Trends and opportunities in basic research in microgravity materials processing

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Internationally the microgravity materials research has reached a matured state with several innovative programmes by different space agencies. The present article highlights underlying scientific issues that some of these programmes aim to address. The article strives to show that some of these issues are basic in nature and provides knowledge base which are of importance to materials processing in general. These space experiments provide a challenge to the Indian scientists to launch a fruitful initiative.

EVERY major space programme in the world devotes considerable attention to materials. The interest spans from immediate engineering requirement of the space programme to materials behaviour under space environment and finally materials processing in space. The sequence roughly represents the need of the present to the expectations of the future. The interest of the scientific community to understand the processing and the behaviour of the materials under the microgravity conditions arises from several different considerations. It spans from a dream of space colony to requirement of the repairs in manned and unmanned space exploration and finally to scientific curiosity. One expects that materials processing in space will not only enable us to understand the role of gravity in some of these processes but also enable synthesizing new materials. The programme of NASA and European Space Agency (ESA) have also a significant component aimed at education and sensitization of the general public and scientific communities towards issues involved in the exploration and exploitation of the space in larger time-frame.

There are several facilities, which are used for materials processing experiments under microgravity conditions. These are drop tubes and the sounding rockets, aircraft with parabolic flight path, space shuttles and space platforms and surprisingly in recent times, balloons. Among these, the drop tubes and towers are essentially ground-based experiments and provide few seconds of microgravity time. The parabolic flight experiment of NASA also can be classified in this category. In early days, NASA carried out experiments of materials processing using recoverable capsule in sounding rockets. However, it is now replaced by balloon experiments, which are more versatile in terms of payload weight and cost. Most of the space experiments are however carried out in space shuttles by NASA and ESA and in spacecrafts like *SALYUT* (*SALYUT/MIR* space platforms) by Russia. The various experimental programmes on materials sciences by different space agencies are listed in Table 1. India is currently planning to embark on similar new programmes in this area. The article by Thomas *et al.* in this special section deals in some detail the platforms for space experiments and the Indian perspective on this issue.

Although initial experiments for microgravity research faced several technical difficulties, the capabilities have matured in recent time to yield significant scientific information. The experiments utilizing United States microgravity payload at the Cargo Bay in shuttle flights and in particular, the cooperative programmes like the one between NASA and CEA/CNES of France (acronym MEPHISTO) yielded important results on solidification behaviour of melt. The International Microgravity Laboratory Mission (IML) 1 and 2 carried out a large number of experiments, including several critical ones for materials science like melting, solidification, dendrite and

Table 1. Major international programmes related to materials processing in the world

Space agency/country	Programme
NASA	Drop tube
	Parabolic flight with planes
	Sounding rockets
	Space shuttle
	International space station*
ESA	Drop tube
	Balloon experiments
	Space shuttle (in collaboration with NASA)
	International space station
Russia	Space stations
Japan	Drop tube
	Balloon/sounding rocket
	Collaboration with NASA in space shuttle
China	Drop tube
	Balloon experiments
India	One experiment in Russian space station
	New initiatives in planning stage

*To be operational beyond 2004.

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crystal growth. The recent experiment in this line for which considerable efforts have been made is the programme of 'Measurement of thermo-physical properties'. In the rest of the article we shall briefly describe some of the scientific issues involving materials science microgravity experiments that are being pursued internationally.

Measurement of thermophysical properties

The microgravity environment provides excellent opportunities to obtain the benchmark properties of a liquid. Since the liquid can be undercooled, it provides rare opportunities in measuring properties of a metastable liquid. The latter is important in understanding the solidification behaviour of a wide range of metastable materials. In general, the effort to measure physical properties of the liquid under terrestrial conditions suffers from the presence of gravity-driven fluid flow. In some cases elimination of this effect is attempted by evaluating the contribution of the fluid flow theoretically. However, experiments in the microgravity condition are expected to yield results, which primarily reflect a situation where the transport of the fluid is only diffusive in nature. Some of the current efforts on these measurements are detailed below.

Specific heat

In general specific heat of most metals is similar near the melting point and generally close to ~ 30 JK^{-1} mol^{-1}. It is often observed that increase in temperature leads to a decrease in specific heat followed by an increase. This behaviour is often taken to reflect the existence of clusters just above the melting point. For undercooled liquids, the experimental results suggest an increase in specific heat with undercooling. The limited specific heat data in the undercooled liquid suggest universal behaviour in comparison to its value at the melting point. Fecht and Johnson[1] have shown that it is given by a relation $C_p^l(T) - C_p^l(T_m) = 9 - 9T/T_m$ JK^{-1} (g.atom)$^{-1}$. The data on specific heat of alloys is extremely limited. However, they suggest that undercooled alloy melts show a similar behaviour with increase in specific heat with decreasing temperature.

The major difficulty of getting specific heat data for the metastable melt is its stability. The container often triggers the nucleation of the solid, thus making the measurement of metastable liquid extremely difficult. Microgravity provides an excellent opportunity to overcome some of the difficulties by resorting to container-less processing. The scheme of the experiment is due to Fecht and Johnson[2]. These investigators proposed a variant of noncontact ac calorimeter used in low temperature physics. Here, power to the levitated droplet in space is modulated and the temperature response is followed with a noncontact thermometer. Under a certain frequency

condition (ω), the temperature change (ΔT_ω) and modulation can be related by a simple expression given by $\Delta T_\omega = P_\omega/2\omega C_p$. Thus, specific heat can be determined accurately.

In the IML2, the specific heat measurements as a function of undercooling were carried out on glass-forming alloy systems. In particular, reliable data of specific heat change at low undercooling were obtained for the glass-forming ZrNi and at moderate undercooling for NbNi alloys. The results suggest increase in specific heat with increasing undercooling. For example, the specific heat of ZrNi was found to follow an empirical relation[3]:

$$C_{\text{ZrNi}(T)} = 57.8 - 1.002 \ (10 - 2T) \ \text{J/Kmol}.$$

Thermal and electrical conductivity

The thermal conductivity of a melt arises from the vibration of atoms. However, this contribution cannot be separated from the contribution due to the convective flow in earth. Generally, the thermal conductivity of metallic melt is obtained from the Weisman–Franz law, which relates to the thermal and electrical conductivity.

$$\kappa/\sigma_{\text{el}}T = L_o,$$

where L_o is the Lorenz number and consists of natural constants only. The electrical conductivity is less affected by the convection and hence can be more reliably measured. However, the melt has to be contained in a crucible and this restricts the measurements at high temperature. Thus, the data exist only for low melting materials. One of the ways out of this is to levitate the melt and measure the electrical conductivity by an inductive method using an oscillatory circuit[4]. Unfortunately, this method is accurate only for simple geometry and particularly for sphere. Terrestrial experiments always yield a distortion in spherical shape. Thus, microgravity seems to be the most suitable method for the measurement of the electrical conductivity over wide temperature range. This also permits measurement of the conductivity of the metastable liquids with a potential of yielding new results. Such experiments are planned as a part of the TEMPUS facility developed by Germany and being utilized jointly by Germany and USA.

Surface tension and viscosity of the melt

Microgravity provides excellent opportunity for reliable measurement of the surface tension and viscosity of the melt over a wide temperature range. The prospect of measuring these quantities for the metastable melt at different undercooling makes it attractive for understanding the liquid–solid phase transition. The oscillating drop technique is particularly attractive for microgravity envir-

onment since the drop can be suspended freely and will have a perfectly spherical shape. Thus one can induce oscillations and follow them around the spherical equilibrium shape. The frequency (ω) and damping (Γ) of the oscillation can be related to the surface tension (σ) and viscosity (η) through the Rayleigh and Kelvin equations.

$$\omega_R^2 = 32\pi\sigma/3M,$$

$$\Gamma_\kappa = 20\pi R\eta/3M.$$

Both these equations assume perfectly spherical shape and the experiments in earth require corrections. No such corrections are needed for the microgravity experiments.

Some of the systems on which experiments were carried out in IML2 are Au, Cu, AuCu, Zr and Zr alloy ZrNi (ref. 3). Table 2 presents some of the available results. The results on surface tension indicate a difference with that obtained on the earth. However, if one incorporates the correction in shape in the earth, the agreement seems excellent. The viscosity results, however, were less reliable since it could not be established that the observed damping is only due to internal friction and no other contribution has entered the process. Clearly a great opportunity exists in the future to carry out more experiments in this direction.

Phase selection

The microgravity materials processing provides an opportunity to study the microstructure selection mechanisms and the pathways for the microstructure evolution during liquid–solid transformation. The nucleation of solid has been extensively studied in the last half a century. The nucleation of solid in the melt occurs by the fluctuation process, which brings the cluster of atoms with right configuration into existence. The stability of these clusters depends on surface energy since the formation of the cluster leads to the creation of an interface, which results in a positive energy contribution to the total free energy. Thus, nucleation requires higher undercooling which provides the additional driving force needed to overcome this barrier. This is well treated theoretically and is popularly known as the classical theory of homogenous nucleation[5]. The presence of catalytic surface may aid the process of nucleation by providing surfaces where the new phase can form. In this way, the positive energy contribution of the

surface newly created by the embryo of the solid can be reduced. Since melt is confined in a container, the container surface often provides the catalytic surface. In addition, the melt contains impurities and undissolved oxides, which can act as catalytic sites. Thus, most of the solidification processes encountered in nature start by the heterogeneous nucleation process. In an effort to prove the classical nucleation theory, one resorts to cleaner melt which is then subdivided to segregate the impurities into fewer particles. Repeated subdivision leads to a large fraction of particles, which are free of impurity particles, and the solidification behaviour of these particles is used to arrive at a test for the classical nucleation theory[6,7]. An alternative approach can be to process the melt in space, which avoids the need for a container. Further, it is possible to create clean vacuum so that the possibility of formation of dispersed impurities can be eliminated. Thus, a considerable number of experiments in drop tubes, and space platforms are directed to the undercooling and nucleation studies. Table 3 gives the undercooling achieved to date using long drop tubes[8]. Since the grain structure originates during the nucleation, it has a great implication on the grain refinement. The elimination of the heterogeneous nucleation sites also leads to other possibilities. In the absence of the heterogeneous nucleation of the equilibrium phase, it is possible to form metastable phases, which may have a nucleation advantage in certain temperature domains over the homogeneously nucleated solid. The first drop tube experiment, in fact, revealed these possibilities when a metastable bcc phase was reported[9]. The possibilities of forming a hierarchy of metastable phase are much greater in alloys and these include glass formation. The MSL1 mission of NASA in 1997 has carried an experimental module designed to study the evolution of phases in ternary Fe–Cr–Ni steel. The results suggest clear distinction between the space-processed and the earth-processed alloys. This difference owes its origin to not only the nucleation process but also the growth process and the impact of fluid flow in these processes[10].

Table 2. Surface tension data obtained in IML2 experiments using oscillating droplet technique[3]

System	Relation (T in $^{\circ}$C)
Au	1.149 – 0.14 (T – 1064) N/m
Au$_{56}$Cu$_{44}$	1.205 – 0.15 (T – 910) N/m
ZrNi	1.545 + 0.08 (T – 1010) N/m

Table 3. Maximum undercooling of pure metals achieved in long drop tubes[19]

Element	Under cooling ΔT (K)	Reduced undercooling ($\Delta T/T_m$)
Hf	530	0.21
Ir	430	0.16
Mo	520	0.18
Nb	525	0.19
Pt	380	0.19
Re	975	0.28
Rh	450	0.20
Ru	330	0.13
Ta	650	0.20
Ti	350	0.18
W	530	0.14
Zr	430	0.20

Microstructure evolution

The evolution of microstructure in the reduced gravity conditions is expected to be significantly different. The expectation flows from the fact that under the microgravity condition, gravity-driven fluid flow is absent. This coupled with the effect of volume change can yield new results. Efforts already exist to construct a microstructural selection map from the results obtained in the drop tube and levitation experiments[11]. We shall now discuss some of the issues of microstructure development under reduced gravity conditions.

Dendritic growth

The theories of the dendritic growth assume diffusive mass transport at the solid–liquid interface. In a classic experiment, Glaksman et al.[12] have demonstrated the significant role of the gravity-driven convective flow in the growth of dendrites. These investigators have grown dendrites at different orientations with respect to the gravity vector and have shown that the growth is maximum when the dendrites are aligned along the gravity vector. This effect is significant only at low undercooling. Further, the evolution of side branches was found to depend very sensitively on the 'g' vector. Often the side branches were found to be asymmetric and to have large branches towards the direction of the gravity-induced flow[13,14]. The constant r^2v, where r is the tip radius of the dendrite and v is the velocity of the dendrite has been found to vary in the presence of fluid flow. Thus, microgravity provides excellent opportunities for carrying out critical experiments on dendritic growth. The first significant space-borne experiment in this direction was Isothermal Dendritic Growth Experiments (IDGE) as part of the United States Microgravity Payload (USMP2) launched by NASA in 1994. The programme is the culmination of ten years of efforts costing approximately $12 million. The results of the experiments confirm the significant effect of convection in the terrestrial experiments. The growth velocity can increase by a factor of 2 at low undercooling. The microgravity experiment show a much better match with that predicted by the diffusional dendritic growth theory[15]. However, the issue of solute partitioning influencing the dendritic growth is still open. Further experiments were carried out on the dendritic growth of Ni with 0.6% C in MSL1 in 1997, which also highlighted the effect of solute partitioning on the dendritic growth[16].

Micro and macro-segregation

The issue of segregation in solidified structure is of crucial importance in determining the properties of the solidified products. Considerable attention has been given to understand the origin of segregation during solidification processing. From these studies, it is clear that convection plays a crucial role in determining the extent and nature of the segregation. The possibility of microgravity experiments has given a fillip to these activities. In Box 1, S. N. Ojha details some of the issues and experimental efforts in this direction.

Eutectic solidification

Like dendritic growth, the utilization of microgravity is also expected to provide new insight to eutectic growth. The coupled growth of two phases during the eutectic growth is due to the lateral coupling of the diffusion field of the two growing phases. Thus, the gravity-driven fluid flow, which can change the effective diffusion, is expected to play a role in the growth process in the earth. The established theory of eutectic growth, however, considers only the diffusive coupling and ignores the convective flow. One of the important conclusions of this theory due to Jackson and Hunt is a relation between growth velocity (V) and eutectic spacing (λ) given by

$$\lambda^2 V = \text{constant.}$$

The influence of convective flow on this relation suggests a deviation. One often observes a larger spacing. A modification of this has been suggested where the flow velocity gradient at the interface has been incorporated[17]. The modified relation is given by

$$\lambda_\varepsilon^2 V = (\lambda^2 V)_o/(1 - (B'G_u\lambda_\varepsilon^2/D),$$

where G_u is the flow velocity gradient at the interface, B' a dimensionless constant and $(\lambda^2 V)$ is the Jackson–Hunt constant.

In a large number of experiments, the microgravity processing yields a finer eutectic spacing consistent with the prediction of higher diffusivity in the earth due to convection. However, there are examples of opposite behaviour. There are attempts to rationalize these observations[18]. Reliable long-term experiments in the space platform are yet to be performed, which can yield new information. Experiments on spacelab-1 on AgCu alloy also suggest improvement of the regularity and perfection of the eutectic compared to that grown in the earth.

Microgravity processing of immiscible alloys

From the beginning of the space programme, a large fraction of the microgravity materials science experiments focused on phase separation and monotectic growth in immiscible systems. Both convection and sedimentation play an important role during solidification processing of such alloys. Microgravity processing was expected to produce a fine and uniform two-phase dispersion of the

immiscible components. The experimental results have yielded mixed results. In some of the drop tube-processed samples, fine dispersions of the second phase have indeed been obtained. Some of the works have also highlighted the role of wetting by the crucible in determining the micro-structure. However, the recent long-term experiments in the space platform belied this expectation. Obviously, factors like differential contraction due to phase sepa-ration, which may lead to void nucleation and flow asso-ciated with the density difference may play an important role in addition for the microstructure development under the microgravity conditions. The immiscible alloy experi-ments also highlight the phenomenon of wetting under the microgravity conditions.

Other opportunities

In recent times the microgravity materials experiments have covered a large number of fields. The issues dis-cussed in the present article are only some representative of these samples. There exist considerable efforts in

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Box 1. Macro segregation during dendritic solidification

The microstructure developed during solidification of a casting influences its mechanical properties. Rejection of solutes during freezing of alloys results in constitutional supercooling of the melt ahead of the solid–liquid interface, which subsequently governs the formation of cellular and dendritic morpho-logy of the primary phase. The solute build-up in the interdendritic or intercellular regions and also in the melt ahead of the solid–liquid interface causes thermo-solutal convection and resultant macro seg-regation in the solid. This effect also influences the primary spacing of cells and dendrites. In case the density of the solute elements is less than that of the solvent, the convection of the melt causes longi-tudinal macro segregation and formation of channel segregates or freckles in the cast alloy[1]. Alterna-tively, if the solutes rejected ahead of the interface have density higher than that of the solvent metal, then the macro segregation would occur in the transverse direction and causing stifling of dendrites[2]. In both the cases the primary dendrite arms spacing has been reported to be different from that predicted based on theoretical analytical models of solidification. The primary spacing shows a systematic decrease with increasing convection. The concentration gradient consequently existing in the liquid adjacent to the mushy zone and within the mushy zone is affected. Such convection has been shown to be responsible for composition variation of the alloy observed in the casting and also during directional solidification. Analytical models basically consider diffusive mode of solute transport, whereas thermo-solutal convection is inevitable in terrestrial experimental conditions.

A recent investigation on directional solidification experiments of Pb–Sb alloy carried out over growth rates varying from 0.8 to 30 μs^{-1} in a positive temperature gradient of I40 kcm^{-1} provides a better insight into the role of convection on cellular–dendritic transition and resultant macro segregation in binary alloys[3,4]. The microstructural examination of a Pb–6Sb alloy indicates cellular to dendritic transition at a growth rate of 1.5 μs^{-1}. The primary arms spacing is observed to increase with growth rate in the cellular solidification regime, but it continuously decreases with growth rate in growth regime of dendritic solidification. The maximum primary arms spacing occurs at the growth rate corresponding to cellular to dendritic transition. Chemical analysis data reveal large macro segregation in the longitudinal section of the alloy. A decrease in growth rate results in an increased degree of macro segregation. A parameter, K_c, as effective partition coefficient represents the extent of convection in the melt and consequent degree of macro segregation of the alloy with varying growth rates even for solidification with cellular–dendritic interface, as it has been earlier used for the planar front solidification condition. Microgravity experiments are suggested to eliminate the effect of thermo-solutal convection on primary spacing of cells or dendrites.

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Box 2.   Foam in space

Polyurethane foam was produced in low gravity for the first time around 1995. The cause and distribution of different void or pore sizes are elucidated from a direct comparison of unit-gravity and low-gravity samples. Low gravity is found to increase the pore roundness by 17% and reduce the void size by 50%. The standard deviation for pores becomes narrower (a more homogeneous foam is produced) in low gravity. Both a Gaussian and a Weibull model fail to describe the statistical distribution of void areas, and hence the governing dynamics do not combine small voids in either a uniform or a dependent fashion to make larger voids. Instead, the void areas follow an exponential law, which effectively randomizes the production of void sizes in a non-dependent fashion, consistent more with single nucleation than with multiple or combining events.

NASA hopes to make a transparent solid in space. NASA thinks it has the potential to change the way your house is built. How? With an almost weightless solid that is an amazing insulator. The substance is called Aerogel, and nicknamed 'frozen smoke'. 'It's only three times denser than air – three times heavier than air – so we're almost close to a free-floating solid', said NASA research scientist David Noever. 'A block the size of a human of Aerogel would weigh one pound.' It is strong too – that human-sized block would be able to support half a ton, the weight of a small car. And due to its intricate, foamy composition of silicon and air, Aerogel is a highly-rated insulator. Developed more than 60 years ago, Aerogel has a smoky appearance because some of the bubbles are different sizes.

But experiments on suborbital rockets show that weightlessness can make it easier to form a more uniform product. With all the air bubbles the same size, the gel becomes transparent. 'From a single pane of Aerogel you might have the equivalent insulation of 20 to 30 glass window panes', Noever said. 'So you're looking at, in theory at least, heating the house with a candle.' Researchers at NASA's Marshall Space Flight Center are preparing a special experiment to be flown on the shuttle *Discovery*. They want to see if astronauts can make a clear form of the substance. 'I believe Aerogel will be a material that will be used in the 21st century almost as much as plastic is being used today', said NASA research scientist Laurent Sibille. 'Can you imagine your world without plastic?' The experiment was set for December, but scientists say it could be some time before the arrival of this airy substance in homes. Also it is proposed to prepare Aerogel microspheres in space.

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growing large complex crystals and in particular those which contain elements with large density differences. Space has definite advantage in controlling the stoichiometry of these crystals. Almost all the space agencies have major programmes on crystal growth. In recent times, several experiments have highlighted the advantage of space experiments in understanding the problem of sintering. The short article by A. Tiwari in this special section discusses some of the issues. The reduced gravity environment also provides a great opportunity for designing new materials. An example of this is foam, which has attracted a great deal of attention in recent times. Some of the work on foams and the possibilities are described in Box 2 by B. K. Dhindaw. Lastly, space, in addition to reduced gravity, also provides a high vacuum environment. This often poses potential challenge to materials, in particular, for electric application. Considerable research has been directed to the problem of dielectric breakdown in space. Some of these issues are discussed in a short article by T. Asokan.

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