.9 x 10¹⁶ ions/cm²

Fig. 9
Ga⁺ FOCUSED-ION-BEAM IMAGES
UNCOATED HUMAN ERYTHROCYTES, CRITICAL POINT DRIED
SIM vs SEM IMAGING (AFTER Ga⁺ IMPLANT)

55 kV UC-SIM

10 kV HITACHI HFS-2 SEM

36 μm FULL SCALE

36 μm F.S.

18 μm F.S.

18 μm F.S.

Fig. 10
GA⁺ FOCUSED-ION-BEAM IMAGES
UNCOATED EPROM CHIP (INTEL D 2758)
SIM IMAGING (UNAFFECTED BY SURFACE
CHARGING OF PASSIVATION LAYER)
60 kV UC-SIM

360 μm FULL SCALE
180 μm F.S.
72 μm F.S.
36 μm F.S.

Fig. 11
UNCOATED EPROM CHIP (INTEL D 2758)
SEM IMAGING (AFFECTED BY SURFACE CHARGING OF PASSIVATION LAYER)
VS. SEM IMAGING OF AREAS Ga⁺-IMPLANTED DURING SIM IMAGING
25 kV HITACHI HFS-2 SEM

Fig. 12
Ga\(^+\) FOCUSED-ION-BEAM IMAGES
ELEMENTAL CONTRAST IN SECONDARY ELECTRON IMAGES FROM ION VS. ELECTRON BEAM SCANS

320 \(\mu\text{m} \) FULL SCALE  \(\text{Au on Fe}\)  320 \(\mu\text{m} \) F.S.

320 \(\mu\text{m} \) F.S.  \(\text{Ag on Fe}\)  320 \(\mu\text{m} \) F.S.

60 keV UC -SIM  10 keV COATES & WELTER SEM

Fig. 13
Ga⁺ focused-ion-beam images

Recrystallized, polished, HNO₃-etched brass

Evolution of secondary electron image under continuous raster ion bombardment

160 µm full scale
Elapsed time: 0

160 µm full scale
Elapsed time: 10 min.

160 µm full scale
Elapsed time: 15 min.

64 µm f.s.
After 200 min. at 160 µm f.s.

Fig. 14
GA⁺ FOCUSED-ION-BEAM IMAGES
RECRYSTALLIZED, POLISHED, HNO₃-ETCHED BRASS
CRYSTALLOGRAPHIC CONTRAST IN SECONDARY ELECTRON SIM IMAGES VS. SURFACE TOPOGRAPHY IN SEM IMAGES

64 µM FULL SCALE

64 µM F.S.
60 keV UC-SIM

15 µM F.S.
10 keV COATES & WELTER SEM
GA+ FOCUSED-ION-BEAM IMAGES
RECRYSTALLIZED, POLISHED, HNO₃-ETCHED COPPER
SECONDARY ELECTRON IMAGES FROM ION VS. ELECTRON BEAM SCANS

180 µm FULL SCALE

180 µm F.S.

180 µm F.S.
60 keV UC-SIM

180 µm F.S.
25 keV HITACHI HFS-2 SEM

Fig. 16
Ga$^+$ FOCUSED-ION-BEAM-IMAGES
RECRYSTALLIZED, POLISHED, HNO$_3$-ETCHED BRASS
CONTRAST REVERSALS DUE TO PRIMARY ION-CHANNELLING
SECONDARY ELECTRON IMAGES, 60 keV UC-SIM

NORMAL INCIDENCE

$10^\circ$ NORTH-SOUTH TILT

Fig. 17
Ga$^+$ FOCUSED-ION-BEAM IMAGES
RECRYSTALLIZED, POLISHED, HNO$_3$-ETCHED COPPER
CONTRAST REVERSALS DUE TO PRIMARY ION-CHANNELLING
SECONDARY ELECTRON IMAGES, 60 keV UC-SIM

NORMAL INCIDENCE

180 μm FULL SCALE

10° NORTH-SOUTH TILT

180 μm F.S.

180 μm F.S.

Fig. 18
Ga⁺ FOCUSED-ION-BEAM IMAGES
RECRYSTALLIZED, POLISHED DIE STEEL
CONTRAST REVERSALS DUE TO PRIMARY ION-CHANNELLING
SECONDARY ELECTRON IMAGES, 60 keV UC-SIM

NORMAL INCIDENCE

10° NORTH-SOUTH TILT

64 μm FULL SCALE

64 μm F.S.

32 μm F.S.

32 μm F.S.

Fig. 19
GA$^+$ FOCUSED-ION-BEAM IMAGES
DETECTION OF SHOCK-INDUCED DAMAGE IN POLISHED METEORITIC IRON CRYSTAL BY ION-CHANNELLING CONTRAST IN SECONDARY ELECTRON IMAGES
60 keV UC-SIM

NORMAL INCIDENCE

18 µm full scale

10° TILT

18 µm F.S.

18 µm F.S.

Fig. 20
GA$^+$ FOCUSED-ION-BEAM IMAGES
RECRYSTALLIZED, POLISHED, HNO$_3$-ETCHED COPPER CONTRAST REVERSALS DUE TO PRIMARY ION-CHANNELLING COMPARISON OF SE vs SI IMAGES, 60 kV UC-SIM

NORMAL INCIDENCE

10° NORTH-SOUTH TILT

360 µm FULL SCALE

360 µm F.S.

360 µm F.S.

Fig. 21
Ga\textsuperscript{+} FOCUSED-ION-BEAM IMAGES

RECRYSTALLIZED, POLISHED, HNO\textsubscript{3}-ETCHED COPPER
CONTRAST REVERSALS DUE TO PRIMARY ION-CHANNELLING
COMPARISON OF SE vs SI IMAGES, 60 kV UC-SIM

NORMAL INCIDENCE

10\textdegree NORTH-SOUTH TILT

SE

360 \textmu m FULL SCALE

360 \textmu m F.S.

SI

360 \textmu m F.S.

360 \textmu m F.S.

Fig. 22
GA⁺ FOCUSED-ION-BEAM IMAGING AND ETCHING
RECRYSTALLIZED, POLISHED, HNO₃-ETCHED COPPER
CORRELATION OF ION-CHANNELING EFFECTS
IN SE, SI EMISSION AND SPUTTERING

180 µM FULL SCALE;
SI IMAGE

180 µM FULL SCALE;
SE IMAGE

17 µM F.S.
TOTAL ION-BEAM WRITING DOSE OF
2.9 x 10¹⁸ IONS/CM², IN:
TOP LINE: SINGLE SCAN
BOTTOM LINE: 100 SCANS

17 µM F.S.
ION-BEAM WRITING DOSES ARE:
LINE 1: 3.1 x 10¹⁷ IONS/CM²
LINE 2: 7.8 x 10¹⁷ IONS/CM²
LINE 3: 1.2 x 10¹⁸ IONS/CM²
LINE 4: 3.1 x 10¹⁸ IONS/CM²

Fig. 23