# Materials science research in microgravity – Current status and an experimental case study

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**The paper presents purpose and advantages of carrying out experiments in microgravity environment. Current areas of research in materials science being investigated in shuttle flights and those selected and pursued for ground-based research with possible flight experiments are discussed. Details of two experiments conducted on missions STS-78 and STS-87 are presented. The criterion of selection of these experiments, related ground based research and final flight definition and development issues are addressed. Finally, the paper briefly touches on the broader advantages of microgravity experiments related to general science awareness programmes at all levels.**

WHAT are the secrets that gravity masks? Gravity is a dominant factor in many chemical and physical processes on earth; but, as gravity's effects are eliminated, what are the results? Could new alloys be formed? How would liquids of unequal densities then mix? There are many theories and experiments which predict the answers to these questions, but the only way to answer and fully understand them is to effectively eliminate gravity as a factor. The microgravity environment of space or free fall conditions offer scientists several unique characteristics for conducting experiments that erase the effects of gravity.

In the beginning, research in microgravity environment was mainly pursued to develop novel processing techniques to design materials with unique properties. Later the objective was broadened to use microgravity to seek and understand quantitative cause and effect on relationships between the processing, properties and structure of materials. According to NASA, the objective of the microgravity programme is, 'using the microgravity environment of space as laboratory to explore physical phenomena sensitive to reduced gravity and studying the role of gravity in technologically important processes'. There are questions generated on values of fundamental properties. In this case, benchmark experiments are important candidates for investigations in microgravity. Benchmark experiments are designed to achieve a measurement accuracy not possible in a one-gravity environment. This would imply testing of theories to new levels of resolution that will serve as a standard for many years. This area encompasses research on transient equilibrium phenomena, as well as on other thermophysical measurements of interest in materials science. Last but not the least, is the interest and involvement of a large academic community of science teachers and students from schools and colleges in the programme, to create a new and unique awareness to improve the standards of teaching and learning. The broad microgravity science disciplines pursued by NASA/ESA are the following.

## Biotechnology

Microgravity environment is used to investigate bioprocessing phenomenon. The programme currently supports two major research areas; crystal growth of biological macro-molecules, cell and molecular science, focusing in particular on proteins and viruses.

## Combustion science

The combustion science research programme focuses on understanding the important processes of ignition, propagation and extinction during combustion in low gravity. Research is directed for achieving fundamental knowledge of combustion processes as well as addressing issues of fire safety in space. Shape and size of the flame and role of soot formation in combustion are studied.

## Fluid physics

The purpose of the microgravity fluids research programme is to improve understanding of how the presence of gravity either limits or affects the fundamental behaviour of fluid dynamics and transport phenomena. In low gravity, density-driven convection is greatly reduced, and therefore allows study of surface tension gradients or other phenomena that also drive such flows. The other important objective of this programme is to contribute to the knowledge of gravity-dependent fluid phenomena that may have an impact on their research in other microgravity science disciplines such as materials science and combustion.

## Materials science

The materials science programme uses the unique characteristics of the space environment to study fundamental

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issues in materials solidification and crystal growth. The current paper discusses in detail materials science-related research in the microgravity environment of space.

The phenomenon that is expected to be most influenced under microgravity environment is phase transformation from liquid to solid occurring during the solidification processing. Figure 1 shows the types of structures that form at the solidifying interfaces. The names on left describe the shape of the geometry of the interfaces. Some of the parameters effected by solidification conditions are:

- Type of interface
- Cell and dendrite tip velocities
- Shape and curvature of cell and dendrite tips
- Intercellular and interdendritic spacings
- Secondary arm spacings of the dendrites.

All the above parameters shown in Figure 1 strongly influence mechanical properties of the cast ingots of materials. These parameters, apart from thermal and compositional parameters, are significantly influenced by gravity-driven convection[1]. Hence, the relevance of solidification studies under microgravity conditions.

Figure 1 also shows the structure formation when insoluble particles or inclusions are also present in the system. After solidification, particles can be engulfed or pushed and the resultant structure can look different. This represents solidifying matrices in crystals, composites and commercial castings and ingots.

It should be appreciated that most of the engineering products at some stage of their manufacture have to undergo solidification processing. For example, any product made of metal or glass originates after casting of an ingot. Therefore understanding solidification science is of great importance. However, pursuing the original goal of microgravity processing, i.e. to develop novel processing techniques, semiconductor crystal growth in space was pursued vigorously for long time. Some excellent quality large size crystals of even compound semiconductors, have been grown in space. However, as mentioned earlier, it was realized soon that in microgravity environment, suppressing the buoyancy-driven convection can manifest

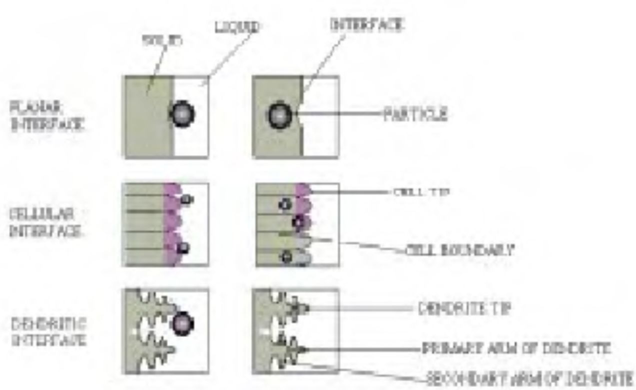


**Figure 1.** Schematics of various microstructures observed during solidification of melt containing insoluble particles.

strongly the influence of other physical phenomena like marangoni convection. It should be realized that surface energy is an important physical phenomenon that still needs to be understood. In summary, the potential benefits expected from the experiments conducted under microgravity are:

- Better understanding and control of materials properties for applications varying from high performance opto-electronic devices to corrosion-resistant metals.
- Better understanding and prediction of microstructure-controlled properties of cast metals and alloys.
- Improved utility of mathematical models for predicting materials properties during terrestrial processing.

So far the microgravity-related experiments related to materials science discipline have been presented as generalizations. It would be interesting to list what it means in terms of exact topics[2] on which lately experiments have been conducted in microgravity on space shuttles by NASA and ESA on various missions. If we consider the microgravity experiments conducted in the eighties by erstwhile USSR, they are more or less similar to those conducted by NASA, except that more experiments on semiconductor crystal growth were conducted by USSR compared to NASA. All the experiments listed below were approved by the NASA materials science programme, and were selected after rigorous scientific and technical peer review in the mid-nineties.

The following is a list of NASA's current experiments in progress and slated for running in future missions including International Space Station:

1. Coupled growth in hypermonotectics.
2. The effect of convection on morphological stability during coupled growth in immiscible systems.
3. Isothermal dendritic growth experiment.
4. Orbital processing of eutectics.
5. Orbital processing of high quality Zn-alloyed CdTe compound semiconductors.
6. Crystal growth of selected II–VI semiconducting alloys by directional solidification.
7. Growth of solid solution single crystals.
8. Comparison of structure and segregation in alloys directionally solidified in terrestrial and microgravity environments.
9. The pushing/engulfment transition for insoluble particles in metal and organic matrices.

ESAs current experiments on space shuttle mission STS (Space Transportation System):

1. Effects of convection on interface curvature during growth of concentrated ternary compounds.
2. Equiaxed solidification of aluminium copper alloys.
3. Comparative study of cells and dendrites during solidification of an aluminium–nickel alloy at 1-*g* and under microgravity.

The following are NASA experiments seriously pursued by NASA on ground with possible flight opportunities:

1. Fundamentals of mold-free casting: experimental and computational studies.
2. Effects on nucleation by container-less processing.
3. Investigation of the relationship between undercooling and solidification velocity.
4. Combustion synthesis of materials in microgravity.
5. Application of parallel computing for two- and three-dimensional modelling of bulk crystal growth and microstructure formation.
6. Fundamental studies of solidification in microgravity using real-time X-ray microscopy.
7. Investigation of vibrational control of the bridgman crystal growth technique.
8. Measurement of the viscosity and surface tension of undercooled melts under microgravity conditions and supporting MHD calculations.
9. Role of dynamic nucleation at moving boundaries in phase and microstructure selection.
10. Effects of gravity, reaction environment and composition on the combustion synthesis of the $TiB_2$–$TaB_2$ system.
11. Detached solidification: Steady state results.

Experiments temporarily discontinued by NASA from flight experiment list due to inadequate scientific analysis and understanding of the ground-based experiments:

1. Gravitational effect on the development of laser weld-pool and solidification microstructure.
2. *In situ* monitoring of crystal growth using MEPHISTO.

Among the above, the last two experiments need some comments. Laser weld pool or for that matter general weld pool behaviour in microgravity environment is not only important from the point of view of understanding the structure of the welded region but also to understand how to design structures in space through welding, for example in construction of a space station. However, for the purpose of developing appropriate welding technology for the space station, welding experiments purely from the point of view of technology, are being conducted in space. But this area of research still needs to come up with appropriate model and experimental design to make microgravity experiments fruitful. In comparison, *in situ* monitoring of crystal growth using MEPHISTO facility[3] that involves monitoring Seebeck voltage or signal, as it is called, at the solidification interface generated by interface undercooling due to kinetics and/or solutal effects, was theoretically well understood. The model was based on sound physical principles and the experiments were conducted on a sophisticated set-up in consecutive four missions. Analysing the results of experiments so far has been difficult because a clear quantitative relationship between the Seebeck signal and the interface undercooling is yet to be established for non-planar interfaces. Therefore, pending further development of science, these experiments are on hold. Nevertheless, both the experiments have high potential in terms of science returns by further microgravity experiments. Therefore, experiments related to measurement of Seebeck signal in convection-free environment of space in Glovebox are planned to be pursued.

The summary of the salient features of the materials response in the microgravity environment has been given in Table 1. For further understanding of the development of microgravity research programmes, it would be interesting to look at the triangles[2] given in Figure 2. Some terminologies in this need to be explained.

Ground-based research: Research carried out in the ground-based laboratories, DC-9 aircraft and drop tubes in low gravity or simulated low gravity with appropriate model developments is termed as ground-based research. It is to be noted that experiments on DC-9 and drop tube, where microgravity environment is available for periods less than 20 sec are also considered as ground-based research.

Flight definition research: At this stage, the research programme is carried out for final determination, i.e. whether the research will go for flight. A science panel and an engineering panel conduct a review after which the research experiment goes to the next stage; flight development phase.

It can be seen that over the years ground-based research has increased in number compared to flight approved

**Table 1.** Materials response as observed in microgravity experiments

| Microgravity environment | Materials response |
|---|---|
| Buoyancy-driven convection flows minimized | Precise temperature and composition control for high quality crystals |
| Body force effects minimized | Uniform spacing and alignment in multiphase materials |
| Container-less melt processing | Eliminate contamination and nucleation due to containment |
| Interfacial phenomena | Wetting and surface energy driven flows |

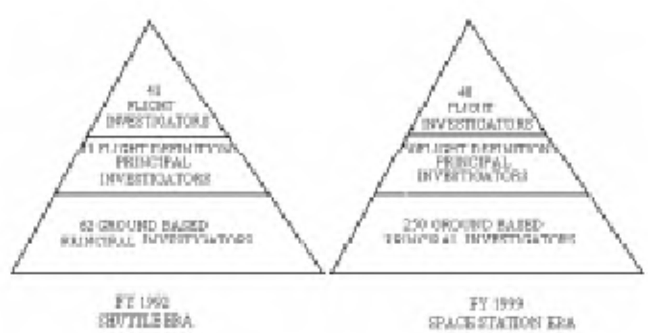


**Figure 2.** Development of the NASA microgravity materials research programmes.

experimental research. Initially research experiments run on flight without adequate ground studies led to unexpected results and most of the times science objectives were missed. For example, the initial experiments to determine the effect of microgravity on the alteration of the metallic eutectic structures led to some difficulty in the analysis of the results. Normally, the effect of microgravity environment is judged on the basis of comparison of solidification of two samples under identical solidification conditions and in identical set-up in ground conditions and in microgravity. But in case of eutectics, the problem to resolve was whether the change in structure resulted due to the absence of convection in the melt or due to the altered conditions of heat transfer in the furnace in microgravity. This can happen because, the film of gas (often helium), between the furnace tube and the specimen cartridge becomes stagnant, due to the absence of buoyancy-driven convection in microgravity. Currently, these issues are being addressed in a limited way by rigorous heat transfer modelling. It is to be noted that this issue is also of importance, to crystal growth experiments. The above problem therefore became again a candidate for intensive ground-based research. The ground research is necessitated for identifying suitable science concept and hardware development for flight experimentation. Thus, the three phase research programmes, viz. ground-based research, flight definition stage and final flight investigation have evolved.

## Experimental case study

The development of a research programme leading to two shuttle flight experiments in missions STS-78 and STS-87 is now described. The main objective of this section is to show the evolution of space lab experiments, from identification of the problem, to its successful execution in the space.

The research programme is entitled: 'Particle engulfment and Pushing by solidifying interfaces' ('PEP' in short). The phenomenon of interaction of particles with melt interfaces has been studied since the mid 1960s[4]. While the original interest in the subject was mostly theoretical, researchers soon came to the realization that understanding particle behaviour at solidifying interfaces may yield practical benefits. The problem is relevant to:

- Growing of $Y_1Ba_2Cu_3O_{7.8}$ superconductor crystals from an undercooled liquid. Knowledge of particle behaviour can help in obtaining uniform distribution of 211 precipitates.
- In cryobiology this knowledge can help in creating conditions for engulfing the cells by the solidifying interface, thus leading to their uniform distribution. At the same time salt build up in front of the interface has to be avoided, otherwise build up of salt leads to destruction of the cell due to change in pH.
- The ice–water interface during frost formation in the northern hemisphere leads to pushing of the soil and causes cracks in the high ways, termed as frost heaving.
- Last but the most researched aspect has been to utilize this knowledge to obtain uniform distribution of the reinforcing ceramic particles in metallic matrix. During solidification, if the particles are pushed by the interface, the resultant composite would have non-uniform distribution of the particles, causing loss of mechanical properties.

The inference from the above is that, for a research problem to be candidate for space experiment, it is desirable that it should have multifarious applications. Next, the problem must have a quantitative measurable parameter. In this case, experimental evidence demonstrates that there exists a critical velocity of the planar liquid/solid interface below which particles are pushed ahead by the advancing interface and above which particle engulfment occurs.

There are several models developed by different scientific groups to relate critical velocity with different thermophysical parameters. One such model is due to Stefanescu *et al.*[5]. This model originated from the concept in Stefanescu *et al.*[6]:

$$V_{cr} = \left( \frac{\Delta\gamma_o a_o^2}{3\eta K^* R} \right)^{1/2}$$

where $V_{cr}$ is the critical velocity for engulfment; $\Delta\gamma_o$ is the difference between the interfacial energies of the liquid, solid matrix and particle; $\mu$ is the liquid viscosity; $R$ is the radius of the particle; $a_o$ is the interatomic distance and $K^* = K_P/K_L$, is the ratio of thermal conductivities of the particle and liquid, respectively.

Most of the analytical models follow the above pattern. Two points that need to be noted are, these models are generated assuming that the interaction between the particle and the melt interface occurs in the absence of convection. Development of analytical models to understand the physics of the interaction is only possible assuming absence of convection. The basic physical parameter in the above models that need to be known accurately is $\Delta\gamma_o$. Here it must be pointed out that accurate determination of $\Delta\gamma_o$ in essence, requires accurate value of surface energy between the two solids. This is a physical parameter of importance in many technological activities apart from composites. For example, deposition of thin films on audio/video systems, CDs and even for characterizing the life of refractories in steel melting units. Therefore, its accurate determination is an important fundamental issue. For correct determination of critical velocity, experiments need to be conducted in a convection-free environment in space. Here again, it may be noted that accurate determination of critical velocity value and its substitution in a previously validated model, can also give accurate value of $\Delta\gamma_0$ for the system under investigation. In a way, these experiments can be categorized as benchmark experiments for determining $\Delta\gamma_0$.

However, before the experiments for measurement of critical velocity were approved in the space shuttle, the research was initiated in ground conditions in 1987 (ref. 6). Concurrent development of models relating critical velocity with thermophysical parameters and experimental measurement of the critical velocity were pursued. Experiments were also conducted in KC-135 in parabolic trajectories[7]. In 1992, NASA accepted a project for systematic investigations under ground conditions to refine the model, and for developing the experimental procedures for measurement of critical velocity. At the end of about two years, the investigations were evaluated for the flight definition stage and approved for possible flight in the space shuttle mission in June 1996. There were few riders about technical requirements of the experiment. One such requirement was to develop a technique for characterization of the particle redistribution. The other was the design of the specimen-holding cartridge, such that in space marangoni convection does not affect the liquid metal at the interface. For this spherical zirconia particles were chosen, so that they can be pictured by X-ray transmission microscopy, for aluminum matrix samples of 1 cm diameter. Design of the cartridge, evolved where minimum effect of marangoni convection is expected, is shown in Figure 3. The special design[5] of the graphite spring-loaded sample, compensating the shrinkage of the solidifying melt, and thus avoiding creation of free surface, is the special feature of the cartridge selected for flight sample FM1.

Later, in the year 1995, the project was subjected to science and technical readiness review and approved for running in Advanced Gradient Heating Facility (AGHF), which was designed and fabricated under the supervision of ESA at Toulouse in France. After the review, it was accepted, that the PEP experiment can successfully be carried out in the space shuttle and significant science returns would be obtained. Three experiments were conducted in the space lab (AGHF facility) aboard space shuttle *Columbia* in the mission STS-78 in June 1996. The location of the space lab in *Columbia* is shown in Figure 4. There was a tunnel from the middeck to get into the lab. The experiments were run by French scientist/ astronaut J. J. Favier. His work was only limited to changing the cartridges in AGHF. The experiments were controlled from ground by telemetry from ground facilities at Marshall Space Flight Centre (MSFC) at Huntsville, USA. The parameters controlled were hot zone temperature to control thermal gradients, furnace translation speed and location of the initial directional solidification interface. The experiments were mostly run during the sleep period of the astronauts so that no disturbances and jitters were there due to physical workout or walking of the astronauts. After the experiments, the cartridges were opened at ESA facilities. After preliminary examination of the integrity of the cartridges, they were brought to the University of Alabama for detailed examination of the samples. The results of the experiments have been published elsewhere[5,8].

The results did show a critical velocity of engulfment of zirconia particles in aluminum matrix, different from ground measurements, in line with model prediction. However, because of the limitation of the number of samples which could be run on this mission, there were scanty data points to confirm the model.

The above experiments were conducted on samples of ceramic (zirconia) dispersed in metal matrix (aluminium). In the second series of PEP experiments, the behaviour of insoluble particles (polystyrene and glass) in transparent organic matrices (succinonitrile and biphenyl) was studied. Here it can be pointed out that succinonitrile solidifies in a non-faceted manner and biphenyl in a faceted manner[1]. The experiments were to be conducted in a Horizontal Gradient Facility (HGFQ) similar to the one in which ground experiments were conducted. The science concept was the same as in PEP on STS-78, therefore after preliminary review the science concept was approved. A second review was slated in March 1996. Before that, it was required to demonstrate that the required science returns were not possible by conducting experiments on parabolic trajectory flights. HGFQ was flown on DC-9 flights and it was demonstrated that stability of the melt interface is not at all achieved in the short duration of

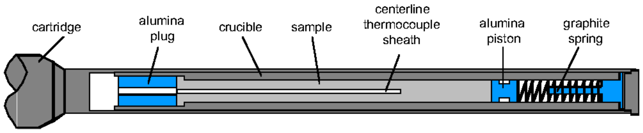


**Figure 3.** Cartridge design for sample FM-1 used in PEP experiment on STS-78.

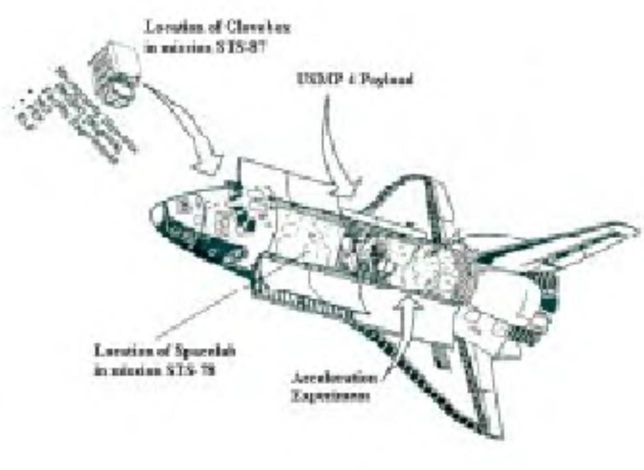

**Figure 4.** Location of spacelab and Glovebox in space shuttle *Columbia* on missions STS-78 and STS-87, respectively.

20 sec of microgravity obtained in these flights. The review in March 1996, was mostly from the point of view of assessing, in necessity of running the experiment in long duration microgravity to correctly evaluate critical velocity in the systems proposed to be studied. After rigorous review, the science readiness was approved. The results of the ground-based research have been published elsewhere[9]. The technical readiness was redesigning the HGFQ for mounting in Glovebox facility to be flown in space shuttle *Columbia* in mission STS-87 in November 1997. This was accomplished with redesigning of the facility and its construction with the help of Tecmasters Company. Mostly the design constraints were power requirements, ease of sample loading and video recording of the activities at the interface of the melt at a magnification of 400 ×. Experiments in Glovebox were performed by the active involvement of the astronauts for running the experiments during flight. The video recording of the experiment was directly transmitted to ground laboratory at MSFC, where the investigators could monitor the experiments and could, in a limited way, adjust the experimental parameters. Eight experiments were run during the mission in November 1997. The payload specialists on the mission were astronauts Takoi Doi and Kalpana Chawla. The astronauts were trained on running the samples in the hardware prior to the flight, when the HGFQ facility was at the University of Alabama and in MSFC at Huntsville.

One major unexpected result, that was noted in the very first run in space, was the strong influence of the non-wetting nature of polystyrene particles in molten succinonitrile matrix. This influenced strongly their dispersion behaviour in the melt. The individual dispersed particles immediately came together and formed large agglomerates. This has been shown in Figure 5. The picture has been taken from the website of this experiment where further details of the experiments and USMP4 mission are available[10]. This behaviour of agglomeration was not observed under ground conditions, perhaps due to the effect of convection that did not allow the non-wetting character of the particle and matrix to form agglomerates. Although some agglomeration was expected in reduced gravity environment, but not to the extent seen. This points out that, presently continuing experiments can open up new issues in science for further investigations. For example, in this case, the physics of the wetting behaviour.

For the success of the PEP experiments, it was necessary to follow the behaviour of individual particles in front of the interface as, in this case only, the correct critical velocity could be evaluated. For this, the interaction facility with the astronaut was used. The runs were re-designed, such that the solidified matrix was remelted at different stages and re-run. This helped in getting good science returns from the experiments. All the experiments were video-recorded. The full recording was obtained only in the recording facility on the shuttle. In the ground when the shuttle goes in orbit at certain positions, the link on the video channel is lost. The results of the experiments are still under processing. Critical velocities for several diameter ranges of particles have been measured. However, for the validity of the model, critical velocities over a range of diameter of particles are required. In the mission STS-87, the data for particles in the range from 1 to 3 μm could not be obtained as particles in this diameter range tended to agglomerate heavily and in some runs, they were not available at all as single particles in front of the melt interface. But overall, the data available form the eight experiments conducted during this mission were sufficient to add to the understanding of the physics of the particle–melt interface. These experiments demonstrate that, although the direct involvement of astronauts in running the experiments is very expensive in terms of astronaut time, the science returns can be salvaged in case of unexpected behaviour in microgravity.

Another short case history[11] involves the isothermal dendrite growth experiment (IDGE), conducted on mission STS-87 by a group in the Materials Science and Engineering Department of Rensselaer Polytechnic Institute at Troy, NY. This experiment has some interesting

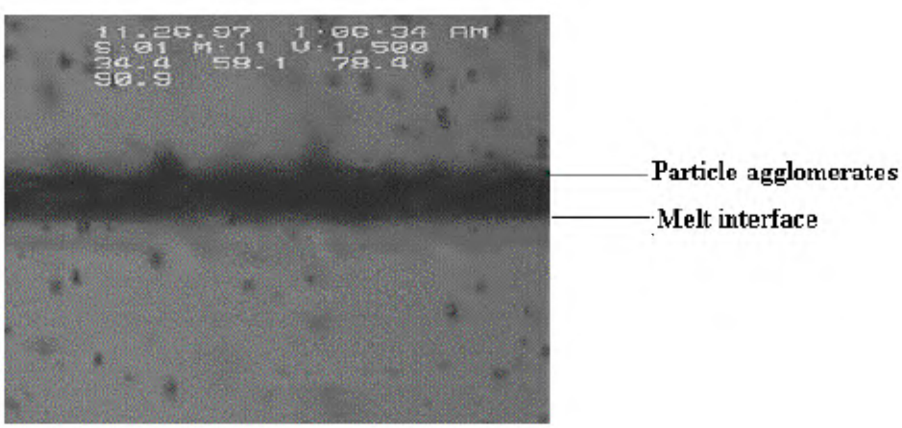


**Figure 5.** Particle agglomeration in front of the moving interface of succinonitrile in space shuttle experiment. Particles are polystyrene of diameter range 2–15 μm.

facts. The series of experiments conducted on IDGE in space shuttles has resulted in the setting up of a center for Initiatives in Pre-College Education (CIPCE) at Rensselaer and the Greater Capital Region Teacher Center (GCRTC). Here, during the preparation for STS-87 mission activities, 25 science teachers were selected, to attend a two-week summer institute where lectures from content experts, hands on activities and development of specific curricula were undertaken. These curricula would affect teaching of about 1200 secondary school students in concert with STS-87 activities.

It would be in order now to summarize some of the salient points, that need to be taken into consideration, for selecting and planning experiments to be conducted in microgravity environment. These experiments could be of short duration as in drop towers, parabolic flights, balloons and sounding rockets or long duration as in space shuttle or space station.

A good number of ground experiments should be supported, in order to choose among the flight experiments. It may be noted that experiments in drop towers, parabolic trajectory flights and perhaps balloons can be taken as part of ground experiments. What should be the minimum requirements for selection in both these categories?

### *Ground-based research*

- Provides intellectual underpinnings of the flight programme.
- Experimental and theoretical inputs need to be developed concurrently.
- Well-articulated microgravity relevance.
- Demonstration of the role of gravity; benefits to be accrued from conducting research in microgravity.

### *Flight experiments*

- High scientific and technical merit.
- Well articulated need for a long duration, high quality microgravity environment established through ground research.
- Clear perspective whether the flight experiments are suitable for full telemetry control of experiment or in Glovebox with direct involvement of astronauts.
- Experimental and theoretical maturity to support a science concept review within two years, considering the present pace of developments.

There is abundant and compelling evidence that any space experiment must be planned, executed and analysed carefully following the knowledge base developed from terrestrial studies. When such an approach has been pursued, real progress and significant advancement of microgravity science have been achieved.

1. Kurz, W., Fischer, D. J., *Fundamentals of Solidification*, Trans Tech Publication, 1986.
2. Proceedings of the NASA Microgravity Materials Science Conference, Huntsville, Alabama, 10–11 June 1996.
3. Favier, J. J., Garandet, J. P., Rouzaud, A. and Camel, D., *J. Crystal Growth*, 1994, **140**, 237–243.
4. Uhlmann, D. R., Chalmers, B. and Jackson, K. A., *J. Appl. Phys*., 1964, **35**, 2986–2993.
5. Stefanescu, D. M., Juretzko, F. R., Dhindaw, B. K., Catalina, A., Sen, S. and Curreri, P. A., *Metall. Trans. A*, 1998, **29**, 1697–1706.
6. Stefanescu, D. M., Dhindaw, B. K., Kacar, S. A. and Moitra, A., *ibid*, 1988, **19**, 2847–2855.
7. Dhindaw, B. K., Moitra, A., Stefanescu, D. M. and. Curreri, D. M., *ibid*, 1899–1904.
8. Downey, J. P., Report on LMS, Marshall Space Flight Centre, Alabama, Huntsville, USA, February 1998.
9. Sen, S., Dhindaw, B. K., Stefanescu, D. M., Catalilna, A. and Curreri, P. A., *J. Crystal Growth*, 1997, **173**, 574–584.
10. http://liftoff.msfc.nasa.gov/shuttle/usmp4/science/pep1.html
11. Fourth United States Microgravity Payload, Minutes of the Third Investigators Working Group, Huntsville, Alabama, USA, 9 September 1997.

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