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P-52

TECHNICAL REPORT

NASA Contract NAS8-36634
 Period: March 1, 1992 - August 31, 1992
 P.I.: R. B. Lal, Professor of Physics
 Alabama A and M University
Normal, AL 35762

1. The flight sample from IML-1 mission was hand carried to EDO/Barnes Engineering Division, Shelton, CT on February 24, 1992. The whole crystal was photographed and a small piece from IFTGS-2 crystal (only crystal grown during IML-1 mission) was cut parallel to a-axis of TGS for X-ray synchrotron analysis. The rest of the crystal will be used for I. R. detector fabrication and electrical property measurements.
2. The whole crystal IF-TGS-2 from IML-1 mission was examined by X-ray diffraction imaging technique at Brookhaven National Laboratory. The major surface of the grown crystal was substantially uniform, not normally seen in earth grown crystals on (010) face. Some faceted edge growth was seen which grew at different rate. The lattice orientation of the crystal was found to be uniform to 1-2 arc second locally, and limited by seed uniformity to 8 arc seconds for the entire crystal. Polystyrene inclusions of all three different sizes (199 μm , 486 μm , and 646 μm) were seen. No prominent growth related screw dislocations were seen in seed or in the new growth. Few surface-treatment related edge dislocations are seen in seed uniformity to 8 arc seconds for the entire crystal. The interface between the seed and new growth is scarcely visible. The lattice orientation of space growth is identical within a fraction of an arc second to that of seed. More detailed studies will be done on this crystal after polishing when the next x-ray beam line will be available sometime in October or November, 1992.
3. The flight crystal IFTGS-2 was then cut in thickness parallel to (010) face about 3 mm from the seed side. This left the bottom portion mostly seed and the top part mostly space grown. The whole slice was then cut unto two pieces perpendicular to (010) face. One piece has been reserved for electrical measurements and other used for I.R. detector fabrication. The results of I.R. detectors fabricated at EDO/Barnes Engineering Division are included in Table I. The D^x (detectivity) of the flight crystal at 100 Hz is equal or better than the flight seed. This is a significant result inspite of the problems encountered during the mission and a comparatively fast growth of TGS on (010) face.

(NASA-CR-191956) [A STUDY OF
 TRIGLYCINE SULFATE (TGS) CRYSTALS
 FROM THE INTERNATIONAL MICROGRAVITY
 LABORATORY MISSION (IML-1)] Monthly
 Technical Report, 1 Mar. - 31 Aug.
 1992 (Alabama A & M Univ.) 52 P

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4. The optical work is progressing satisfactorily. Detailed description of work is given in an enclosure from MetroLaser.
5. A meeting was held in San Diego, CA on August 19, 1992 with Drs. James Trolinger, William R. Wilcox, and Lia Regel to discuss the plan for the particle data analysis.
6. The subcontract with Clarkson University has been modified to include efforts for particle data analysis.
7. Dr. Lal attended and presented the preliminary results of IML-1 mission at a meeting at NASA headquarters on May 7-8, 1992 and at the Investigator working group meeting at ESA-ESRIN, Frascati, Italy on June 15-17, 1992. A copy of the viewgraph presentation at IWG meeting is attached with this report.
8. Due to high humidity in the laboratory the electrical measurements on the flight crystal has not been started. It is expected that we will start these measurements in later part of September 1992 or early October 1992.
9. We have not yet received the IML-1 acceleration data for the mission. This data is required for further analysis of particle data.
10. The graduate student working in the project left this university to persue Ph.D. in electrical engineering somewhere else. So far we have not been able to recruit a new student.

TABLE 1

SPACE GROWN TGS

DETECTOR PERFORMANCE DATA

FREQUENCY (HZ)	FLIGHT CRYSTAL TOP SLICE D _T (1000-K, 1, 1) DETECTOR S/N (1)	FLIGHT CRYSTAL BOTTOM SLICE D _B (1000-K, 1, 1) DETECTOR S/N (2)	BROTHER SLICE OF FLIGHT CRYSTAL D _B (1000-K, 1, 1) DETECTOR S/N (3)	BROTHER SLICE OF FLIGHT CRYSTAL D _T (1000-K, 1, 1) DETECTOR S/N (4)	BROTHER SLICE OF FLIGHT CRYSTAL D _T (1000-K, 1, 1) DETECTOR S/N (5)	BROTHER SLICE OF FLIGHT CRYSTAL D _T (1000-K, 1, 1) DETECTOR S/N (6)
100	3.0 x 10 ⁸	3.5 x 10 ⁸	4.6 x 10 ⁸	2.5 x 10 ⁸	2.4 x 10 ⁸	-
250	2.8 x 10 ⁸	2.6 x 10 ⁸	3.4 x 10 ⁸	1.4 x 10 ⁸	1.9 x 10 ⁸	-
500	2.1 x 10 ⁸	1.9 x 10 ⁸	2.4 x 10 ⁸	9.7 x 10 ⁷	1.7 x 10 ⁸	-
1000	1.5 x 10 ⁸	1.0 x 10 ⁸	1.5 x 10 ⁸	6.9 x 10 ⁷	1.2 x 10 ⁸	-

*NOTE: 0.5mm DIA. VOID NOTED IN FLIGHT CRYSTAL TOP SLICE.

MetroLaser

18006 Skypark Circle, Suite 108, Irvine, CA 92714-6428 Tel: (714) 553-0688 Fax: (714) 553-0495



REVISED

September 3, 1992

Dr. Ravindra B. Lal
Alabama A & M University
Carter Hall, Physics Department
P.O. Box 71
Normal, Al 35762

SUBJ: NASA Prime Contract NAS8-36634
Our Job No. AL02

Dear Dr. Lal:

This is the second report for 1992 on the subject contract.

Summary of work before this period

MetroLaser has supported Alabama A&M University in the development of the hardware, experiment, experiment planning, simulations, and crew training. Details of this support have been included in previous reports and will not be included here. MetroLaser supported the IML-1 mission in January of 1992. The details of the mission support are included in the last quarterly report.

Work performed during this period.

Summary

Preliminary evaluation of the data was made during the hologram processing procedure. A few representative holograms were selected and reconstructed in the HGS; photographs of sample particle images were made to illustrate the resolution of all three particle sizes. Based on these evaluations slight modifications were requested in the hologram processing procedure to optimize the hologram exposure in the vicinity of the crystal. Preliminary looks at the data showed that we are able to see and track all three sizes of particles throughout the chamber. Because of the vast amount of data available in the holograms, it was recommended that we produce a detailed data reduction plan with prioritization on the different types of data which can be extracted from the holograms.

Dr. Ravindra Lal
September 3, 1992

A set of holograms was selected to represent a quiet period during the flight and a rotation of the space shuttle and another level of reconstructed images was made. In this series of tests, approximately 100 transparencies of particle images were made with the help of W. Witherow. With these transparencies, a few velocity tracks were made of particles over a time period of about four hours. These studies showed that we could measure g and observe a g -jitter type of phenomenon. Values of g observed were in the range from fractions of a micro g to milli g . Various other anomalies were also observed. This effort resulted in photographs, video tapes of particles in motion, particle tracks, and preliminary g - data.

We then worked out a more detailed plan to handle the large quantity of data including the help of Clarkson personnel. Conversations were held with Bill Wilcox and Lya September 3, 1992 Regal towards this end.

Primary Science Objective

The primary science objective of the particle experiment is to observe and quantify minute convection currents in the vicinity of the crystal and to correlate these with crystal growth processes. With the limited amount of crystal growth data, we will attempt to observe, isolate, and quantify the following components of fluid convection:

1. Growth driven convection.
2. Convection due to g -jitter.
3. Convection due to other acceleration forces.
4. Convection due to residual microgravity.

All but the first of these components was well represented with or without a crystal growing. The three IML-1 runs have been designated as:

Run 1a-Cell #1-Cap did not open.
Run 1b-Cell #1-Cap opened and closed
Run 2-Cell J#2-No data.

Run 1a offers the opportunity to observe particle motion without the influence of the crystal.

We will attempt to correlate the presence of convection with other events. Namely, we should be able to more accurately characterize the space shuttle environment, how energy is coupled to an experiment, and its effect on crystal growth.

The primary science objective of the holographic interferometry experiments is to provide a measure of the concentration and concentration gradients in the vicinity of the crystal to support modeling and to help explain the properties of the resulting crystal.

Secondary Science Objectives.

The dynamics of three ensembles of monodisperse particles in suspension will be observed in microgravity through use of the acquired data. This will provide important basic scientific information about g-jitter, inertial random walk, two phase flow, particle dynamics, and the residual gravitational field in the Spacelab. We will be making accurate measurements of convection in a regime never measured before. This will allow the validation of both theory and CFD codes.

Testing the theory of inertial random walk. Inertial random walk, a novel type of diffusion, has been predicted to exist in microgravity, but has not been quantitatively observed in space. It may have a significant effect on certain types of material processing in space. It has been simulated by L. Regal in ground experiments (in one dimension), but actual space data is required to truly test the theory. Our particle experiment is almost ideal for testing the theory of inertial random walk. *Preliminary observations appear to confirm that we have observed inertial random walk and that we have sufficient data to make important conclusions about this phenomenon. This data can be extremely important to the scientific microgravity community.*

Testing Two Phase Flow and Particle Dynamics Theory. Two phase flow has been studied very little in microgravity. The general equations of two phase flow are surprisingly untested because of the difficulty of testing many regimes. The microgravity environment will allow us to examine two phase flow in an extremely low Reynolds number regime where convection would normally dominate the results. The use of multiple particle sizes will allow us to test a variety of two phase flow effects.

Material processing which involves free floating particles depends heavily on particle dynamics and interaction. The statistics of particle diffusion and collision rates have been developed but have not been tested in microgravity. Although our selected particle number density is somewhat low, the study of particle dynamics and interaction should be enhanced by the tracking of all particles in 3-D in microgravity.

Residual Gravitational Field in Spacelab. The equations of motion of the particles leads us to the conclusion that we will be able to accurately measure the residual gravitational field on the Spacelab by tracking these suspended particles. The selection of the size and density range has increased the accuracy of the measurement. We should also be able to measure relatively low frequency changes in g. High frequency changes in g will be filtered out by our measurement. The measurement will be correlated with other recorded events in IML-1 such as the various rolls made during the mission. This should provide a significantly improved characterization of the Spacelab environment.

Observations Made as of August 1992:

1.0 The particle motion

1.1 Equations of Motion

The particle velocity relative to the fluid motion is described by the following equation of motion.

$$v_f - v_p = Ka^2 \left[\frac{\rho_p}{\rho_f} - 1 \right] \left[\frac{dv_p}{dt} - g \right]$$

where ,

$$K = \frac{2\rho_f}{9\mu}$$

and μ is the viscosity of the fluid, ρ_f is the fluid density, ρ_p is the particle density, a is particle radius, and g is the acceleration of gravity. The assumptions are that the Reynold's number is much less than unity, that the particle is not spinning, and that the particle is smooth.

The solution of this equation is

$$v_p = v_0 \exp\left[-\frac{t}{F}\right] + Fg + v_f$$

Where,

$$F = Ka^2 \left[\frac{\rho_p}{\rho_f} - 1 \right]$$

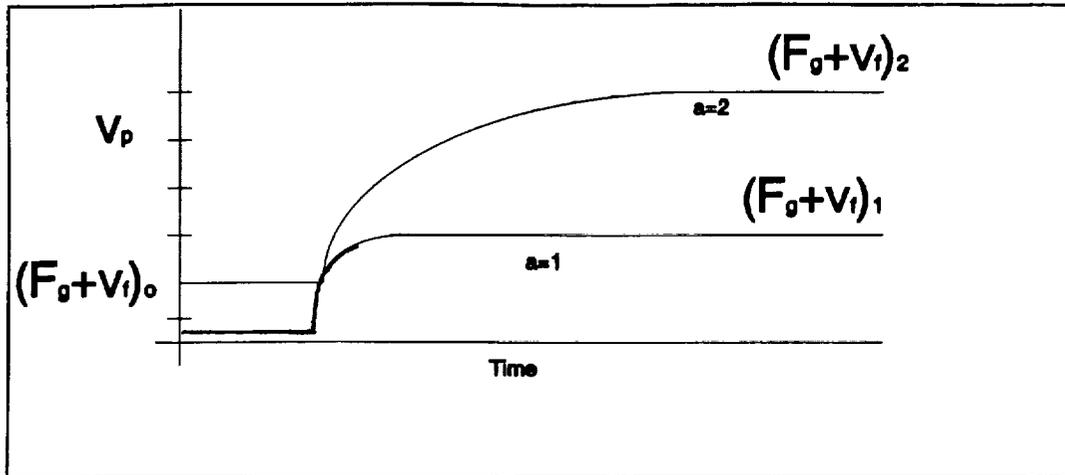
When a step change in fluid velocity takes place, the particles approach the new velocity exponentially with time. The $\exp(-2)$ time constant in this relaxation is

$$t^* = 2F = 2Ka^2 \left[\frac{\rho_p}{\rho_f} - 1 \right]$$

When a steady state is reached, the fluid minus particle velocity is a constant equal to $Fg + v_f$. When a new value of $Fg + v_f$ is experienced by a step change $\delta(Fg + v_f)$ the equation for v_p can be written as

$$v_p = -\delta[Fg + v_f] \exp\left[-\frac{t}{F}\right] + Fg + v_f$$

The following sketch shows how two particles of different size will experience such a change in time. Note that the equilibrium velocity of a particle is proportional to the square of the particle diameter. However, the particle requires a time to reach its equilibrium which is also proportional to the square of the diameter.



Sketch 1-Transient of particles moving in a fluid

1.2 Separating the Effects of various forces on the Particles.

1.2.1 Convection. Our computations show that the particles will attain the fluid velocity in most cases in a fraction of a second. Therefore one method to separate the effects of a steady convection from acceleration and other transient effects is to observe the relative motion of the large and small particles. The velocity of the large and small particles in close proximity will be approximately the same since they are equal to convective velocity of the fluid.

Also, when two particles can be observed moving in different directions, this can be ascribed to convection, since gravity will act on all particles in the same direction.

1.2.2 Gravitational and other acceleration effects. A constant acceleration will result in a terminal particle velocity which is proportional to the diameter of the particle squared. According to Regal, g-jitter will also result in a drift velocity proportional to the square of the particle diameter. G-jitter will result in a random directional change in the particle motion. G-jitter will be the easiest to distinguish when the solution density is uniform so as to remove the effects of random motion driven by convection.

1.3 Observed Particle motion caused by convection.

Convection was observed during the cap removal and during cap replacement, both through the schlieren image of the moving density gradients and through movement of particles. The number density of particles appeared quite large as though few particles had been lost from the original sample.

Vorticity has been observed as manifested in the appearance of particles near each other traveling in opposite directions.

1.4 Caused by Shuttle Roll.

Particles were observed clearly moving during the rolls. The growth and dissolution cloud, however, did not appreciably move. Our holographic data recorded during rolls distinctly shows significant particle motion.. The problem with the data here is that the holograms were recorded at too large a time separation to allow us to track particles. In a typical roll, we recorded only two holograms and the particles recorded in each are uncorrelated, since they have moved so much. We have not decided at this point how to best make use of this data.

1.5 Caused by crystal growth.

The growth and dissolution clouds were clearly observable in the schlieren images. Resolution in the TV downlink combined with the inability to sufficiently monitor prevented observation of minute particle motion expected from crystal growth. We hope to see this in the holographic data.

The particles should move toward the crystal during growing (if influenced only by growth convection) and should move away from the dissolving crystal.

Preliminary holographic interferograms produced during the flight confirm that little convection existed around the crystal. Large density gradients exist near the cap. We do not see the large chunks of crystalline material that seemed appear in the downlink camcorder shots.

2. Acceleration of Gravity

2.1 During Normal Conditions

Limited monitoring and TV downlink resolution prevented measurement of motion in real time. The main source of useful data should be the holograms recorded during the mission.

We have made some preliminary measurements to determine the g level required to result in the observable particle motion. Figure 1 illustrates some of the preliminary particle track data for three different particle sizes over approximately a four hour period. The Figure clearly illustrates a random type of motion. Each vector represents the motion during eleven minutes. The crystal and sting is shown in the bottom left hand corner. The particles usually move at a velocity less than one diameter per hour, although in this Figure near the end of the particle track, a correlated acceleration of about three particle diameters took place for all three particles in eleven minutes. From the above equations, the following value of g results for a given particle velocity:

$$g = \frac{9}{2 a^2} \frac{\mu v_p}{[\rho_p - \rho_f]}$$

The following values are useful for sample calculations in the present case.

Viscosity of TGS solution-1.58 Centipoise

Density of polystyrene latex spheres- $1.05 \times 10^3 \text{ kg/m}^3$

Density of the TGS solution- $1.145 \times 10^3 \text{ kg/m}^3$

To convert centipoise to Newton sec/m² multiply by 10^{-3} .

To convert kilogram force to Newtons multiply by 9.806

Therefore,

$$g = 7.48 \times 10^{-5} \frac{v_p}{a^2}$$

Three particle diameters were used

1. 199 micrometer
2. 383 micrometers
3. 646 micrometers.

383 micrometer particle moving one diameter in one hour, corresponds to a velocity of approximately 1.06×10^{-7} meters/second as a typical motion. If convection is negligible, this leads to an acceleration of approximately

$$g = 5.4 \times 10^{-5} \text{ m/sec}^2$$

or about $5.5 \mu\text{g}$.

2.2. During Shuttle Roll .

Measurements may be made using both the recorded TV and holography since the motion is quite large. Insufficient monitoring time during the mission made this impractical in real time. Here is a condition in which gravity is turned on then off. We should see the particles accelerate, then decelerate. As mentioned above we do not have enough data to monitor the motion during a roll. Therefore, we should monitor the motion after a roll to watch the particles decelerate. This should provide useful data about the solution, since all of the particles should have reached an equal velocity by the end of the roll. Their deceleration may lead to a measurement of the viscosity of the solution.

2.3. Caused by Other effects.

All of the other forces on the particles are expected to be extremely small. Measurement of these effects will require extremely accurate tracking of the particles, especially if small effects are to be subtracted from the larger motion effects. The data collecting time during the mission was not really as long as we had hoped, so the measurements must be made more

accurately than we had planned, and use of statistics may be necessary. The large number of particles in the field should provide excellent statistics.

3. The Concentration Variation

3.1 Immediately after cap opening.

Immediately after cap opening, large density gradients could be observed in the vicinity of the crystal. Since the knife edge was in the wrong orientation, we had difficulty in determining whether the crystal was growing or dissolving. At first by making some assumptions, we concluded that it was growing. When we finally got a crew member to correctly orient the knife edge, we immediately identified that the crystal was dissolving. A dark cloud surrounded the crystal.

3.2 During Shuttle Roll .

The concentration gradients moved surprisingly little during roll. This suggests that the solution density varied by only a small amount. We should examine the interferograms during this time to confirm this.

3.3 During crystal dissolution.

The expected dissolution cloud was observed. This cloud persisted for hours with little motion.

3.4 During crystal growth.

The classic change was observed as the dissolution cloud lifted off of the seed. This concentration gradient remained in the region above the crystal for hours, while the growth cloud of reduced concentration began to grow inside of the higher concentration dissolution cloud. This will be extremely interesting to see in the interferograms. This large concentration of the solution above the crystal as caused by the dissolution may account for the rapid crystal growth observed.

3.5 Caused by Other effects.

Diffusion effects could be observed. The edge of the cloud moved slowly outward. g levels did not disturb the shape of the cloud. This may be useful in measuring diffusion coefficients.

Crystals away from the seed created concentration gradients. However, there were surprisingly few of these visible in the schlieren image, especially surprising since we had seen so much undissolved material in the camcorder downlink.

4.0 Data Reduction/ Interpretation Plan

All holograms have now been processed at MSFC in specially designed development tanks that measure the plate density during development. This allows all plates to be developed at optimum density (Intensity transmission of 0.16).

A meeting was held with Bill Witherow on 16 July 1992 to discuss the IML-1 data reduction and to further develop a data reduction plan. During this meeting, Witherow and I agreed upon a plan to recommend.

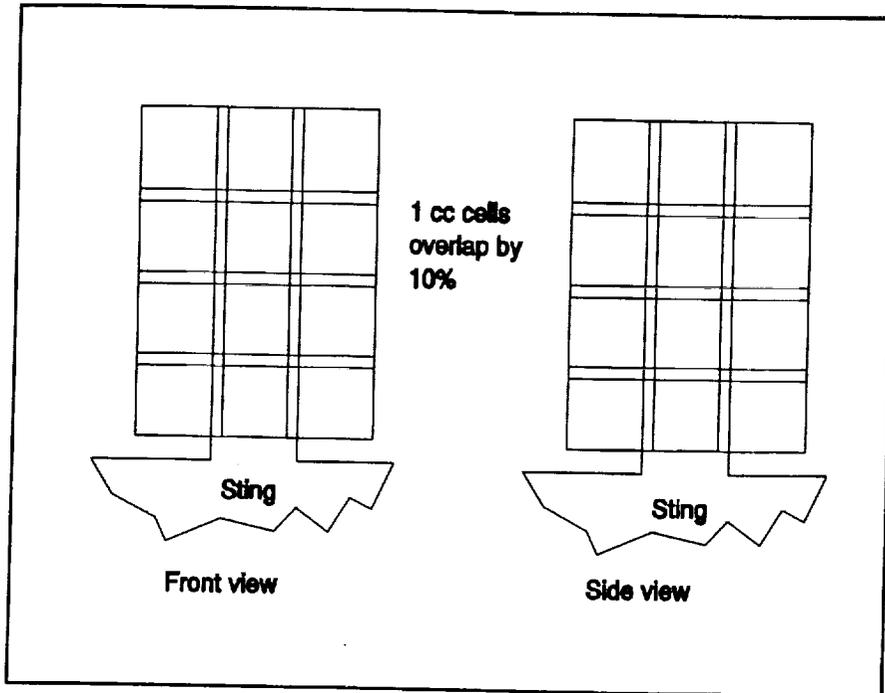
On the following day, the plan was discussed in a second meeting attended by Rudy Ruff, David McIntosh, Jim Trolinger, Bill Witherow, Donnie McCaghren, and Ric Cummings. The meeting was called by David McIntosh for the purpose of reviewing his status, to propose a course of action, and to get direction.

A number of considerations should be examined in determining the data handling:

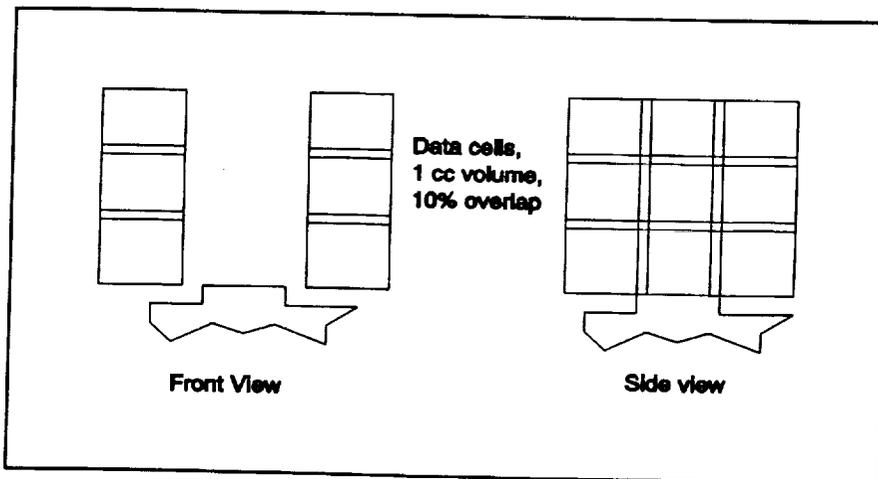
1. The particle data appears to be extremely important and may be useful to a wide audience in the future. Even now it would be extremely convenient to make the data available in Irvine, CA (MetroLaser) in Potsdam, NY (Clarkson), and in Normal, AL (A&M).
2. There will be a large quantity of data. To begin with, we estimate the need to analyze a minimum of hundreds of images.
3. The images analyzed so far were digitized and then printed. These appear totally adequate for our needs. The process is quick and convenient.
4. David McIntosh expressed an interest in being involved with the data reduction process all the way to an end result; not just in providing raw data.

The following course of action was proposed and tentatively agreed upon for the data:

A selected set of images should be digitized. Sketches 2 and 3 show the sets of images relative to the crystal. The idea is to track particle motion near the crystal surface to achieve the primary science objective and to track particles far from the surface to achieve secondary objectives.



Sketch 2-Regions desired for data collection for Run 1b



Sketch 3-Regions desired for data collection for Run 1a

From previous results, we concluded that if the particle images are reconstructed at unity magnification directly onto a lenseless CCD or Vidicon, the magnification is about correct for resolving enough particles with sufficient magnification for focusing and tracking. This makes the images approximately 1 cm (plus or minus 20%, depending upon the Vidicon or CCD array used) in diameter. This allows a particle to travel many diameters in the same frame,

removes any of the aberrations that may be added by other lenses in to the optical train and simplifies traversing. Therefore, we recommend this procedure for reconstruction of all particle images. The 1 cm. dimensions in the sketches are nominal only and may actually vary from one setup to the other. In any event, the designated number of frame widths of any conventional TV system should provide ample coverage. The data regions should overlap by about 10 % so that each image will have particles in it that match an adjacent image. The camera will be focused to the center of each of the data volumes.

The following table summarizes the data to be extracted from the holograms.

Run no.	No. of holograms	Images/hologram	No. of Images
1a	43	18	768
1b	62	36	2232
1b	62	1 interferogram	62

The images of particle fields and interferograms will be digitized in the HGS and will be transferred over the NASA ethernet or internet system to the SSL VAX and will be stored on the SSL worm drive. The VAX will be addressable by designated groups who will be able to access the digitized images through the use of internet. It is assumed that MetroLaser and Clarkson will be able to access the SSL VAX and transport the images into their own image processors. To have access, a group must have access to internet and File Transfer Protocol (FTP) capability. We have not yet recommended a specific image processor or processing algorithm. Each group can work with existing frame grabbers and image processors. This will ultimately make the images accessible to any group in the world who is deemed qualified to receive the data. After the data becomes available to the various groups, specific data reduction tasks will be provided to each.

We must produce a suitable header which contains all required information to identify the data. The header will be transferred with the image file as an ASCII file. In the present state, the HGS can store up to about four (4) images per floppy disk. With the use of Bernoulli drives this can be expanded by about a factor of 50.

The following summarizes the desired measurements in order of priority:

1. Concentration of Solution near the crystal
 - a. During dissolution
 - b. During growth
 - c. During unusual shuttle motion

2. Convection

- a. Growth driven.
- b. G-jitter driven.
- c. Due to other acceleration forces.
- d. Due to residual microgravity.

The importance of a complete experimental analysis of convection may be reduced because of the way the experiment turned out. This data may be extremely difficult to interpret because of the poor environment of the seed.

3. Acceleration

- a. Due to residual microgravity.
- b. G-jitter driven.
- c. Due to other acceleration forces.
- d. Growth driven.

4. Particle Mechanics

- a. Inertial random walk.
- b. Two Phase Flow.
- c. Particle interactions

Measurement of the above quantities will be achieved through the production and analysis of interferograms, photographs, and video images. Most of the phenomena to be produced with the particle data comes from the same type of measurement made at a different time or location, namely, a particle position-time track. The following measurements are required:

Measurement	Type	Location/Time
1. Concentration	Hol. Int. Tomography	Near xtal/All
2. Convection		
Growth driven	Part track small particles	Near xtal
g-jitter driven	Part track small particles	Away from xtal
residual g	Part track small particles	Away from xtal
other	Part track small particles	Specific/roll
3. Acceleration		
residual g	Particle track large particles	Away from xtal
g-jitter	Particle track Large particles	Away from xtal
other	Large particles	Specific/roll
4. Particle Mechanics		
Inertial random walk	Particle track large & small part	Away from xtal Away from xtal
Two Phase Flow.	Particle track Large & small part	Everywhere/roll
Particle interactions	Large & small part	Anywhere

Data taken during the rolls will provide:

1. A forcing function for low Reynold's number two phased flow.
2. A reference acceleration

The number of holograms to be reduced depends upon the quantity being measured. During rolls, all holograms will require reducing. Early in the crystal growth, many of the interferograms will be required. Later when the growth rate stabilizes, the number of interferograms required will be much less. Except for the study of g-jitter and inertial random walk, only a limited number of particle holograms will be required to be reduced, since little change occurs except during rolls.

5.0 Action Items

A number of action items are open as a result of this meeting.

Who	Action	When
Wetherow	Get DECnet running Renew VAX account Establish worm drive operation Establish image and header S/W	
McIntosh	Write header Reestablish DECnet tie-in Estimate required digitizing time Begin digitizing images to disk	
Trolinger	Write up latest data reduction plan Establish FTP capability Provide info to Clarkson Make inputs to header info	
Batra	Produce complete flight data matrix	

6.0 Assistance requested from Clarkson University

Lya Regal has considerable experience with inertial random walk. Her advice and input to our approach and to our data interpretation could be extremely valuable. I recommend that we solicit her help and advice. Also, Bill Wilcox and staff can provide useful modeling and analytical assistance in addition to assistance with data interpretation.

As a minimum the following tasks could prove extremely useful:

1. Review the equations of motion, assumptions, and preliminary computations to validate our analytical approach.
2. Examine and quantify all of our assumptions and generalize our equations where necessary.

Quantify the following forces and influences that we have neglected so far:

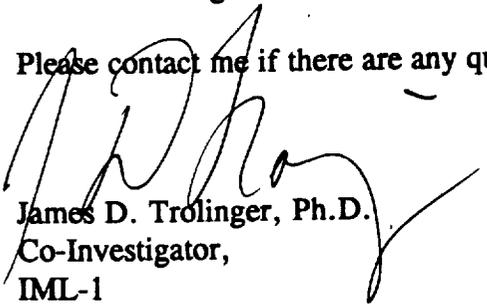
- a. Brownian motion
- b. Electrical forces
- c. Particle rotation effects
- d. Other

3. Produce an inertial random walk equation set for our geometry.
4. Provide image analysis assistance, producing particle track data for images supplied through the internet.
5. Assist in data interpretation and comparison to theory.
6. Make other suggestions as to ways to interpret and make use of the data.

The following are a sample of technical observations and concepts which I feel could benefit from discussions with various scientists.

1. In some cases we are seeing large and small particles moving with nearly the same velocity. Our current interpretation is that this identifies a convection dominated condition.
2. In other cases we observe particles moving in opposite directions. We interpret this as a sign of vorticity.
3. In several cases particles reverse directions inside of an eleven minute period. We would interpret this as random inertial walk if large particles moved significantly larger distances than smaller. However, this is not the case. What then could create such dynamics?
4. What else besides g-jitter can cause particles to move in the observed zig-zag type of motion?
5. We have an eleven minute time resolution so we cannot resolve short term random inertial walk. How will this limit our ability to evaluate random inertial walk in general?
6. Particles have a relaxation time which is proportional to their diameter squared. A typical relaxation time computes to 20 minutes for larger particles and two minutes for smaller particles. Can this account for our observation that smaller particles seem to be traveling too fast relative to larger particles?

Please contact me if there are any questions.



James D. Trolinger, Ph.D.
Co-Investigator,
IML-1

A Study of Solution Crystal Growth in Low-g
[An experiment on the First International Microgravity Laboratory (IML-1)]

Ravindra B. Lal and Ashok K. Batra
Department of Physics
Alabama A&M University

William R. Wilcox
Clarkson University

James D. Trolinger
MetroLaser

IML-1 Investigators Working Group (IWG) Meeting
June 15-17, 1992
ESA - ESRIN
Frescati, Italy

ACKNOWLEDGEMENTS

- **National Aeronautics and Space Administration Office of Microgravity Science and Applications**
- **STS-42 Payload crew**
- **Alternate Payload Specialist**
- **Mission Managers Cadre**
- **Mission Scientist's team**
- **Many NASA/MSFC and Teledyne Brown Engineering personnel**

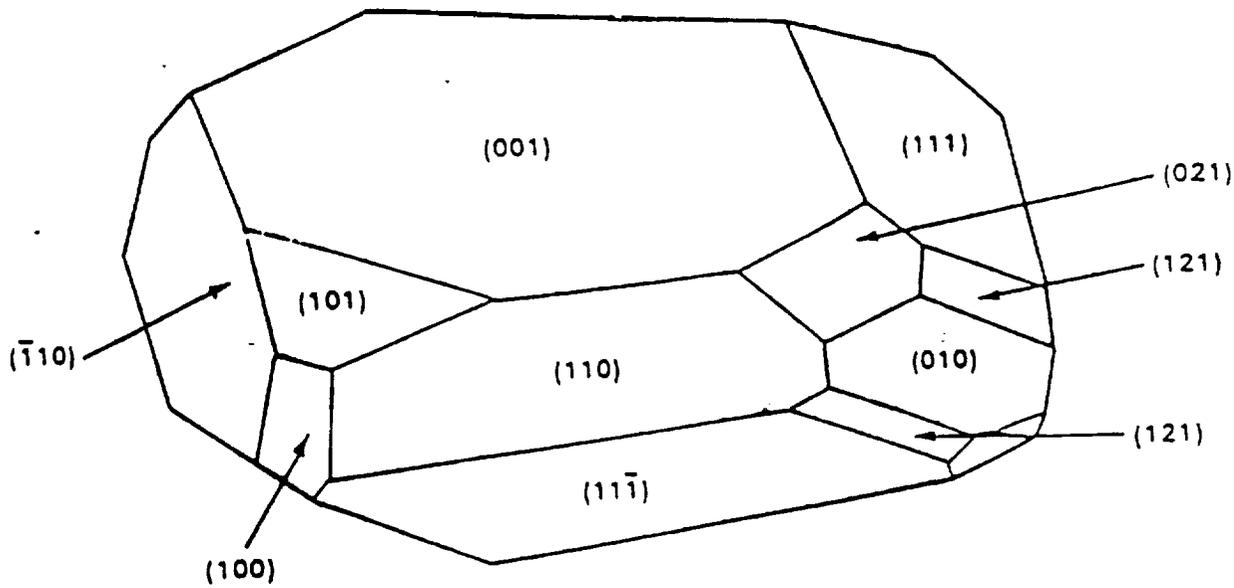
Objectives

To grow crystals of triglycine sulfate (TGS) using modified Fluid Experiment System(FES)

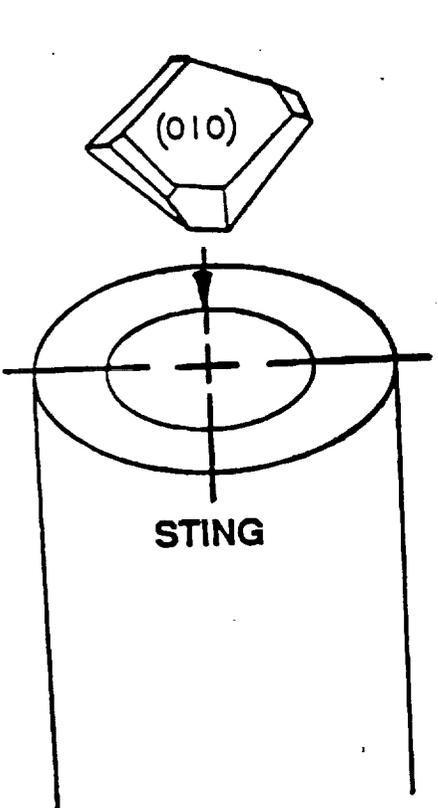
To perform holographic interferometric tomography of the fluid field in three dimensions

To study the fluid motion due to g-jitter by multiple exposure holography of tracer particles

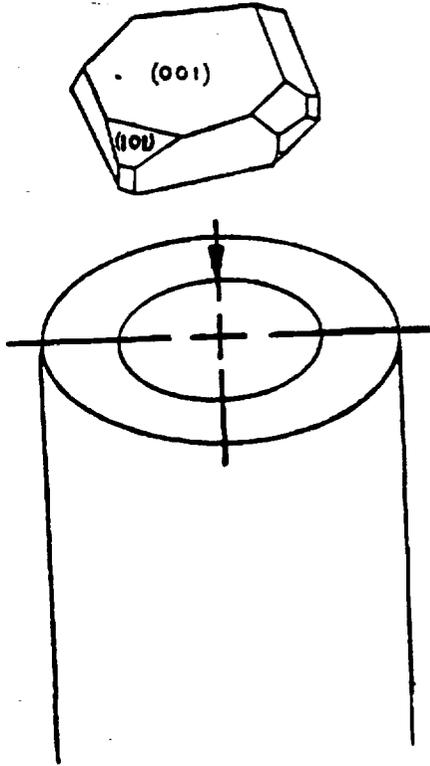
To study the influence of g-jitter on the growth rate



CRYSTALLOGRAPHIC FACES OF TGS CRYSTAL



TGS-1



TGS-2

TGS GROWTH RUNS ON IMI.-1 MISSION

Summary of experiment planned for IML - 1 mission

1. TGS -1 Run (Isothermal) the sting and fluid temperature same and below the fluid saturation temperature.

$$T(\text{sting}) = T(\text{fluid}) < T(\text{sat.})$$

(010) Oriented polyhedral seed (1 cm)

Study of fluid motion due to crystal growth and g-jitter by multiple exposure holography of tracer particles in TGS sloution.

Holographic Optical Elements (HOE) for 3 - D view.

2. TGS - 2 Run (Polynomial) - the sting temperature follows a predetermined third order polynomial

(001) Oriented polyhedral seed (1.5 cm)

Growth of TGS crystal

HOE for 3 - D view of crystal and fluid field

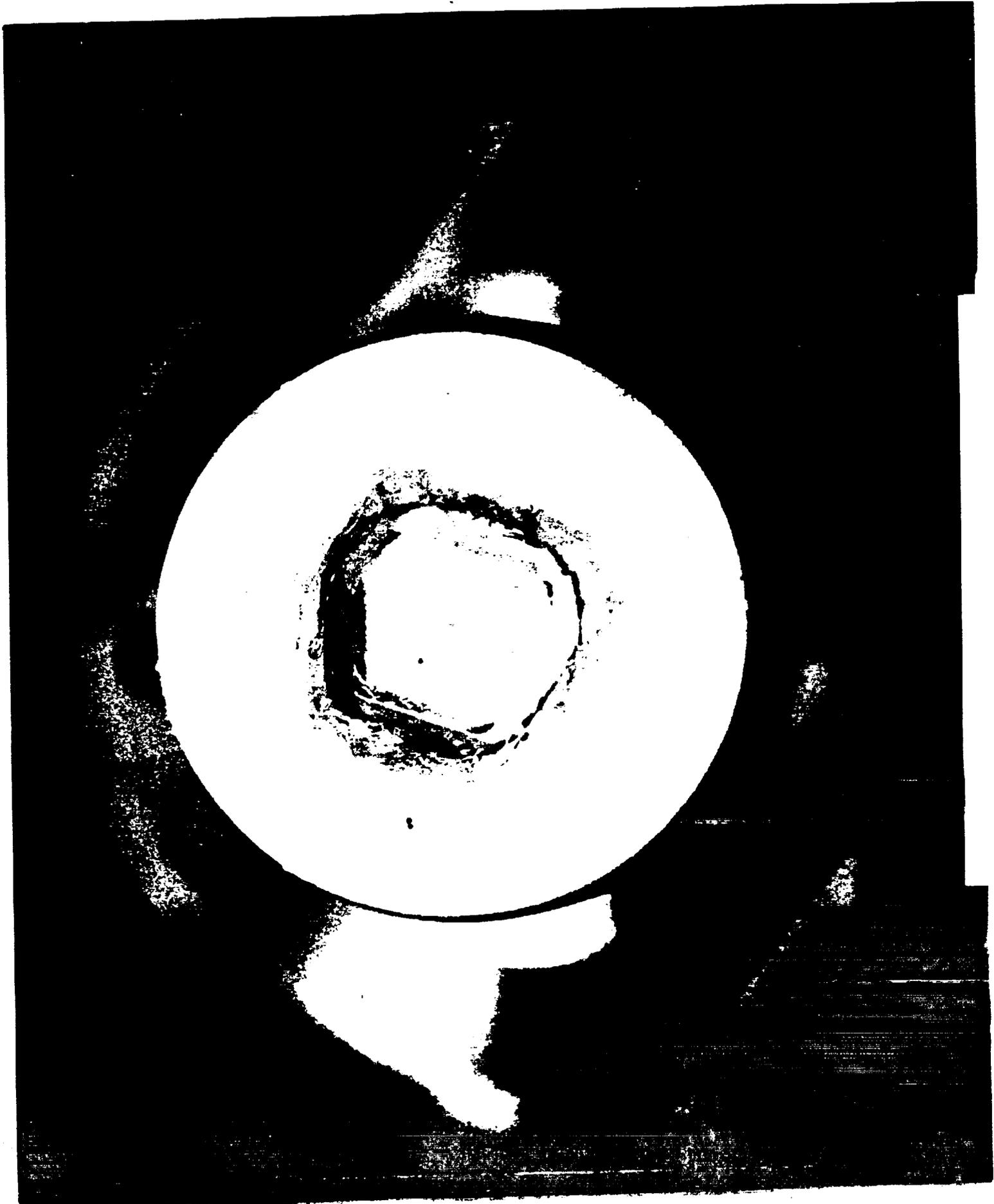
Fluid Experiment System (FES) Performance During IML - 1 Mission (continued)

- * **Extra day of mission great help.**
- * **Stirrer in TGS - 1 run also did not work**
- * **Experiment was initiated without attaining predetermined temperature profile.**
- * **Obtained particle motion data**
- * **Partial objectives of first experiment run were met.**
- * **Objectives of second experiment run (crystal growth) were not met**
- * **TGS - 1 experiment performed with cap closed**

Progress Of TGS-1 Crystal Characterization

- . No apparent increase in total thickness of the seed after growth.
- . Total thickness of the grown crystal 3.50 mm.
- . Particles of $600\ \mu$ trapped inside new growth.
- . Minimum new growth $600\ \mu$.
- . Crystal has been cut for X-ray diffraction imaging, detector fabrication and electrical measurements.
- . Grown crystal cut parallel to (010) face in to two parts.
- . Cut samples have been electroded for detector fabrication (top new growth and bottom seed).
- . Two detector cells made on each piece.
- . Data for D^* (Detectivity) indicates higher values compared to seed.
- . No visible interface between seed and new growth.

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TGS-1 Flight Seed (010 Orientation)

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TGS-1 Crystal after IML-1 Flight (010 Orientation)

TABLE 1

**SPACE GROWN TGS
DETECTOR PERFORMANCE DATA**

FREQUENCY (HZ)	FLIGHT CRYSTAL TOP SLICE D* (1000-K, 1, 1) DETECTOR S/N (1)	FLIGHT CRYSTAL BOTTOM SLICE D* (1000-K, 1, 1) DETECTOR S/N (2)	FLIGHT CRYSTAL TOP SLICE D* (1000-K, 1, 1) DETECTOR S/N (3)	FLIGHT CRYSTAL BOTTOM SLICE D* (1000-K, 1, 1) DETECTOR S/N (4)	BROTHER SLICE OF FLIGHT CRYSTAL D* (1000-K, 1, 1) DETECTOR S/N (5)	BROTHER SLICE OF FLIGHT CRYSTAL D* (1000-K, 1, 1) DETECTOR S/N (6)
100	3.0×10^8	3.5×10^8	4.6×10^8	2.5×10^8	2.4×10^8	-
250	2.8×10^8	2.6×10^8	3.4×10^8	1.4×10^8	1.9×10^8	-
500	2.1×10^8	1.9×10^8	2.4×10^8	9.7×10^7	1.7×10^8	-
1000	1.5×10^8	1.0×10^8	1.5×10^8	5.9×10^7	1.2×10^8	-

*NOTE: 0.5mm DIA. VOID NOTED IN FLIGHT CRYSTAL TOP SLICE.

OBJECTIVES OF HIGH RESOLUTION SYNCHROTRON X-RADIATION DIFFRACTION IMAGING

GENERAL:

- * Lattice regularity
- * Identification of inclusions and dislocations
- * Growth mode and stability
- * Interface location and characterization

SPECIFIC TO FLIGHT PROGRAM

- * Characterization of potential seed crystals as a basis for selection
- * Characterization of space growth
- * Location of seed / space growth interface
- * Comparison of space grown crystals and control ground crystals

**RESULT OF INITIAL EXAMINATION OF X-RAY DIFFRACTION IMAGES OF
TGS CRYSTAL GROWN ON IML -1**

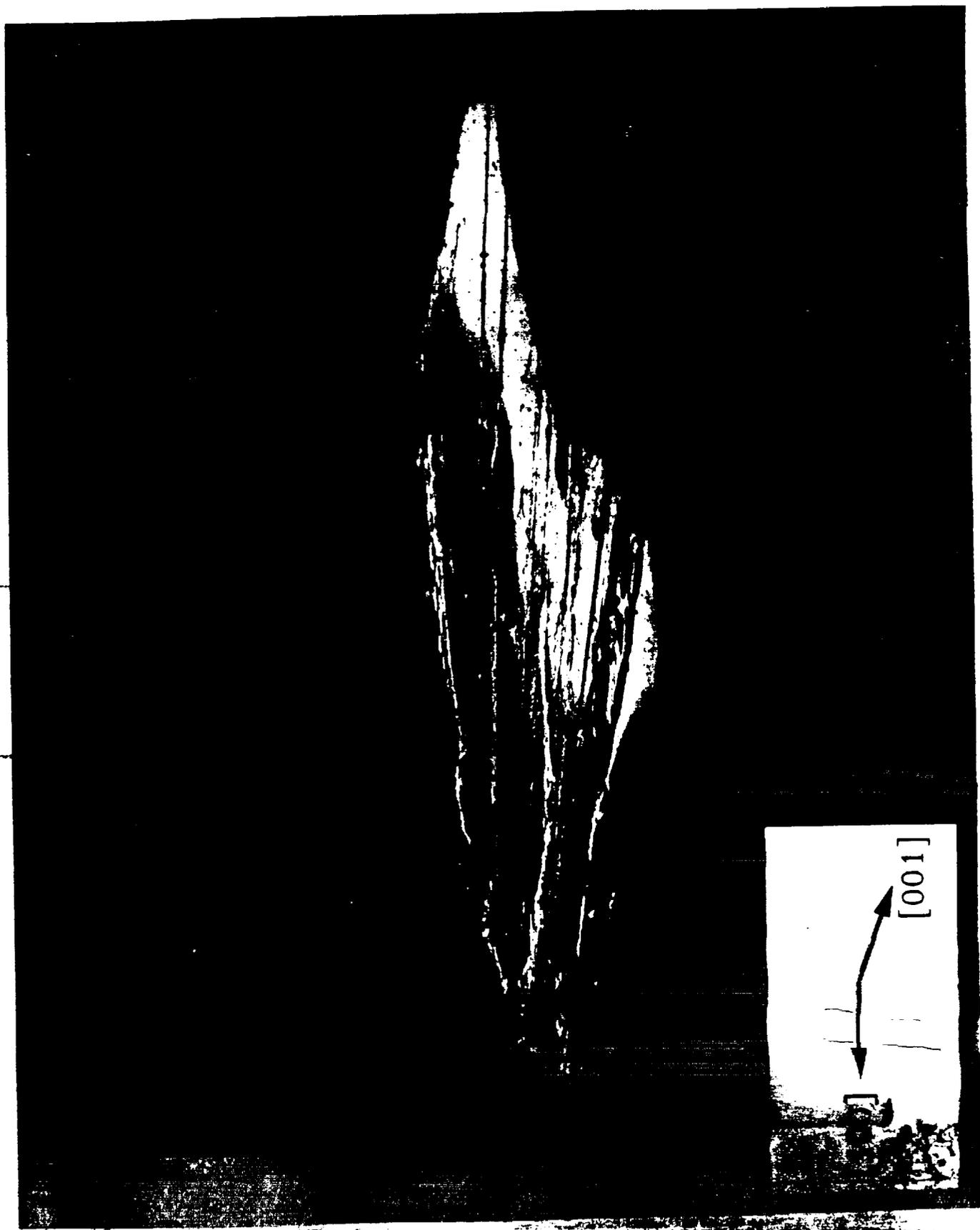
- * **Surface Morphology**
 - Major surface substantially uniform
 - faceted edge growth at different rates

- * **Internal Character**
 - Lattice orientation is uniform to 1 - 2 arc seconds locally , and limited by the seed uniformity to 8 arc seconds for the entire crystal *
 - Polystyrene inclusions of three sizes
 - Few crystallographic defects:
 - o No prominent growth related screw dislocations in seed or space growth
 - o Few surface-treatment related edge dislocations in seed ; none identifiable with new growth

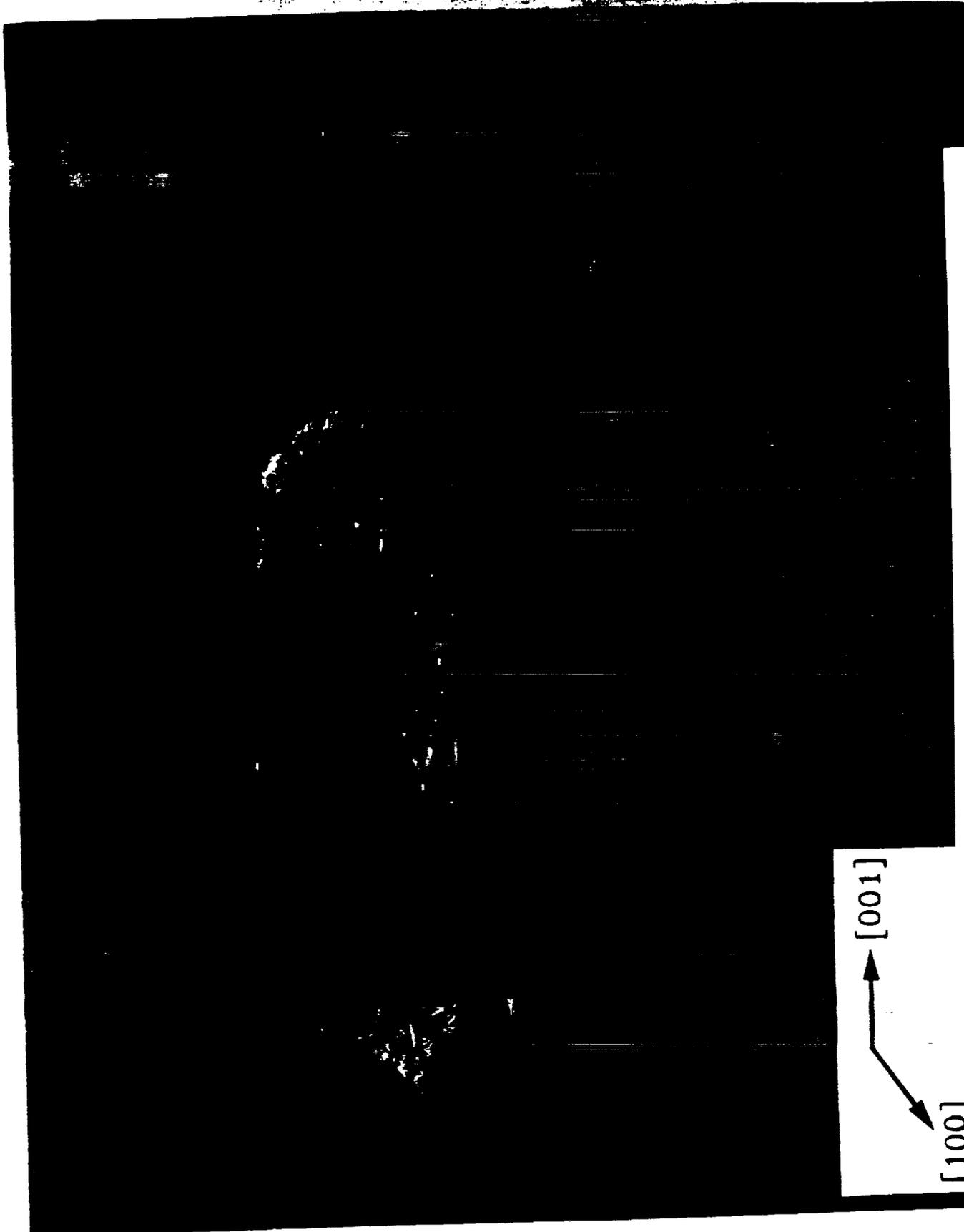
- * **Interface Character and Location**
 - Interface scarcely visible: crystallographically continuous; lattice orientation of space growth identical within a fraction of an arc second to that of seed
 - Position tentatively determined ; precise determination will require polish of cut edge surface

- * **Crystal Cutting Techniques Are Critical to Satisfactory Seed Preparation**

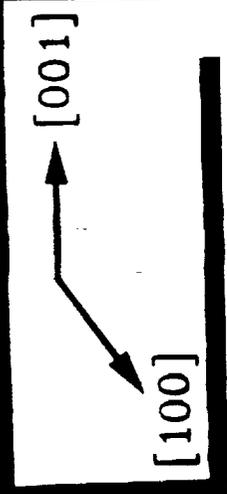
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Cleaved Surface of Control Crystal F TGS-2 (Run 1)



Polished Surface of Brother Crystal 1F TGS-2 (Run 1)

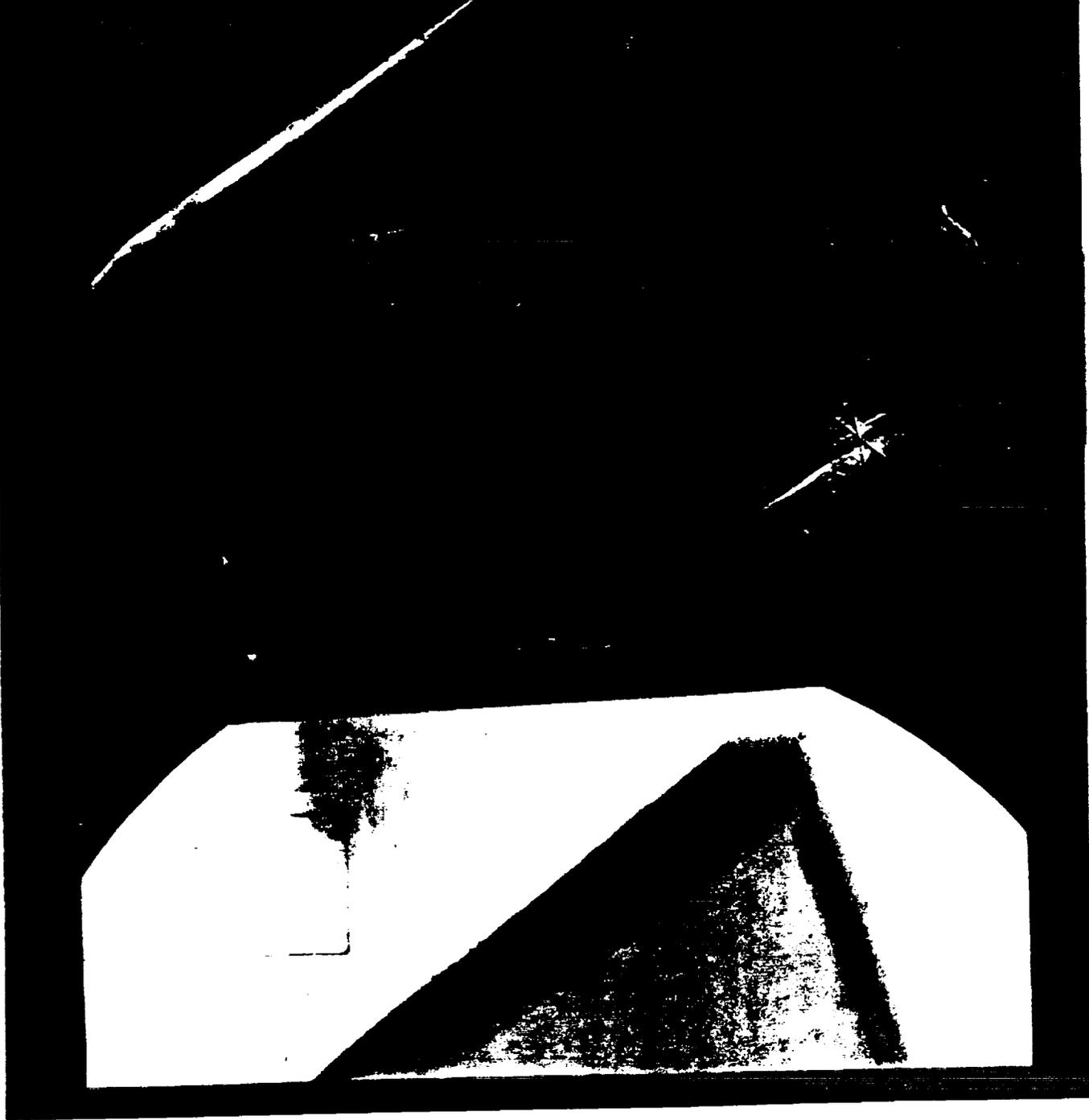
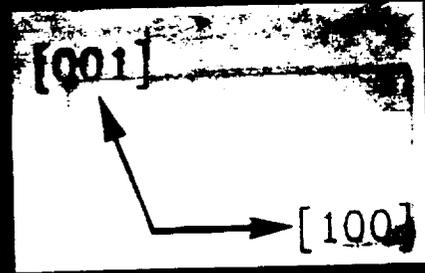


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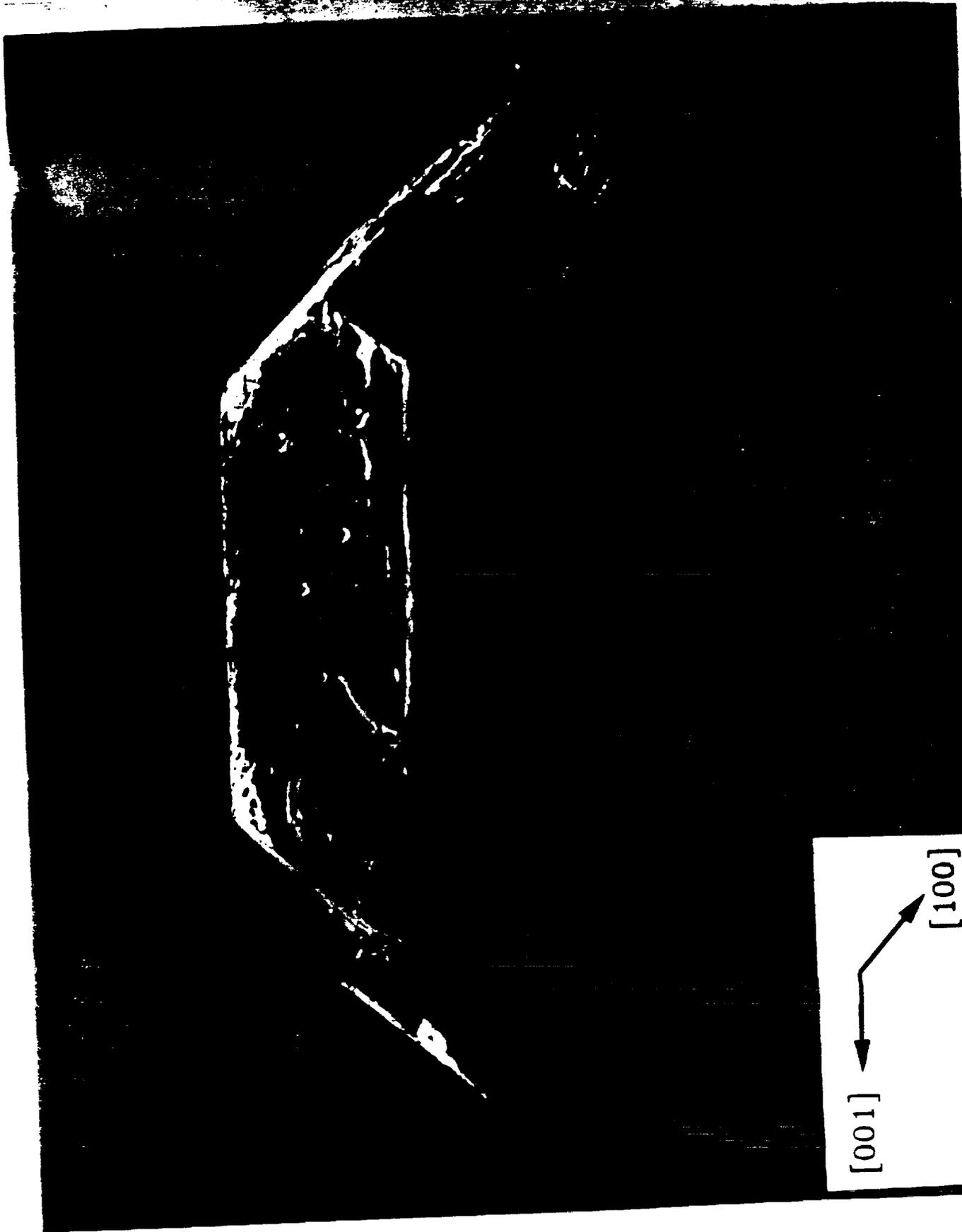
Interior of Cleavage Damage and Growth Related Defects in Control

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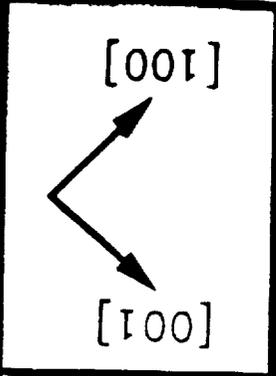
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Interior of Brother Crystal 1F TGS-2 (Run 1)



Growth Surface of IML-1 Crystal 1F TGS-2 (Run 1)

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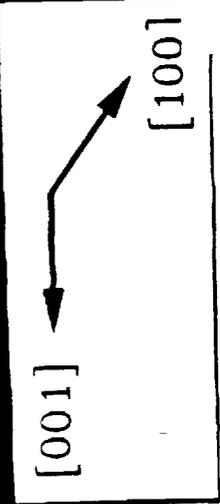


Interior of IML-1 Crystal 1F TGS-2: Polystyrene (Rin 1)

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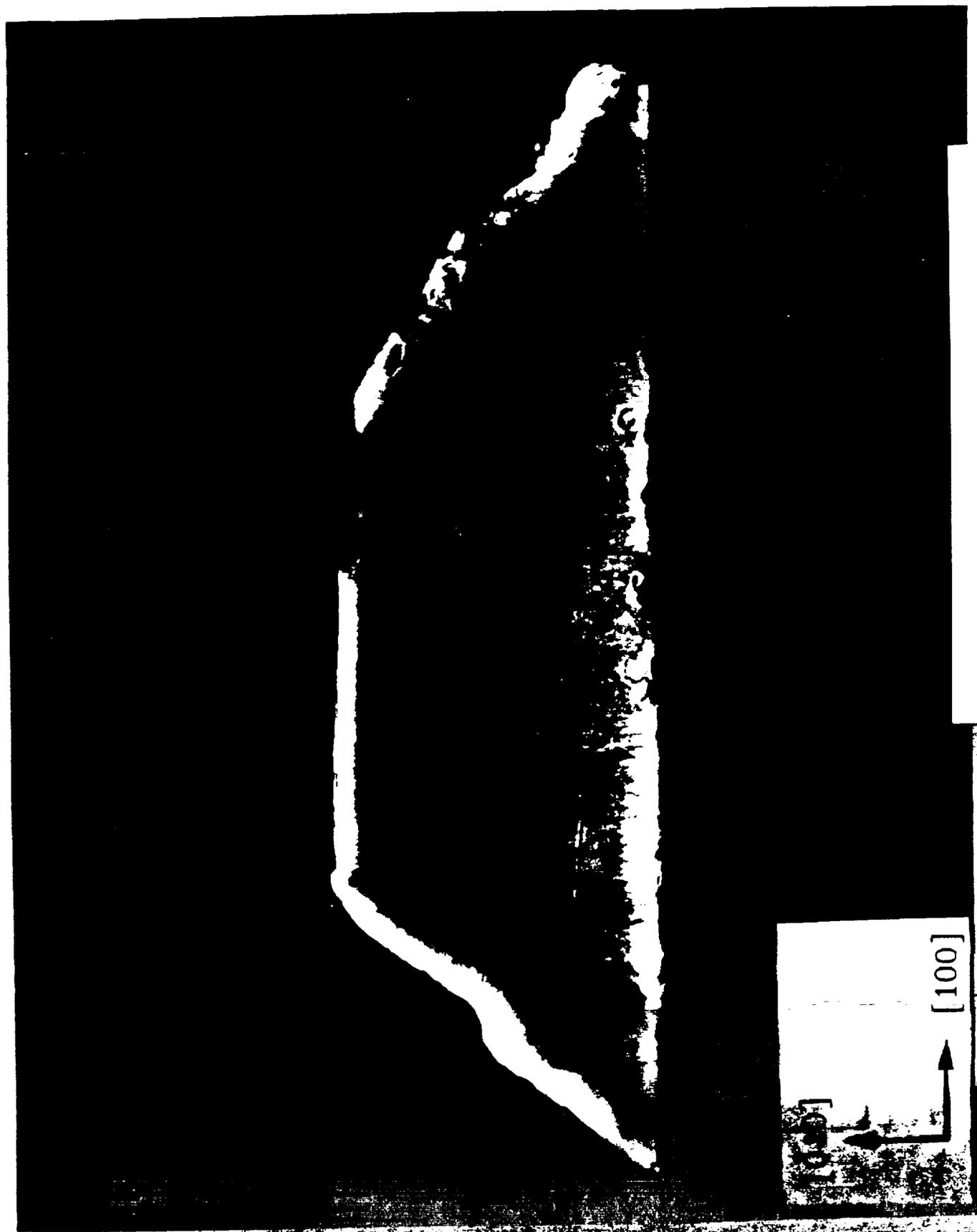


Interior of IML-1 Crystal 1F TGS-2: Dislocations In Seed (Run 1)



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Cut Edge of IML-1 Crystal 1F TGS-2 (Run 1)

$[100]$

**Holography Measurements
Primary Science Objectives**

Help explain the effects of the Spacelab Environment on Crystal Growth

- Observe and quantify convections
- Growth driven convection.
 - Convection due to g-jitter.
 - Convection due to other acceleration forces.
 - Convection due to residual microgravity.
- Correlate the presence of convection with other events.
- Correlate convection with crystal growth.
- Provide concentration and gradients in the vicinity of the crystal
- Support modeling

Secondary Science Objectives.

Collect basic scientific data in microgravity and particle, and fluid mechanics.

- **Observe g-jitter.**
- **Testing the theory of inertial random walk.**
- **Test Two Phase Flow and Particle Dynamics Theory in microgravity.**
- **Measure Low Frequency Residual Gravitational Field in Spacelab**

Summary of the Experiment

Approximately 650 holograms were taken in microgravity experiments.

- Four look angles, three in transmission and one reflection
- Cell one with cap removed from crystal, including dissolving and growth.
- Cell one with cap in place.
- Data covering 7 hours.
- Data covering 16 hours for cell one, with cap open.
- Video coverage

Variance between planned and actual experiment

Except for the shortened experiment times the data collected appears to be sufficient to complete secondary science objectives almost completely.

- Holographic interferometry data covers the restricted crystal growth experiment.
- Particle data covers shorter periods of low gravity than desired.
- Interruptions by spacecraft rotation:
 - Will give information about convection introduced by rotation.
 - Will reduce data accuracy
- Non uniform solution concentration will degrade the data quality.
- Lack of accurate concentration information will reduce data accuracy.

Data Reduction/Interpretation Plan

The vast amount of data produced in IML-1 requires a specific, prioritized data reduction plan.

Preliminary order of priority of importance

<u>Measurement</u>	<u>Type</u>	<u>Location/Time</u>
1. Convection:		
Growth driven	Part track small particles	Near xtal
g-jitter driven	Particle track small particles	Away from xtal
residual g	Particle track small particles	Away from xtal
other	Particle track small particles	Specific/roll

Data Reduction/Interpretation Plan (Cont'd)

<u>Measurement</u>	<u>Type</u>	<u>Location/Time</u>
2. Acceleration		
residual g	Particle track large particles	Away from xtal
g-jitter	Particle track large particles	Away from xtal
other	Large particles	Specific/roll
3. Particle Mechanics:		
Inertail random walk	Particle track large & small particles	Away from xtal Away from xtal
Two Phase Flow	Particle track large & small particles	Everywhere/roll
Particle Interactions	Large & small particles	Anywhere
4. Concentration	Hol. Int. Tomography	Near xtal/All

Preliminary evaluation of data and value suggests that we will be able to meet of the science objectives for TGS 1 Run

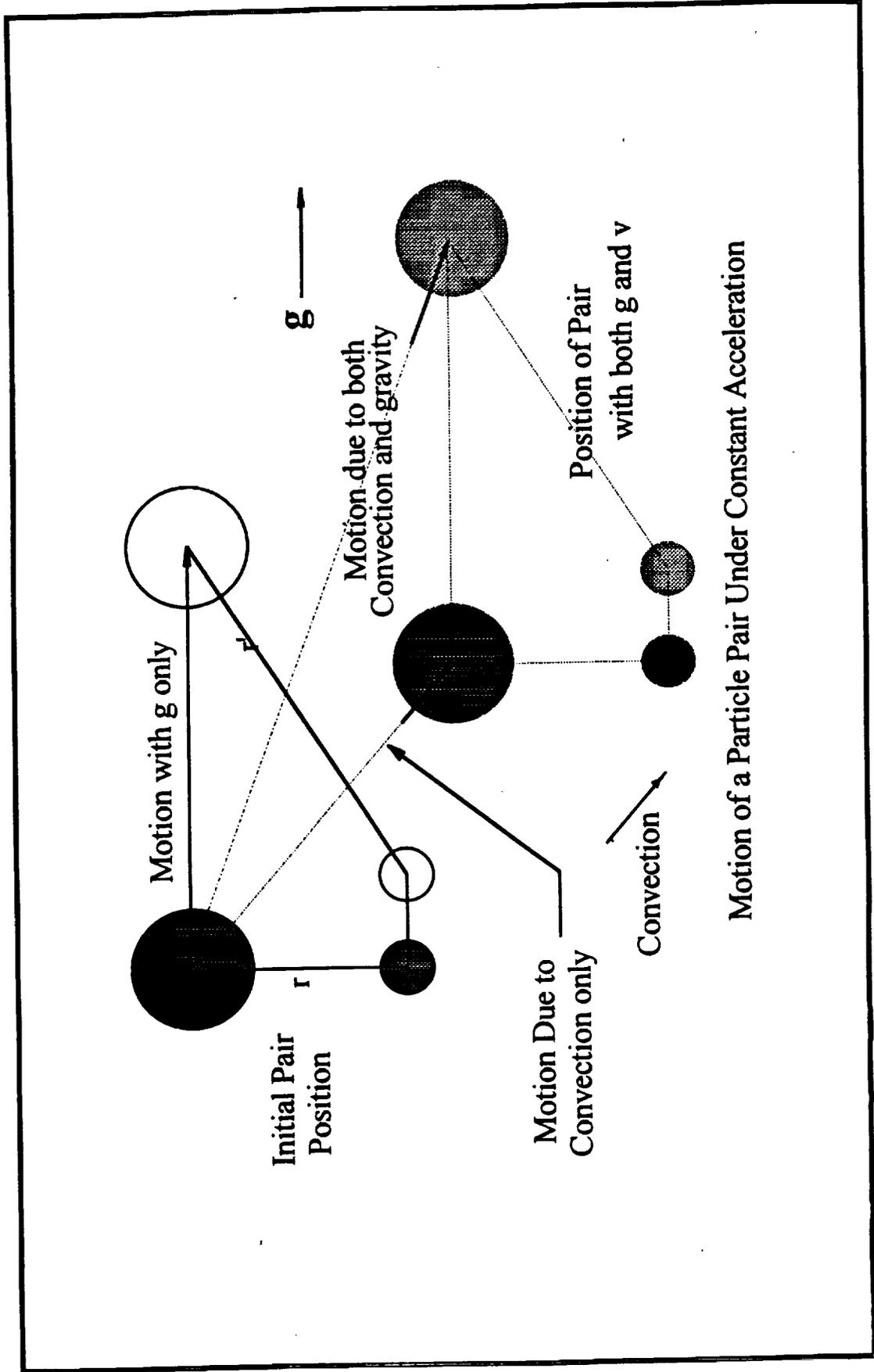
- **All holograms have been developed.**
- **The quality of all holograms is good.**
- **The suspended particle content in the TGS solution is good.**
- **All three particle sizes clearly resolvable in holograms over most of the cell.**
- **Holograms examined to date have been taken with sufficient time resolution to allow tracking from one hologram to the next.**
- **Most of the particles move enough to allow accurate measurements.**
- **Data reduction and interpretation in progress.**

Data taken during the rolls will provide*

1. Forcing function for low Reynold's number two phase flow.
2. Reference acceleration.

Separating Convection Effects From Gravity Effects.

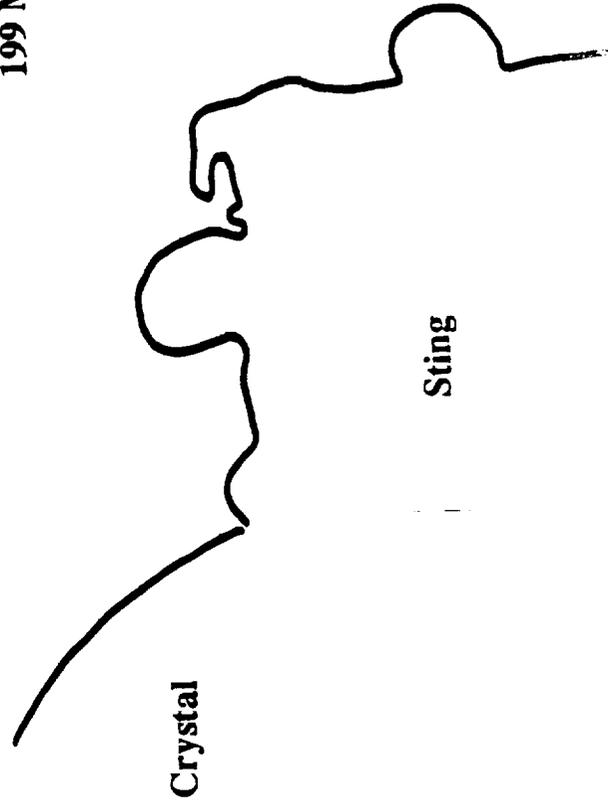
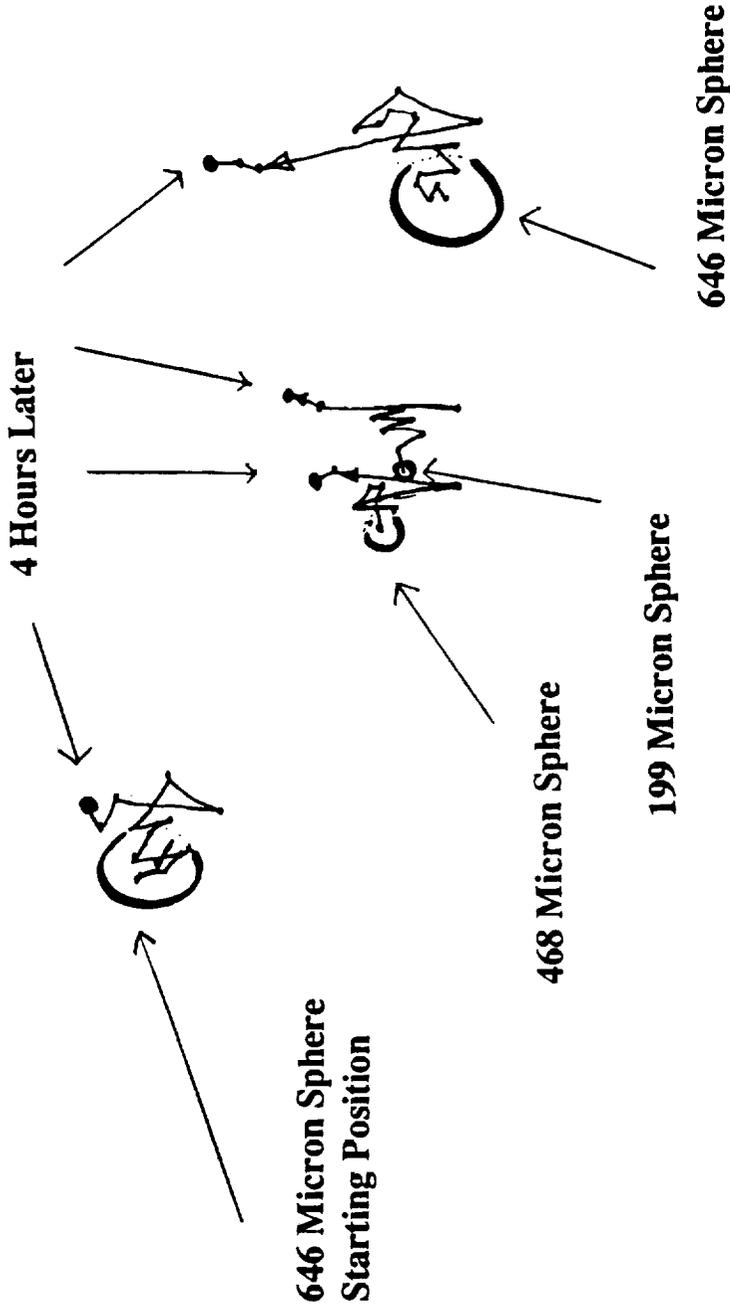
- * **Small and large particles both follow flow**
- * **Convection does not change particle separation**
- * **Under gravity $r = ka^2 g$**
- * **Gravity changes large / small particle separation**
- * **To measure convection requires registration of subsequent holograms**
- * **To measure g effects does not require registration**
- * **This will improve g measurement in presence of convection**
- * **Assumes convection velocity constant over measurement region**



Preliminary Results Show Significant Scientific Potential

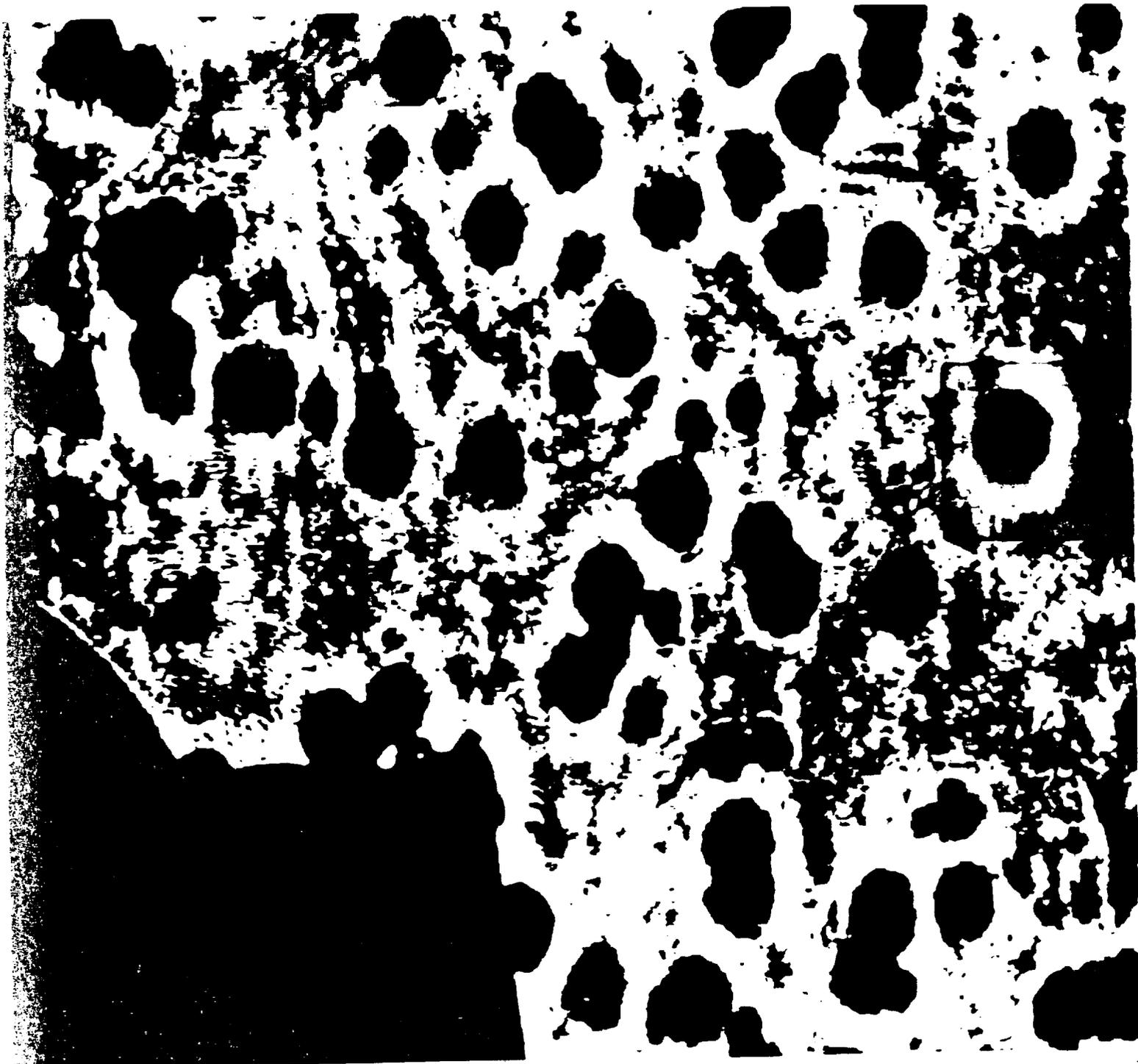
- **Particle tracks show a random walk type of motion**
- **Combined convection and acceleration are observed**
- **Typical measured accelerations range from a few micro g's up.**
- **Some Relatively large accelerations (>100 micro g's) observed during and after maneuvers**
- **Particle motions observed with no thermal and concentrations gradients in cell**

Four Hour Particle Tracks in Microgravity



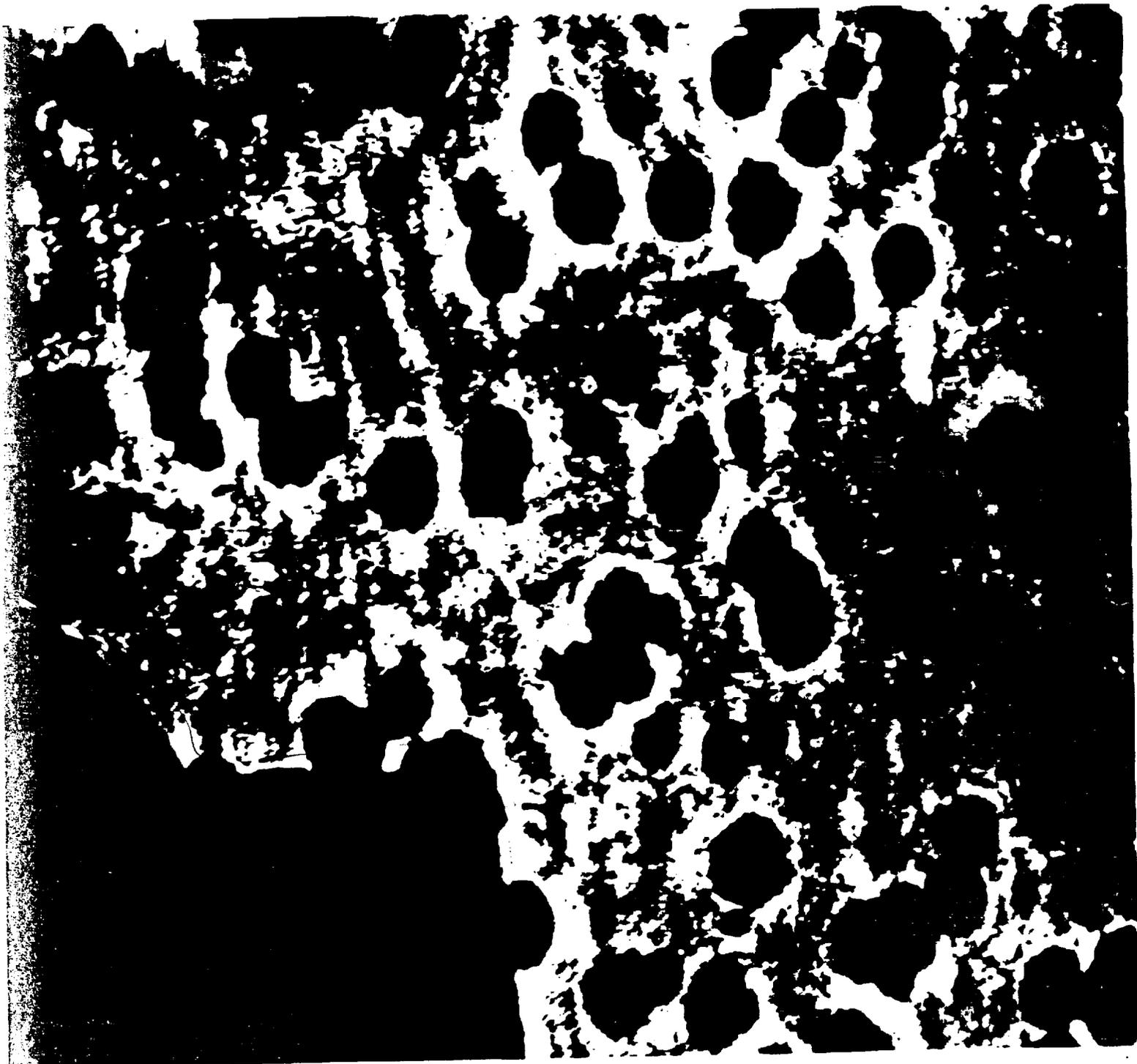
Particle Adhered to Sting

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Particle Field Images Reconstructed from Holograms (167) (one of 16
used to produce previous figure)

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Particle Field Images Reconstructed from Holograms (164) (one of 16
used to produce previous figure)