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MATERIAL RESEARCH  IN MICROGRAVITY


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16. Abstract

A popular discussion is given of microgravity
effects in engineering and medicine gained
from Skylab experience. Areas covered
include crystal growing, liquid surface
properties, diffusion, ferromagnetism,
and emulsions.

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Zero gravity, a peculiar physical experience for astronauts, also
forces the physician and engineers to rethinking:  In the prepartion
and support of the Spacelab activities they recognize a large number
of effects, which are covered to a large extent under terrestrial
conditions.


by Dieter Langbein


Possibilities for material research under microgravity conditions
(zero gravity) have been existing to an increasing degree since about
the middle of the Seventies.  In 1973 a memorandum of understanding
was signed between the American NASA (National Aeronautics and Space
Administration) and the ESRO (European Space Research Organization)
with at that time 9 member states, the predecessor of the ESA
(European Space Agency).  It contains the agreement concerning
the development of the Spacelab and its integration into the American
shuttle system (the space vehicles Columbia, Challenger, and Atlantis).

In the same year, several materials-science related experiments
were flown successfully in the American Skylab program.  In the
following year additional experiments were carried out in the
American-Soviet Apollo-Soyuz program.

Then the ESRO and the Federal Ministry for Research and
Technology (BMFT) announced the opportunity to take part in the
flight in the first common Spacelab and issued an invitation to
submit proposals for experiments.  Originally the third quarter of
1980 had been planned as the starting date for the first Spacelab.

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* Numbers in margin refer to original pagination in German text.

More than one hundred proposals for experiments in the area of materials research were submitted; the certification panels of the BMFT and the peer groups of the ESA selected from this, forty experiments.

For the preparation and support of the Spacelab activities, there then followed, within a brief time, different airplane-, rocket-, and payload programs to be carried along, which shall be discussed in more detail in the following. Already the first investigations in these add-on programs showed that application-technological results could not be expected so quickly and achieved in their full scope, although a few experiments had unexpectedly great success.

The result was a rethinking to the principle-oriented investigations. The success of a few individual experiments as well as the failure of others can always be attributed to a large number of effects, which under terrestrial conditions are largely camouflaged by gravity. These effects were investigated in detail in the time that followed, experimentally as well as theoretically.

Terrestrial gravity and microgravity

All material-science experiments in Spacelab and in the other flight participation opportunities essentially made use of microgravity. The vacuum of space at the temperatures prevailing there can be reproduced, at lower cost also, in earth laboratories; however, microgravity can be achieved for longer periods of time only in the space laboratory.

In this connection microgravity does not mean that the attraction of the earth has decayed to zero because of the greater distance from the center of the earth. At the altitudes customary for space flight orbits of about 300 kilometers, the gravity of the earth still has a value of 90% of the terrestrial value. Rather, microgravity means that all masses or all objects of the system

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are subjected to the same acceleration and can follow it without interference from one another. An astronaut is subjected to the same acceleration as a body flying in space. Therefore he can move in it back and forth nearly gravity-free.

In a water tank on earth, objects with lower specific gravity than water rise to the top. Objects with a specific gravity greater /30 than water sink to the bottom. This is also true for warmer, sometimes of lower, specific gravity and colder, sometimes of greater specific gravity water. If, for instance, the water on the right side of the container is warmer than on the left side, a flow is developed which, on the right, is directed toward the top and on the left toward the bottom. This flow, which correspondingly also appears as wind in the atmosphere, is designated as natural or free convection.

Fig. 1: * Many physical processes which are of interest in materials research, are affected or even camouflaged in terrestrial laboratories. The following figures show this for an experiment about boundary surface convection: separation of the system methanol/cyclohexane under terrestrial conditions (left) and under microgravity in the German rocket program TEXUS (right). The test cell was always heated to 50° Celsius up to complete mixing of both liquids and subsequently – for the case of the TEXUS experiment after rocket launch and onset of microgravity – were cooled from the underside to 10° Celsius. The inserted numbers always denote the time from the start of the separation phase in seconds. In both cases one can first observe a fog formation on the cooled side. The fog front moves slowly upward; it shows a temperature of about 32° Celsius. Behind the fog front the liquid gradually starts to clear up. Simultaneously a few larger cyclohexane drops rise. In the terrestrial comparison experiment, the drops penetrate through the fog front without any noticeable change in velocity. Under microgravity, on the other hand, they quickly become stationary at the fog front. The velocity with which they move toward the fog front is nearly double that under the effect of gravity at the surface of the earth, although under terrestrial conditions buoyancy and boundary surface convection act at the same time and in the same direction.

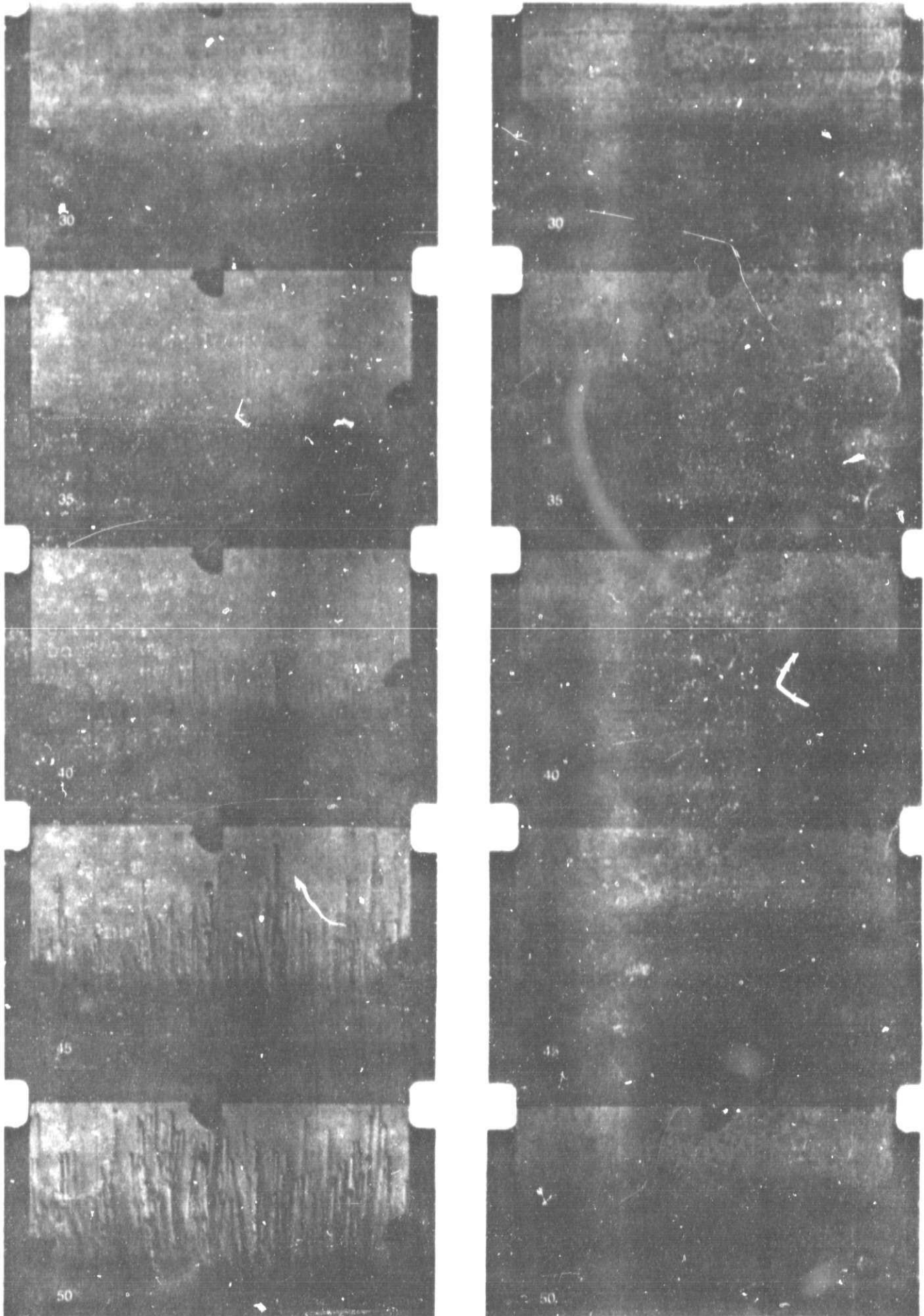*Translator note: Fig. is in color which is reproduced poorly. Please see Foreign Text.

Figure 1

A limited simulation of microgravity is possible by means of bodies or objects of equal specific density. A body with the same specific gravity as water can float in a water tank at any height. However, he experiences at every height a different pressure, as can be noted by everyone who dives to a depth of only a few meters. Therefore by the use of bodies of equal specific density it is not possible to simulate processes which depend in any way on pressure.

Therefore new application-oriented results and new knowledge concerning their physical principles can be expected for those material-scientific experiments under microgravity conditions, for which convection or pressure plays an essential role.

Convection always results in a strong mixture of the particular fluid systems so that other transport processes such as heat conduction or diffusion cannot be observed without interference. Thus a cup of coffee cools off considerably faster than a red pepper hull. The coffee served too hot is drinkable after five minutes at the latest, the red pepper hull served too hot has cooled off within fifteen minutes only insignificantly; in the cooling of the coffee convection plays an essential role, in the cooling of the red pepper hull it hardly has any effect.

The natural convection caused by gravity camouflages not only heat conduction and diffusion, but also other weaker convection contributions in liquids. It has been known for a for long time that convection can also be caused by temperature- or concentration differences along a liquid surface or along the boundary surface between two liquids.

However, quantitative investigations of this boundary surface convection are hardly possible because of the overriding contribution of natural convection in earth laboratories, under conditions of terrestrial gravity. This is true to an increased degree for potential contributions to convection, which are caused by the

growth of a solid phase, thus for example during the solidification
of a previously molten metal or the growing of a crystal from a
liquid phase.

Experiments to investigate such processes under microgravity
conditions must be set up for various durations. The cooling of a
metal melt solely by heat conduction requires only about ten seconds,
the developing or the decay of a uniform flow in a liquid already
about one minute, the cooling of a non-metallic and therefore poor
heat-conducting liquid about ten minutes. For diffusion investiga-
tions hours are generally required. For the growing of perfect
homogeneous single crystals of usable size days or even weeks must
often be scheduled.

Flight participation opportunities

Appropriate flight participation opportunities are offered for
such different experiment times: in airplanes, in rockets, in
Spacelab and probably starting in 1987 on the free-flying space
platform (figure 2). In addition drop towers can be used in the
USA, which make possible up to four seconds of free fall in vacuum
and thus conditions of microgravity.

Airplanes in free parabolic flight make possible for an ascension-
and drop height of 1000 to 2000 meters investigations with micro-
gravity times up to 40 seconds. The German program in this connection
operates under the name KURS (for: short-time experiments under
reduced gravity). In parabolic flights the results of drop tower
experiments can be checked under better conditions and in addition
investigations can be set up possibly for the undulation of liquid
surfaces; they are particularly important for functional tests
of instruments, which are planned for longer periods of time in space
mission and serve for the training of future astronauts, which must
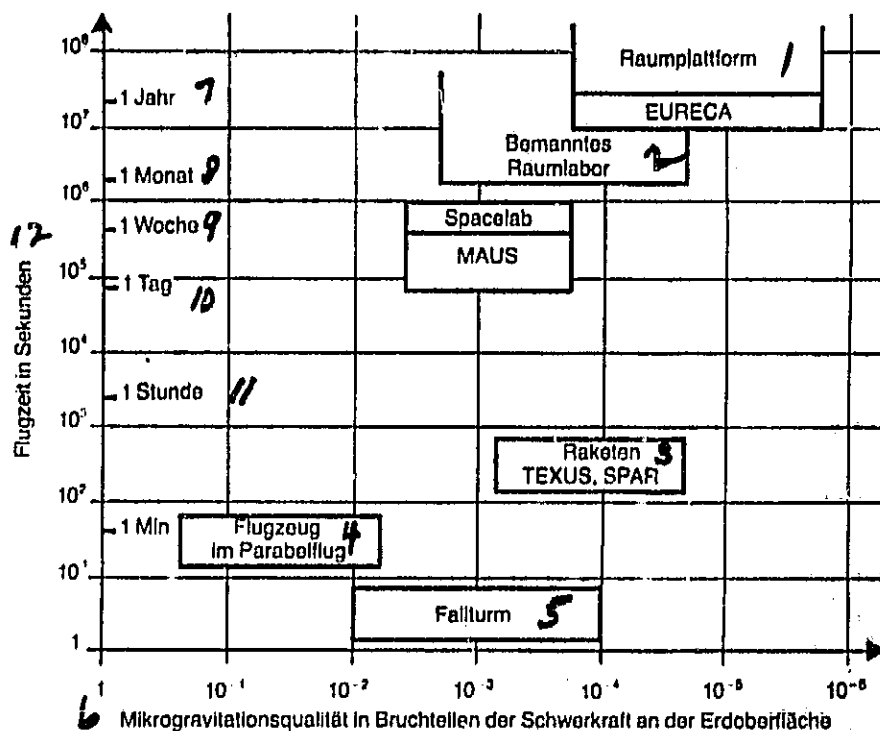be accustomed to zero gravity.

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Fig. 2: Overview of existing and planned flight-sharing opportunities, the there still occurring residual accelerations (so-called micro-gravity quality) and the each time planned or desired flight duration. Residual accelerations during a parabolic flight of an airplane result from air drag, which the pilot can compensate only to an insufficient extent; in Spacelab they are based on movements of the astronauts and of instruments which, in order to maintain the total impulse, effect a reverse thrust on the remaining Spacelab masses.

Key:  1. Space platform;  2. Manned space laboratory; 3. Rockets; 4. Airplane in parabolic flight;  5. Drop tower; 6. Microgravity quality in fractions of the gravity at the surface of the earth;  7. Year;  8. Month;  9. Week;  10. Day;  11. Hour; 12. Flight time in seconds.

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Great technological as well as scientific success has been
experienced until now by the German rocket program TEXUS (for:
technological experiments at zero gravity). Here the British
Skylark 7 rockets are equipped with automatically proceeding
experiments by the space flight company MBB-ERNO and are launched
in Kiruna in the north of Sweden. The TEXUS rockets reach an
altitude of about 250 kilometers, carry a payload of about 250
kilograms, and offer about six minutes of microgravity conditions.

TEXUS 1 was launched in December 1977; since 1981 two TEXUS         /31
rockets are being deployed between the end of April till the beginning
of May in each year. In the past year TEXUS 7 was flown successfully
on May 5 and TEXUS 8 on May 13. The payload always consists of
eight to ten different experiments. The present plan is layed out
till the year 1986.

The corresponding American rocket program SPAR (space processing
autonomous rockets) was started in December 1975. The payload of
the SPAR rockets always consists of about five to ten experiments.
Rockets are also used as carriers for the Japanese microgravity
program; until now six rockets were launched with varying success.

Within six minutes of microgravity conditions it is possible
among others
- to melt metal specimens in a defined manner and to again
  solidify them,
- to set up different liquid surfaces and to investigate their
  undulations,
- to generate temperature differences in liquids and thus
  observe the boundary layer convections,
- to bring liquids partially to boiling and to study the
  movement of the developing bubbles or even
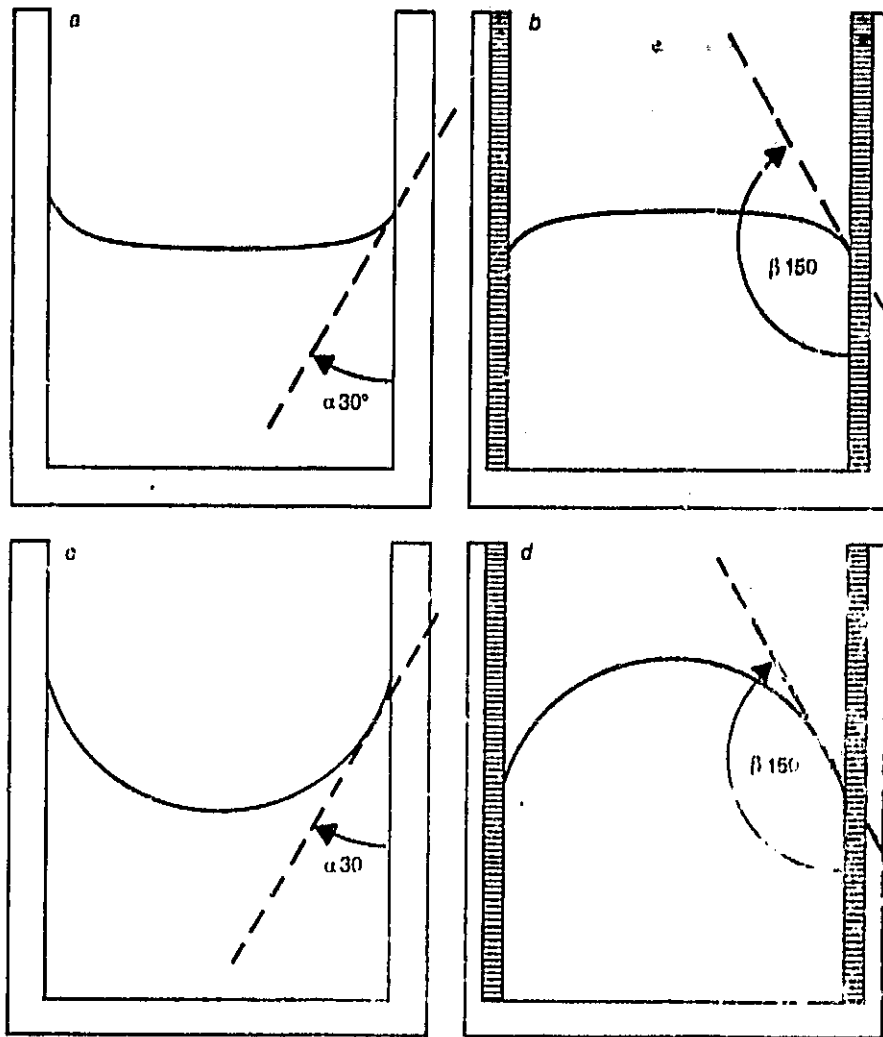- to grow fast-growing single crystals, for instance those
  of silicon.

Figure 3. Schematic representation of the boundary surface shapes, typical for capillarity, of a liquid in a vessel under terrestrial conditions (above) as well as under microgravity (below), for good wetting between liquid and vessel (always left) and poor wetting (always right) - possibly by Teflon coating of the vessel wall.

In Spacelab itself just as in the preceding Skylab missions and in the Soviet Soyuz program, days are available for experimentation time. The first Spacelab flight under the project direction of the ESA took place toward the end of November till the beginning of December of the past year. In the summer of 1985 a second launch of the Spacelab is to follow under German project direction.

During the experimental time of days most of the questions of liquid physics and solidification of metals and metal alloys can be investigated. Even more precipitated single crystals several millimeters in length can be grown; however, at present different crystals are still grown in succession in the same apparatus so that experiments, instead of days, are always limited to only hours.

The experimental installations in Spacelab are compiled into racks which are connected to the central current supply, data storage, and data processing. The so-called materials laboratory encompasses different heating installations for temperatures up to $1500^{\circ}$ Celsius for crystal growing and for melting of metal alloys and glasses. The materials laboratory also includes a fluid physics module for the investigations of liquid surfaces and flows. In a second rack - the process chamber being developed at present - different installations for experiments of liquid physics are assembled. Observations are made by means of a holographic method. A third rack, MEDEA (material scientific experiments double rack for experiment module and installations) will contain a gradient oven with quenching equipment, in which hot metallic specimens can be solidified within a few seconds so that the state obtained under microgravity conditions can be frozen. Also housed in MEDEA is the mirror heating installation ELLI, a rotation ellipsoid with a heating lamp at one focal point; on the other one the sample to be investigated is located.

The Spacelab missions always require a special flight of the space transporter. On the other hand, for the add-on payloads MAUS (for: material scientific autonomous experiments under zero gravity) or GAS (get away specials) we are dealing with single experiments, whose control and supply must be assured internally. Therefore such payloads can be carried along on nearly every space transporter flight, which makes possible a very flexible schedule planning.

The further planning of the ESA provides for 1987 the flight of a retrievable platform EURECA (European Retrievable Carrier). For the nineties the placing of a permanent manned space laboratory under the name Columbus is being considered. The collection of ideas for a European and in particular German participation started in December 1982. One hopes that the principles of the processes taking place under microgravity conditions will have been researched until then to an extent that the growing of especially perfect single crystals, the production of metal alloys not producible on  /32 earth as well as the production of especially pure pharmaceuticals will be possible.

Capillarity

The material-scientific areas for which one expects significant conclusions from experiments under micrographic conditions, will be summarized in the following in six topic areas. The sequence of these topic areas is chosen as far as possible so that the effects of the first ones on those to be treated later are greater than vice versa. Let us begin with the topic of capillarity.

We are used to the fact that the liquid in a glass has a largely flat surface. However, the surface is curved at the edge slightly upward (figure 3a). The liquid rises at the edge although in this way potential energy must be applied against gravity. This energy is obtained by the additional wetting of the glass;

the liquid has a high adhesion.  If instead of the normal glass a
vessel coated with teflon is used, the liquid surface at the edge
is curved downward (figure 3b).  Teflon has a poor wetting capability
for most liquids so that they try to obtain a distance as large as
possible from the edge.  In the first case we speak of wetting
between the liquid and solid material (wetting angle smaller than
$90^{o}$), in the second case of non-wetting (wetting angle greater
than $90^{o}$).

These two forms of liquid surfaces change considerably under
microgravity conditions.  However, the shape is determined just as
before by an energy equilibrium.  But while under terrestrial
conditions the equilibrium between potential energy and wetting
energy is the governing factor, the equilibrium between surface
energy and wetting energy plays the deciding role under micro-
gravity conditions.  The surface takes on that shape which represents
a minimum of surface energy for a specified wetting angle:   it
rises during wetting at the solid edges (figure 3c); for non-wetting
(figure 3d) it tries to get away from the solid edges.  In both
cases it assumes a minimum surface for a specified volume - thus
the spherical shape.

A positive curvature pressure is connected with the convex
surface shape, curved upward on the inside.  The attempt of the liquid
to decrease its surface, thus to contract like a rubber membrane,
increases the pressure in the interior.  Opposed to that there
develops, if the liquid at the edge is pulled upward and if the
surface altogether takes on a concave shape, a negative curvature
pressure.  The pressure in the interior of the liquid is then lower
than in the gas space lying directly above.  This brings about in
narrow capillaries, if the wetting is large compared to the surface,
a high negative curvature pressure:  The liquid rises in the
capillary as long as this negative curvature pressure is compensated
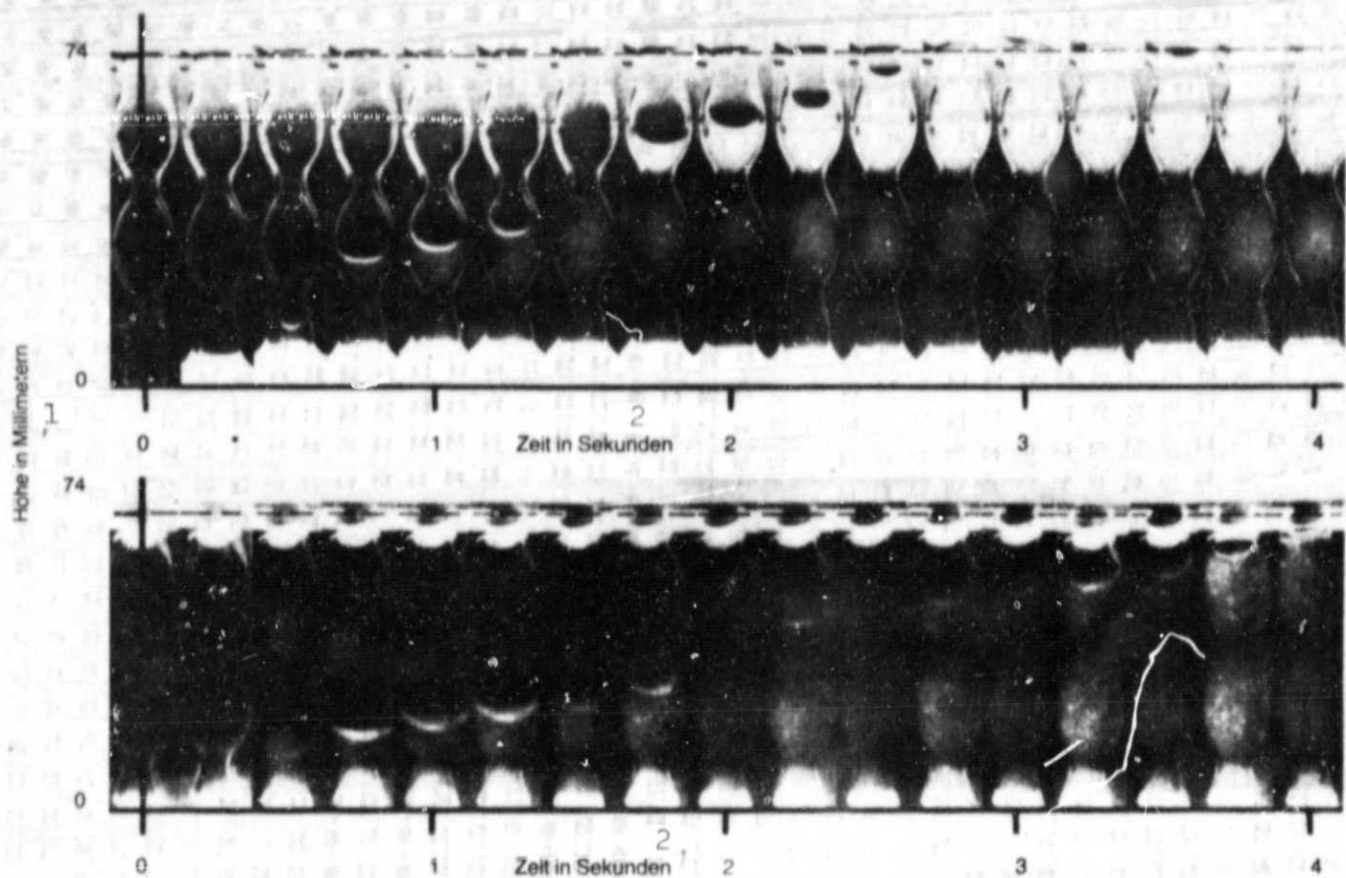by the hydrostatic pressure decreasing in an upward direction.

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Figure 4: Filling of a capillary with different cross-sections.
In the front case (above) microgravity is only simulated - by the
use of two liquids of equal density, here water and a mixture of
cyclohexane and carbon tetrachloride. In the second case (below)
the same capillary is filled with water under microgravity (during
the TEXUS 3 mission). The kinetics of the wetting prevent in both
cases the formation of a spherical boundary surface. The filling
speed under microgravity is slower because of poor wetting. The
photographs were made by Peter J. Sell of the Fraunhofer Institute
for Boundary Surface- and Bioprocess-technology in Stuttgart.

Key: 1. Height in millimeters     2. Time in seconds

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The hydrostatic pressure is the mentioned pressure increase with increasing depth of water. If the same capillary is filled with the same liquid under microgravity conditions, the wetting in the absence of potential energy and hydrostatic pressure provides that the liquid flows into the capillary as long as the liquid supply allows it (figure 4).

Figure 5 shows another example of the boundary surface shape changing under microgravity conditions. A prescribed liquid volume is introduced between two solid pistons through an opening in the lower piston. Under terrestrial conditions, a boundary surface curved at the top inward and at the bottom curved outward develops. On the other hand, a cylindrical boundary surface can be formed under microgravity conditions.

This offers the advantage of geometrical simplicity: The oscillations of a cylindrical liquid column and their instabilities can be calculated more simply than those of other surface shapes. Thus Lord Rayleigh had already predicted that a cylindrical liquid column always becomes unstable and pulls apart whenever its height is greater than its circumference (figure 6). We are dealing here with the same physical effect which allows the water stream flowing out of a faucet to be torn off.

A second essential advantage of working under microgravity conditions is the fact that edge effects can be minimized. Under terrestrial conditions, liquid columns of a few millimeters in height can barely be generated so that the wetting of the pistons by the liquid, which depends to a sensitive degree on its pretreatment, has a telling effect on all physical investigations. Opposed to that, the ratio of surface to edge can be increased appreciably under microgravity conditions.
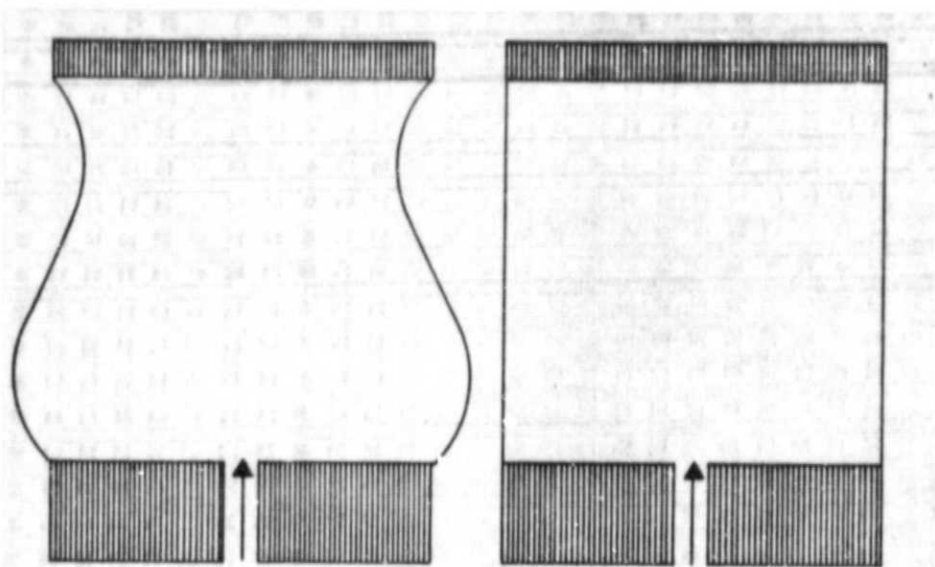
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Fig. 5: Boundary surface shapes of liquid column under terrestrial conditions (left) and under microgravity (right). The specified liquid volume was introduced between two rigid pistons through an opening in the lower piston. Under terrestrial conditions the liquid pressure increases from top to bottom so that along with the curvature pressure the curvature also increases from top to bottom. Under microgravity the pressure is constant over the entire liquid bridge. For a suitable choice of liquid volume and piston distance, a cylindrical boundary surface is formed.

Fig. 6: (p. 16) Tearing away of a cylindrical boundary surface whose height exceeds the circumference by 5 percent, in a computer simulation. Just as a pencil standing on its point topples at the least amount of shaking, thus the boundary surface collapses under the least disturbance (brown). First the boundary surface smoothens again; however, a permanent deformation develops which, during the first six seconds increases slowly, then very rapidly. Finally the deformation phases differ only by fiftieths of a second, and 7.46 seconds after the triggering shaking the boundary surface tears apart. After 7.47 seconds the deformation intersects itself, thus becomes physically non-sensical.

Key: 1. Ratio of height (H) and radius (r) of the liquid column
2. Time in seconds
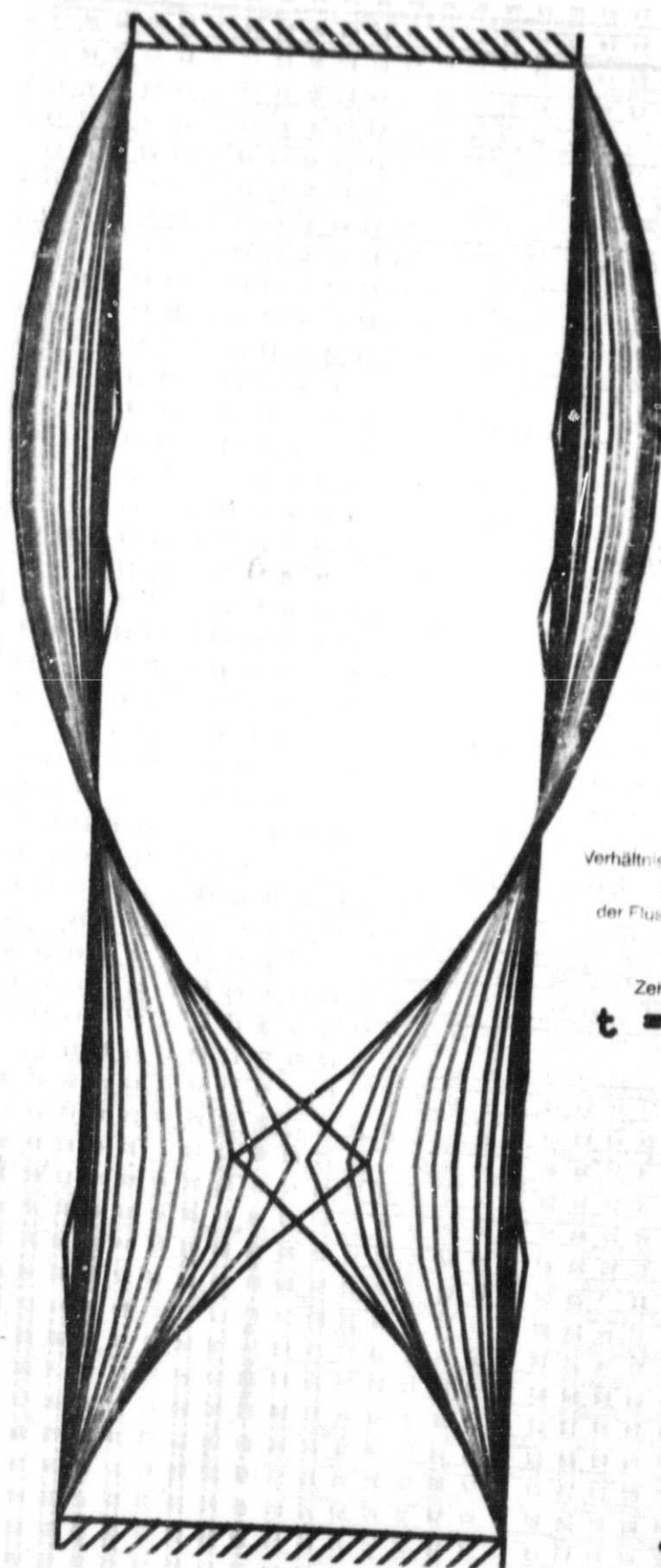
Verhältnis von Höhe (H)
und Radius (r)        ⟩ 1
der Flüssigkeitssäule
H = ∅ 1 a

Zeit in Sekunden  2

| t = | 0 |
| | 1-6 |
| | 6.5 |
| | 7.0 |
| | 7.2 |
| | 7.3 |
| | 7.4 |
| | 7.42 |
| | 7.44 |
| | 7.46 |
| | 7.47 |

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## Boundary layer convection

A phenomenon known from different terrestrial experiences,
which under microgravity conditions becomes the primary cause for
flows of liquids, is the already mentioned boundary layer convection.
One can easily observe on a burning candle that the wick residues,
which have fallen into the molten wax, move at the surface with
higher velocity toward the edge.

If until now the discussion was based on the fact that the
boundary layer energy affects the shape of the boundary layer to a
deciding degree, we shall now consider the effects of different
boundary layer energies at different locations of the boundary layer.
Such differences in boundary layer energy can be caused by temperature-
or concentration differences, but also by external electrical or
magnetic fields.

As a rule an increase in temperature brings about that two
liquids can mix better and that thus their mutual boundary layer
energy decreases. In the same way the boundary surface energy
of two liquids generally changes with the concentration of a third
liquid, which is dissolved in one or in both liquids.

A boundary surface area of high boundary surface energy tries
to draw liquids from areas of lower boundary surface energy. Or,
in order to return to the comparison of the rubber membrane, a
boundary surface area with a strong rubber membrane contracts at
the expense of areas with weak rubber membrane.

The wick residues in the wax of the burning candle therefore
quickly move toward the edge because the temperature of the liquid
wax is lower at the edge than in the center, and because the
boundary surface energy of the wax is thus higher at the edge than
in the center. The wick residues are carried along by the boundary
surface convection of the wax. The free convection acting in the

same direction is not enough to explain the high velocities.

A further example for boundary surface convection under terrestrial conditions is the creation of Benardic cells, which Manuel G. Velarde and Christiane Normand discussed in detail in this journal (September 1980): In a liquid with a free surface, which is heated from below, an unstable equilibrium sets in since the colder and thus heavier liquid layer is positioned above the warmer and thus the lighter liquid layer. The heavier regions tried to sink as soon as any disturbance (a shaking) changes the initially strictly horizontal layer. If the colder regions sink more rapidly as they pick up heat from the surroundings, regular convection rolls (Benard cells) are formed.

An additional example of surface convection can be observed in a brandy glass (if one is not in too great a hurry to drink it). One can observe that brandy always rises in the glass and then again runs down (see also "experiment of the month" in Spectrum der Wissen Schaft, July 1983). This is not a case here of perpetual motion, but rather a consequence of the evaporation of alcohol caused by temperature and concentration differences of brandy. In this way not only the wetting of the glass is changed but also boundary surface convection is generated.

For systematic investigations of the boundary surface convection under microgravity conditions, the rocket programs are particularly suited. Most of the flow-mechanical processes can be made stationary within six minutes. During the German TEXUS program, the boundary surface convection resulting from temperature differences was /34 primarily investigated, and with an arrangement as shown in figure 5, where the upper and the lower pistons were heated differently. Thus a constant temperature difference was maintained along the liquid bridge. A higher temperature on top produces a higher boundary surface energy at the lower edge of the liquid column; there develops, as in figure 7, a flow directed downward along the boundary surface.

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Just as the natural convection, the boundary surface convection
generates for higher flow velocities oscillations, periodic fluctuations
of the flow velocity. Whoever has observed during a rainstorm the
flow-off of water from a steep street, can observe that the waves
are formed with generally quite regular intervals. Wherever the
water layer becomes somewhat thicker as the result of a random
unevenness of the street, the friction resistance is decreased
considerably. The thicker layer drags additional water along with
it and thus stabilizes itself.

A corresponding fact applies for boundary surface convection.
If it is driven by a temperature rise along the boundary surface,
the convection generally reduces this temperature difference. If
we are dealing with a liquid of high conductivity and if the heat
supply via the solid piston is sufficient, temperature rise and
flow velocities are stabilized at a reduced level. However, if the
temperature difference is increased, the temperature equalization
becomes relatively unimportant: The flow reduces the temperature
rise considerably. In this way it reduces itself, and the temperature
rise can begin build up. Flow velocity and temperature start,
periodically - generally with a phase displacement - to oscillate.

Investigations of these oscillations under terrestrial conditions,
if free convection thus plays a part, and under microgravity conditions
in the TEXUS 3, 5, and 8 missions have shown that the frequency
of these oscillations increases with increasing temperature difference
between the wetted pistons and with decreasing size of the liquid
zone. For small sizes under terrestrial conditions as well as
under microgravity conditions it has a value of about 1 Hertz. We
are dealing essentially with a flow disturbance circulating about
the zone axis.

The goal of several research projects is to observe the
movements of drops of liquid B in liquid A as the result of

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boundary surface convection. Fluid B can also be a gas so that we then speak of bubble movement instead of drop movement.

From a pure energy standpoint this motion can be explained as follows: If the boundary surface energy of drop B in fluid A is different at different positions as the result of different temperatures or concentrations prevailing there, the drop will move with a gain in energy in the direction of decreasing boundary surface energy. On the other hand, the kinematic explanation of the drop motion states that the drop has different boundary surface energy at its front- and back side which corresponding to the comparison with the rubber membrane of different elasticity trigger a fluid flow in the direction of increasing boundary surface energy.

The attempts to investigate the droplet movement quantitatively under terrestrial conditions all failed till now because of the simultaneously present buoyance or sinking. Even if the densities of the fluids A and B are carefully equalized, it is not possible to maintain this equalization for different temperatures.

We proved the existence of boundary surface convection of drops during microgravity conditions in an experiment of the mission TEXUS 7 (figure 1), and namely with methanol (liquid A) and cyclohexane (liquid B). These liquids have - as numerous others - the property that they become completely miscible at elevated temperatures, but that they separate with decreasing temperature. This behavior is comparable to that of water vapor in air: At elevated temperatures the air can pick up a greater amount of water vapor (air humidity); however, during cooling the water vapor condenses to fine droplets. A typical example for this are morning fogs.

Investigations of heat transfer and bubble formation in different TEXUS missions can also be assigned to essential parts

of the topic boundary surface convection. Everybody knows that the steam bubbles, which develop on the bottom of a pot filled with boiling water rise as the result of buoyancy. The same is true for carbon dioxide bubbles which pearl upward in a glass filled with the popular beverage champagne.

What can now one expect from microgravity? Do the bubbles continue to adhere to the bottom? Do they migrate to the side as the result of boundary surface convection? Do they coalesce?

In mission TEXUS 3b the cooling medium 113 was made to boil by means of a heated wire. Stable bubble boiling set in (fig. 9). The bubbles moved in apparently random direction along the wire. Much points to the fact that boundary surface convection is responsible for this bubble motion. The heat flux into the liquid is almost equal to that under terrestrial conditions.

## Diffusion phenomena

A physical process generally proceeding very slowly is the diffusion of the molecules of a fluid B in a fluid A. As a rule one can figure that the concentration equalization over a distance of one centimeter requires several hours. Thus it is possible, for example, to layer water saturated with cooking salt and thus of higher specific gravity carefully below pure water in a cup. The cooking salt molecules then penetrate into the pure water only extremely slowly.

Just because diffusion proceeds very slowly in liquids, it is disturbed extremely sensitively by convective effects. If the temperature at the edge of a glass is higher only by a few tenths of a degree than in the center, a substantially larger portion of the molecules of liquid B is transported by natural convection than by diffusion. Therefore it is not possible to measure the diffusion

under terrestrial conditions in vessels having the size of cups. Conventionally thin capillaries are rather used in order to thus suppress the convection to a large extent.

However, this includes a second danger of erroneous measurement. The structure of a liquid - the statistical arrangement of the molecules - in the immediate surroundings of a boundary surface differs from the structure in the interior of the liquid. The average distance between the molecules can be increased or also decreased as the result of the changes in intermolecular attraction. Of two liquid components A and B one can be enriched on the solid wall; in particular the diffusion of this component along the wall can be favored compared to the diffusion in the interior. This then affects the measurement of diffusion in thin capillaries to a decisive degree: One measures the increased diffusion along the wall instead of the normal diffusion in the interior. This hypothesis is supported by the fact that the measured diffusion coefficient (the one generally observed) depends greatly on the diameter of the capillary used; this cannot be attributed solely to the always changed residual convection.

Compared to that, diffusion investigations under microgravity /35 conditions provide by far more favorable conditions. The convective portion of the transport of liquid B in liquid A is eliminated, thus one can work with vessels of suitable sizes. If one decreases the vessel dimensions schematically, one can also determine the presumed contribution of wall diffusion.

In addition microgravity offers the possibility to measure the diffusion exactly on non-level boundary surfaces. The working with level boundary surfaces is typical for the fact that our thinking is connected with natural convection.

Diffusion on non-level boundary surfaces no longer brings about exclusively vertical density differences, and thus under terrestrial

conditions convection also always occurs. Under microgravity
conditions those boundary surfaces are suited for diffusion investi-
gations, which exclude position-related enrichment of one component
at the boundary surface and thus boundary surface convection.
The optimum arrangement to this point of view is a spherical vessel
filled with liquid A, in whose center a drop of liquid B is located.

Diffusion investigations under terrestrial conditions then
become particularly questionable if secondary effects such as
Ostwald ripening are involved. This is the phenomenon where large
drops grow at the expense of small ones. This can be observed in
many areas of our lives.

The Ostwald ripening of drops is based on differences in vapor
pressure or on differences in solubility. Small water drops have a
higher vapor pressure than large drops. The molecules of the small
drop are hurled during their Brownian heat movement because of the
greater surface curvature more frequently into the surrounding air
space than the molecules of large drops. If the air humidity increased
in this way in the vicinity of the small drop arrives through
diffusion in the vicinity of large drop, the latter grows with
corresponding reduction of the air humidity.

The same process occurs if in a liquid A drops of a liquid B
are located, which are partially soluble in liquid A. Small drops
then effect a greater external concentration than large drops.
which thus grow at the expense of the smaller ones. Microgravity
conditions offer the possibility to distribute a larger number
of droplets of a liquid A in a liquid B statistically and to then
observe their Ostwald growth over several days without being influenced
by convection and buoyancy.                                    /36

A similar problem is diffusion at solidification fronts. For
instance, ice freezing from ocean water generally piles up considerable
amounts of dirt, but contains hardly any salt. It pushes the salt

content of the ocean water away during solidification. Similar
phenomena can be found in nearly all multi-component liquids. , .
composition of the solidifying phase differs from that of the liquid
phase so that one or several components are piled up in front of the
solidification front and are pushed away.

Since the solidifying phase freezes the particular concentration
at the phase boundary, the concentration pile-up and its removal
can be reconstructed through diffusion. Thus one can obtain detailed
conclusions concerning the diffusion, especially also concerning
a potentially changed diffusion in the proximity of the solidification
front as well as concerning the solidification speed as a function
of temperature and concentration.

For quick solidification one must figure on non-uniform removal
of the concentration pile-up and as a result with spatial and time
oscillations. An increase in different places of the pile-up can
change the growth rate and can trigger as the result of the volume
change always connected with solidification a local convection - a
so-called microconvection - at the solidification front. The
solidification front then loses its level shape and the solidifying
phase exhibits regions of different structure. In many cases
dendrite growth sets in; the most well known examples for this are
the frost pictures on windows in winter and the beautiful six-ray
snow crystals.

Since these effects are primarily affected by natural convection,
much information can be contained by comparison experiments in
microgravity conditions. Not only investigations of the growth rate
of ice from salt water, but also of the growth rate of aluminum from
aluminum/copper-melts are being prepared. The relation between
microconvection and structure formation is preferably investigated
with the organic substance succinonitrile which readily forms
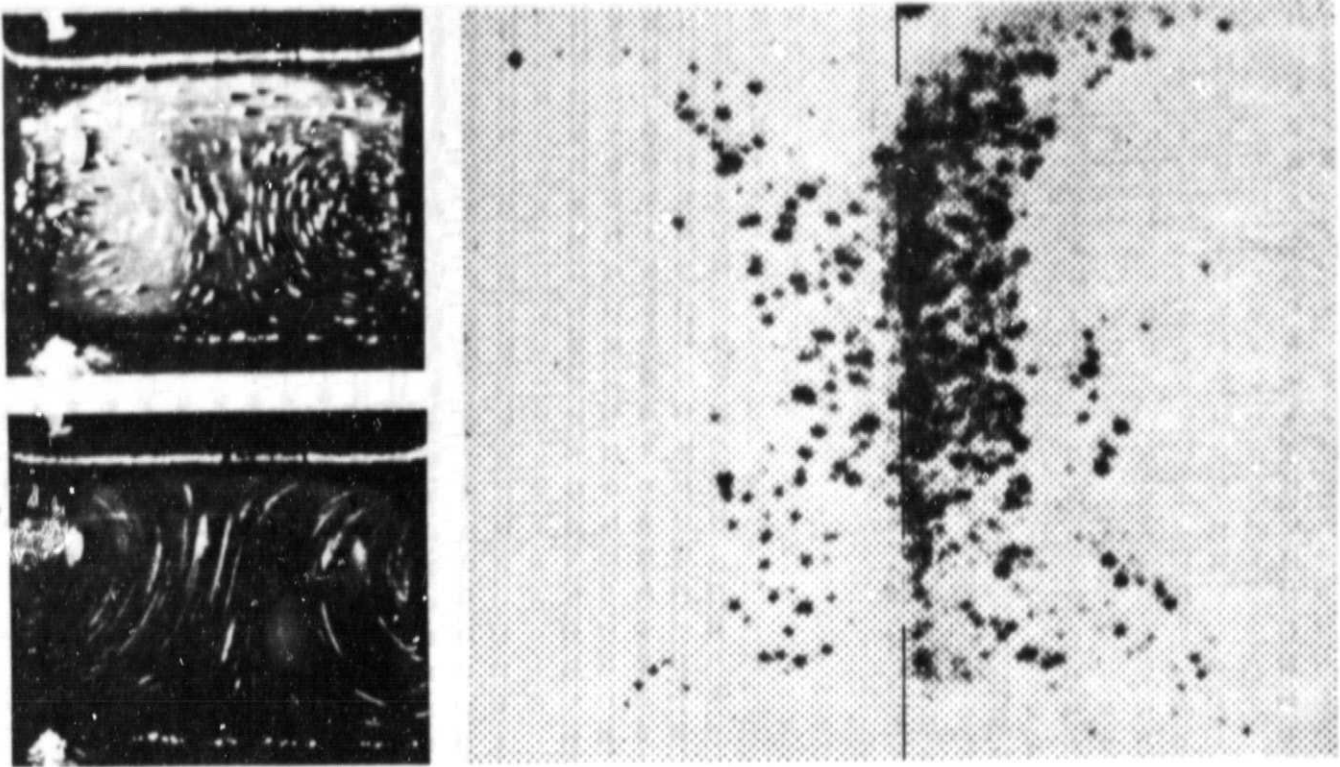dendrite.

Fig. 7: Natural and boundary surface convection in the liquid bridges heated on top. The first case is a sodium nitrate zone with pure surface (upper left). The boundary surface convection dominates the simultaneously present natural convection; the eddy center of the flow lies close to the surface. In the second case (lower left) the surface of the sodium nitrate zone was covered with a high-viscosity oil film so that the boundary surface convection is suppressed and the natural convection dominates. The eddy center has been displaced inward. The flow velocity has, especially along the surface, dropped noticeably. During the TEXUS 3 mission it was possible to investigate (right) pure boundary surface convection in a silicon-oil zone. The low density of the floating particles near the eddy center is noticeable. This can be explained by the initial adhesion of the floating particles to the end surfaces or by the centrifugal forces developing around the eddy centers. The two photographs of the sodium nitrate zone were made by Dietrich Schwabe of the University of Giessen; the photograph of the silicon-oil zone was made available by Chung-Hwan Chun of the DFVLR in Goettingen.

Critical phenomena

The topic critical phenomena spreads over several areas. Let
us remember the superconduction of numerous metals and also nonmetals
at very low temperatures, the disappearance of ferro-magnetism of iron
at high temperatures, and the critical point of liquids or gases,
which are to be chiefly discussed in the following.

All these phenomena are characterized by a rapid changeover
from one state to another or rapid generation of a new phase. Here
the quantities of state depend in the same manner on temperature.

It is known that the cooking time of many meals can be reduced
considerably with a pressure cooker. The pressure prevents the
normal evaporation of the water at 100 Celsius; the temperature in
the pressure cooker rises rapidly (however, the safety valve sets
limits for the impatience of the cook).

If water is heated in a safety vessel one can differentiate
between three cases. If the amount of water is small compared to the
total volume, the entire water evaporates. If the amount of water
is large compared to the total volume, it expands with considerable
increase in pressure; the initially present volume of steam disappears,
the steam is pushed back into the superheated water. Between the
two we have as boundary case a critical amount of water in which
water as well as steam are first present during heating. At a
temperature of $374^{\circ}$ Celsius - that is the critical temperature -
both phases mix completely, however. Then one can speak neither
of liquid nor of gas. The mixture requires a high addition of heat
of mixing: The heat capacity of the system approaches, as also
several other physical quantities, infinity at the critical temperature.

Nearly all liquids or gases have a corresponding critical
point, characterized by critical temperature and critical pressure.
The advantage of microgravity for investigations of the critical
point is the fact that equal pressure prevails in the entire

experimental volume. Thus the critical point can be approached experimentally considerably better and the relevant quantities can be measured more accurately.

The phase separation during cooling of a supercritical mixture below the critical temperature is to be observed by scattering of light as well as interferometrically. Other products are the measurement of the heat capacity and the compressibility.

Just how the critical point can actually be approached accurately depends to a decisive degree on the action of the liquid near the wall. This problem was already addressed in connection with wall diffusion. A smaller average distance between molecules near the wall always forms a seed cell for separation. Thus one can expect that the liquid phase first occurred near the wall.

A direct measurement of the excess of molecules near the wall is planned for the EURECA program. Two structurally identical test cells, one coated with graphite, are filled with sulfur hexafluoride and are connected via a differential pressure pickup. By means of gas addition from a supply vessel the same pressure is set up in both cells for a given temperature. Subsequently the temperature will be decreased in steps whereby as the result of the differences in excesses at the wall (as the result of differences in adsorption) a pressure difference arises between the two cells. The process is then repeated by renewed gas addition. The particular amount of gas required immediately provides an increase of the excess at the wall.

A critical point sets in not only between the liquid and the gaseous phase of the same substance, but also between two limitedly miscible liquids. Such a system is formed, for example, by methanol and cyclohexane with which in the TEXUS-7 mission the boundary surface convection of drops is to be proven (figure 1).

## Alloys with mixing gaps

Experiments for the production of finely distributed mixtures of metal pairs which cannot be alloyed were already adopted at an early time into the micrography program. It is generally known /38 that gold can be alloyed to a large extent with silver; many people wearing jewelry prefer 585 gold to 333 gold. In the same way silver can be mixed to a large extent with copper: One differentiates between 925 sterling silver, 800, 535, and 333 silver. Other much used alloys are brass (copper and zinc) and bronze (usually copper and tin).

Besides this there are metal pairs from which one expects advantageous technological properties which, however, cannot be alloyed in this way. For example, finely divided mixtures of aluminum and lead are to be used as material for sliding bearings. Mixtures of aluminum and indium serve as a model system, and mixtures of manganese and bismuth are valued because of their magnetic properties. However, it is not possible to produce these mixtures from powder even in costly pressed- and sinter operations with the required stability. /39

Fig. 8: Boundary surface convection under terrestrial conditions (above) and under microgravity (TEXUS 3b mission, below) observed by Schlieren optics in experiments by Rolf Brueckner of the TU Berlin. The systems consist of a mixture of toluene and acetone (above) and water (below). At the start of the test the boundary surface lies crossways in the center. In both cases convective eddies develop which arise during the diffusion of acetone from the mixture into the water as the result of concentration differences along the boundary surface. Under microgravity these eddies expand greatly; under terrestrial conditions, on the other hand, gravity prevents their full formation.
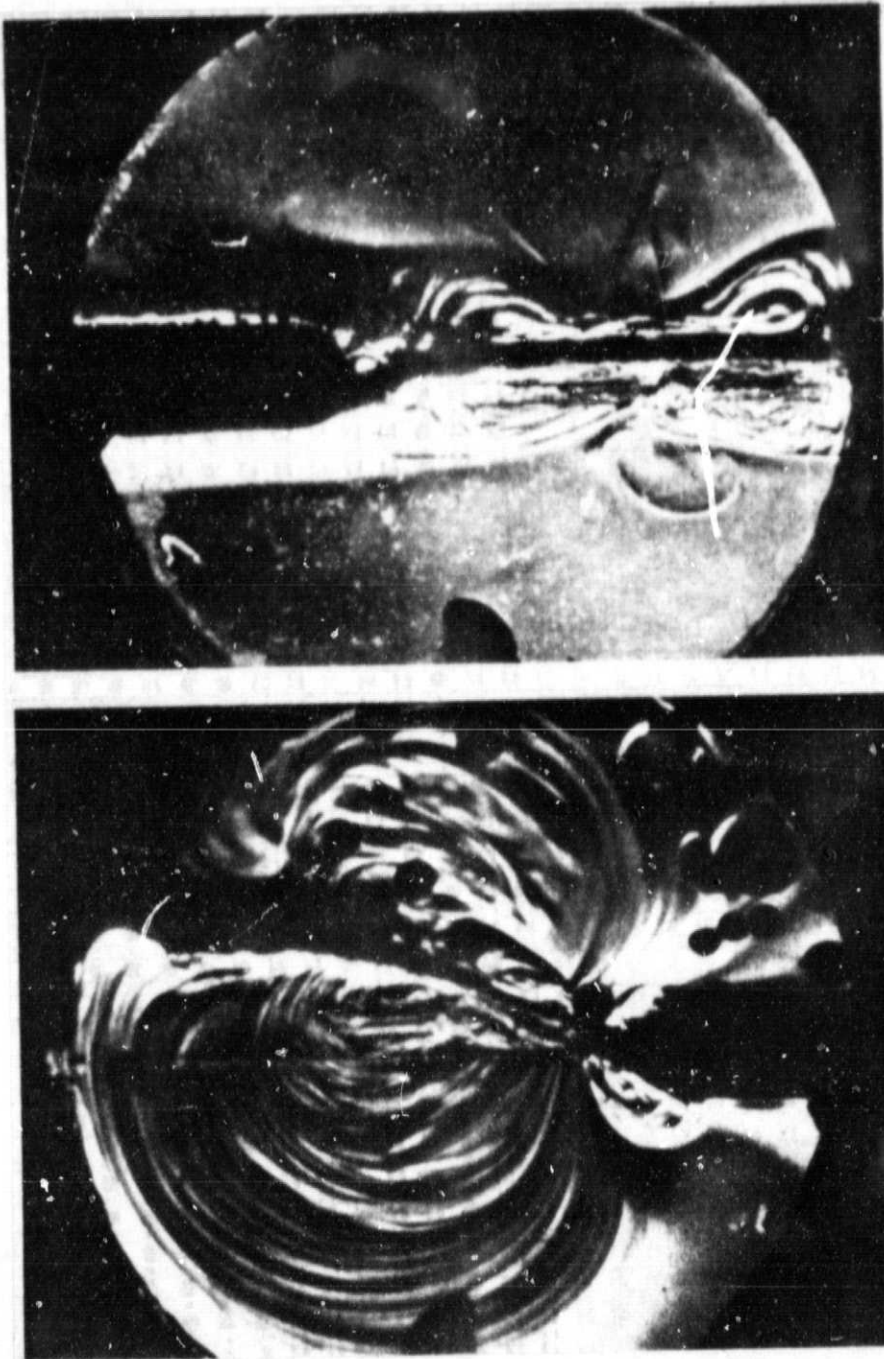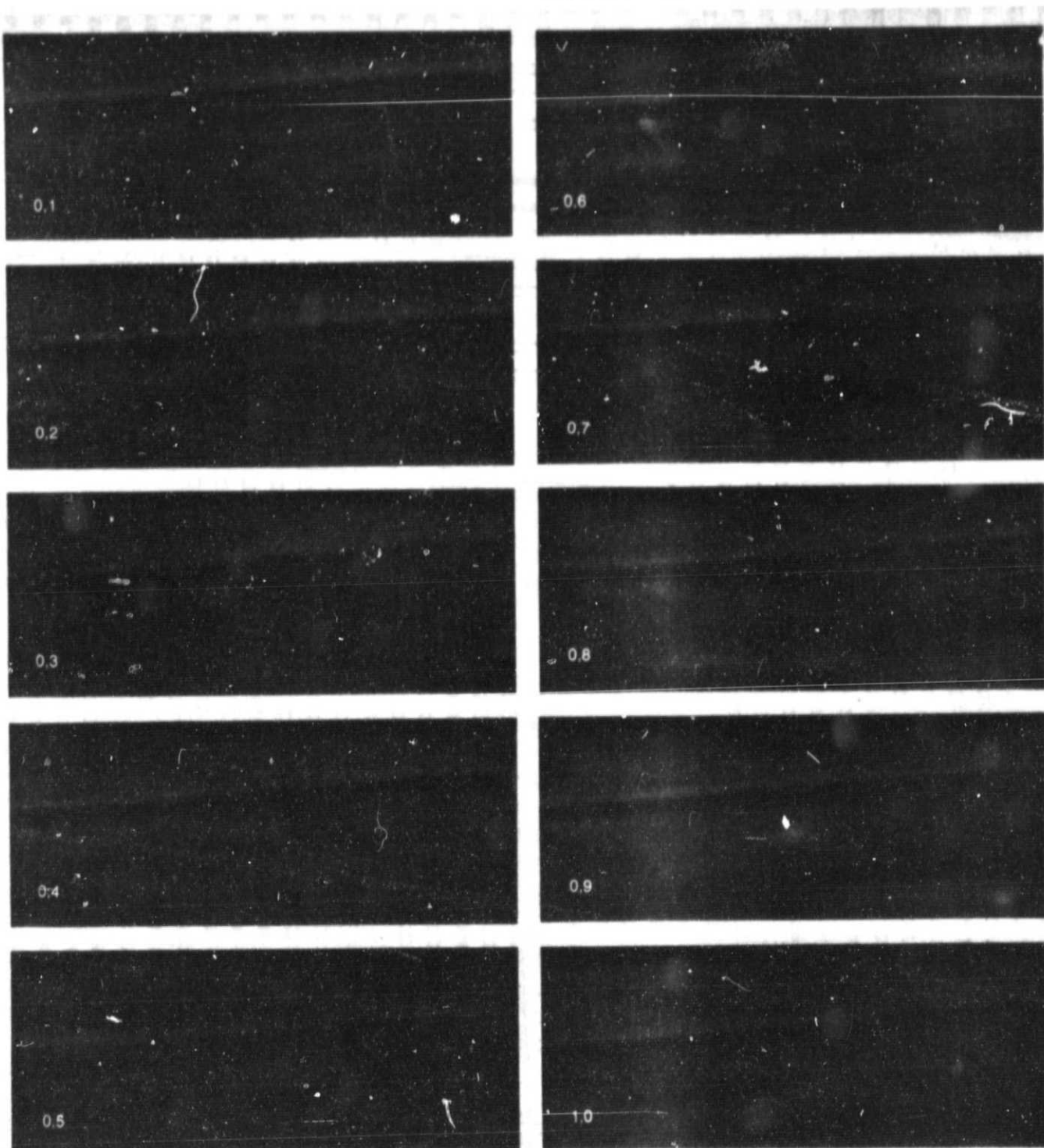
Figure 8

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Fig. 9: As is known, steam bubbles rise in boiling water; in a glass
of champagne carbon dioxide bubbles up. However, what happens under
corresponding processes under microgravity when there is no top
and bottom? Investigations about heat transfer and bubble formation
were set up in different TEXUS missions. There at intervals of tenths
of seconds (printed numbers) phases of an experiment by Johannes Straub
and Armin Weinzierl of the Technical University Munich were photo-
graphed during the TEXUS 3b mission: a heating wire brings the
cooling fluid, just barely below its boiling temperature, to a boil.
Bubbles develop which migrate in apparently random direction along
the wire and then shrink again. Presumably the boundary surface
convection causes the movement of the bubbles.

All these metal pairs have one thing in common: Although the melts are miscible at very high temperatures, they separate again during cooling before solidification. The metal melts are limitedly miscible liquids with similar properties as the mentioned transparent limitedly miscible liquid pairs, but differ from them by their higher critical temperatures, by a better heat conductivity, and by the absence of optical transparency; one cannot see immediately which physical processes occur during the cooling and solidification.

If such metal melts are cooled under terrestrial conditions, the heavier component sinks after the phase separation. As tests in SPAR- and in the TEXUS program showed, a nearly complete seperation of both components also occurred under microgravity conditions - however, with a quite different distribution (fig. 10).

Apparently the described droplet motion as the result of boundary surface convection plays a substantial part in the separation. Since a molten metal sample cools from the outside in, the temperature increases in the melt toward the center. Drops of the components present at a lower concentration migrate in the direction of dropping boundary surface energy, that is toward the inside.

One can well identify a dozen of additional physical effects which take a part in the separation under microgravity conditions. Here the following contributions are important: Structure and composition of a liquid are, as mentioned, different at the boundary surfaces and in the interior. If at the container walls the component is already enriched as the result of good wetting, then the formation of separations of this component is favored there at the same time. A second contribution of differing boundary surface energies of the component is the following: A well-wetting precipitation, which accidently touches the container wall, spreads there and thus causes a local flow of the melt. The same is true for accidental contact and combination of two precipitations of one and the same component in the interior of the melt.

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An essential contribution toward separation is also provided
by the pushing aside of a solidification front. One must again
differentiate between two cases. For one, individual molecules
or atoms can be pushed away, which do not fit into the structure
of the solidifying phase; the accordingly not-inserted component is
- as discussed - piled up at the solidification front. For another,
already produced precipitations or solid foreign particles are also
pushed away. A trend for the incorporation of such particles exists
whenever this is connected with the gain in boundary surface energy.
Therefore precipitates with poor wetting of the solid phase are
incorporated with lower probability than precipitates with good
wetting.

Melts and solidification always proceed with a volume jump.
If water is frozen in a completely filled closed bottle, the bottle
- since water expands upon cooling below 4 degrees Celsius - will
burst. The same is also true if, although a gas volume is present,
the water freezes from top to bottom so that the gas volume can
no longer bring about a volume equalization.

Ice shrinks upon melting. On the other hand, most metals expand
upon melting, aluminum for example by about 6 percent (however, a
few such as antimony and bismuth shrink upon melting just like water).
Expansions during melting or shrinking during solidification favor
the inclusion of precipitates or foreign particles; expansion during
solidification correspondingly favors their removal.

For the further clarification of the separation mechanisms,
systematic theories of experiments in the systems zinc/lead and
zinc/bismuth were conducted in the first Spacelab mission. Lead
and bismuth are metals with opposite volume jumps during melting:
Lead expands, bismuth shrinks so that results concerning the
effects of the volume jump on the separation could be expected.
Since in both systems zinc is the first solidifying component,
the solidification temperatures are always close to one another. /40
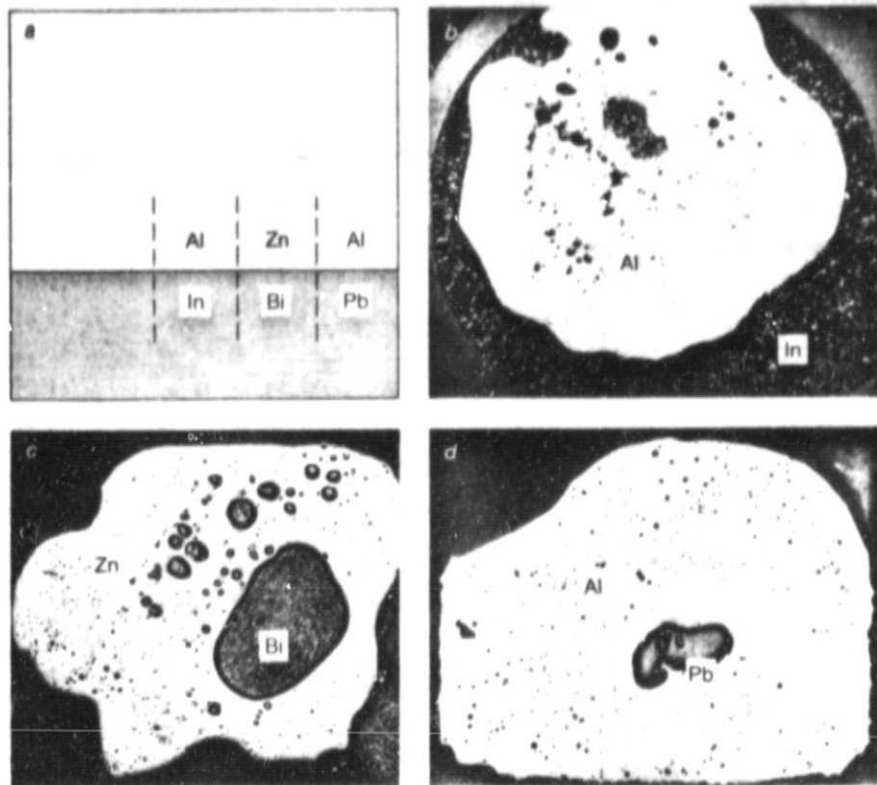
Fig. 10:  Separation of metal alloys with mixture gaps, specifically
of the systems aluminum (Al)/indium (In), zinc (Zn)/bismuth (Bi)
and aluminum/lead (Pb).  Such metal pairs, miscible at very high
temperatures, again separate upon cooling before solidification.  Under
terrestrial conditions (scheme a) ) the heavier component sinks after
phase separation.  The distribution under microgravity is quite
different; Hans Ahlborn of the University of Hamburg and Karl Loehberg
of the Technical University Berlin allowed a mixture of 40% indium in
aluminum to solidify in the SPAR 2 mission (b); Torbjoern Carlberg
and Hasse Frederikson of the Royal Institute for Technology in
Stockholm did the same in TEXUS 2 mission with a melt of 24% bismuth
in zinc (c), and Hans-Ulrich Walter of the DFVLR in Cologne sent along
a melt of 9% lead in aluminum (d) on the TEXUS 7 mission.  Controlling
factors for the distribution of the components under microgravity are
the better wetting of the crucible material by one of the components,
boundary surface convection of precipitates because of temperature
gradients from the outside in, as well as displacement of precipi-
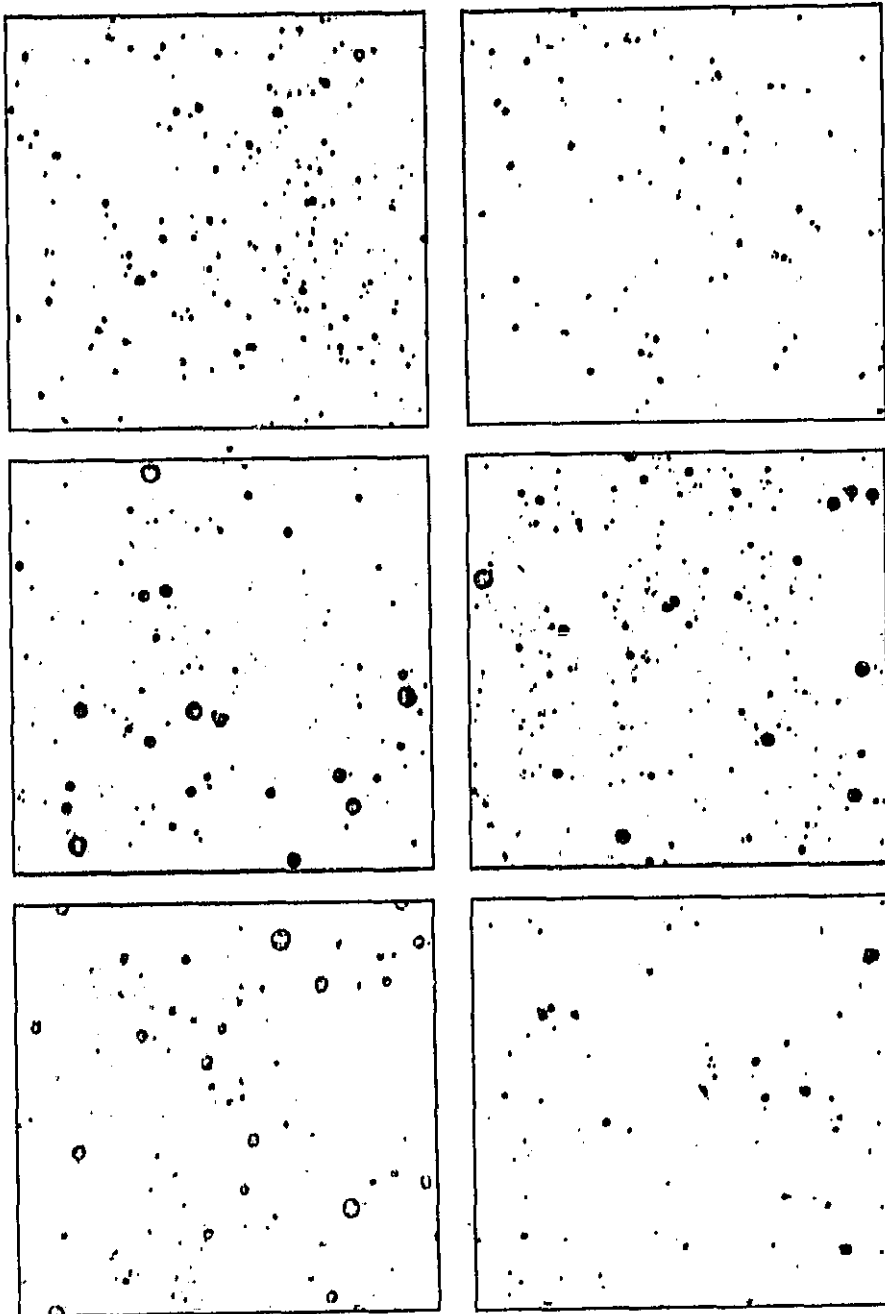tates by the solidification front of the component solidifying first.

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Figure 11

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Figure 11: Ground section photographs of solidified melts from the test of an acoustic mixer, which was used under terrestrial conditions (left) and during the TEXUS 5 mission (right). The samples always consisted of zinc with 3 volume percent of lead. The first third of the sample solidified with the mixer operating (upper row); during the solidification of the second third the mixer was shut off (center row) and during the solidification of the last third (lower row) it was again turned on. The lead inclusions in zinc are expectedly small in the first third because of the good mixing; after the shutoff of the mixer these deposits are considerably greater; after the restart of the mixer the particle diameters decrease. The photographs were made by Wolfgang Heide of the Battelle Institute.

Therefore the experiments could proceed under extensively identical conditions.

For the Spacelab mission in the summer of 1985 experiments concerning the pushing away or the dragging of solid foreign particles through melt- and solidification fronts are planned. A silver sample, which contains titanium-diboride particles of several micrometers in diameter distributed statistically, is first to be melted in an oriented way from left to right and subsequently solidified again from right to left.

Here one can then differentiate between two cases: If there is attraction between the embedded particles and the solidified phase,

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the particles will be carried along toward the right during melting;
upon renewed solidification of the samples they remain on the right.
If there is repulsion between the embedded particles and the
solidified phase, the particles remain where they are during melting;
upon renewed solidification they are pushed toward the left. It is
therefore possible to reconstruct from the final distribution
of the embedded particles the forces acting during melting and
solidification.

Production of solid emulsions

If the separation of metal alloys during cooling is to be
minimized, the use of a mixer suggests itself. It is then no longer
required to bridge the generally large temperature interval between
separation- and solidification temperatures solely by cooling;
rather it is possible to process directly above the solidification
temperature the first solidifying components. An acoustic mixer
was tested successfully in the TEXUS missions 5 and 8 (figure 11).

A second method for reducing the separation is the avoidance
of seed formation. It is known that one can heat pure air-free
water above 100 degrees Celsius without boiling. This phenomenon
is called boiling delay. However, for the least disturbance or
further temperature increase boiling sets in abruptly.

Similar things are true for very pure metals. They can be
cooled below the solidification temperature without immediately
solidifying. Supercooling by up to 20 percent of the absolute
solidification temperature are known. Subsequent solidification
then proceeds extremely fast. Therefore the molecules have no
chance to arrange themselves in accordance with the crystal structure
characteristic of them; rather the solidified metal is amorphous
(vitreous).

That which is true for pure substances, is also proper for
pure mixtures. A melt of limitedly miscible metals can, without

separation, be cooled below the separation temperature if seed formation is improbable as the result of extreme purity.

Thus one can at least go below the temperature interval to be passed through between separation- and solidification temperature, or even exceed it. For an abrupt solidification no substantial separation is then possible anymore.

In order to minimize the seed formation - which, as we know, takes place preferably at the boundary surfaces and particularly at the vessel walls - , one must work without a crucible.  Here the so-called positioners suggest themselves.  For the experiments  /41 under microgravity conditions instruments were developed which keep the sample in position by means of acoustic, electrostatic, and electromagnetic forces.

Growth of single crystals of multi-component semiconductors

For the growth under microgravity conditions one prefers primarily those crystals which can be grown from a solution and which therefore grow very slowly.  Growth rates of a few tenths of micrometers per second, that is a few millimeters per day, are typical.  A more rapid growth brings about crystal structure defects and the formation of new seeds.  There then develops instead of the desired single crystal a structure of numerous irregularly oriented crystals.

Of particular interest are single crystals of the two-component (binary) semiconductor gallium-antimonide, indium-phosphide, and cadmium-telluride as well as of the three-component (tertiary) semiconductor lead-tintelluride and mercury-cadmium-telluride, which are to be used primarily as infrared detectors.  All these materials are to be grown by means of the so-called traveling heater method.  Let us explain the principle of this method with
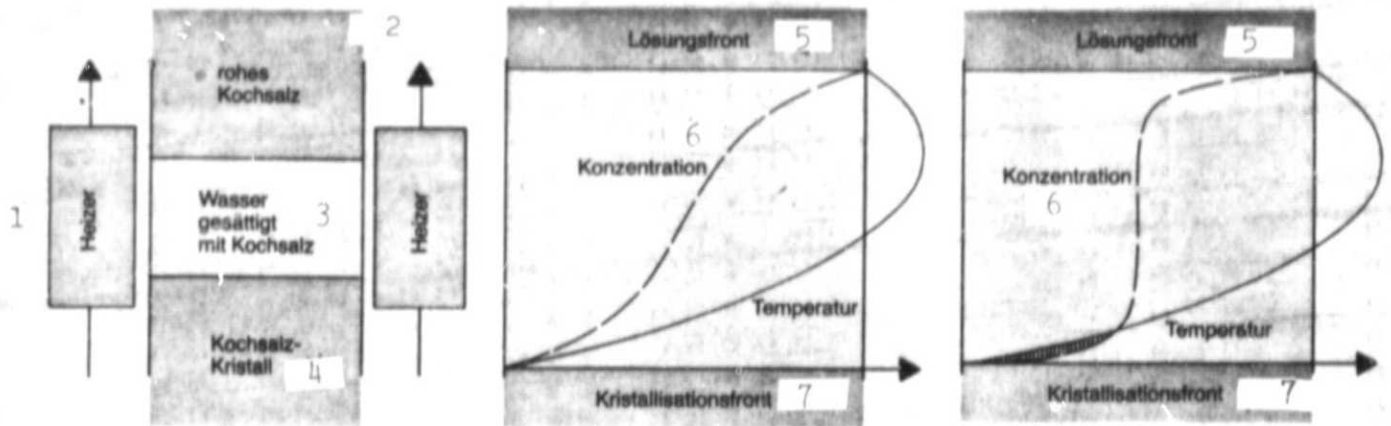
Figure 12: Principle of the travelling-heater method for growing
single crystals, in this case table salt (left). A perfect, well-
developed table-salt cube is placed in the bottom of an ampule as
seedling, then water saturated with table salt, and finally raw
table salt on top. A heating device travels upward along the outside
of the ampule. As a result, on top at the warmer side of the
aqueous zone, salt is continuously dissolved and is crystallized out
below at the seedling. If the growing of single crystals with the
travelling-heater method is to succeed, solution of the raw material
and precipitation of the dissolved material at the crystallization
front must be well tuned to one another. This requires that the
temperature everywhere in the solution zone lies above the crystalli-
zation temperature (center). This must be carefully observed in
the setup of the experiment, the selection of the heater, and the
specification of the growth rate. If the heater travels too fast,
if  the growth rate becomes too high (right); then the raw material
cannot migrate fast enough by diffusion (and, if present, by convection)
from the solution front to the crystallization front. The case can
occur that the temperature in the solution zone lies below the
crystallization temperature, which corresponds to the particular
concentration ("constitutional super-cooling"). In the cross-
hatched area undesired seeds can form at any time; then instead of
the desired single crystal an irregular structure would result.

Key:  1. heater  2. raw table salt  3. water saturated with table salt
4. table-salt crystal  5. solution front  6. concentration
7. crystallization front

the traveling heater by the simple example of cooking salt.

Cooking salt generally crystalizes in the form of cubes. In order to grow a large crystal, one can for one heat small-crystalline commercial cooking salt to the melting temperature of 801 degrees Celsius and then again cool it off in an oriented fashion from one side. The other and more conventional method is to dissolve the cooking salt in water with heating and then to allow it to again crystallize by cooling.

This can be done continuously in an ampule with a device as shown in figure 12: One inserts below as a seed a perfect well-formed cooking salt cube, places above it a layer of water saturated with cooking salt, and then fills the remainder of the ampule with raw cooking salt. If now a heater is made to travel outside o ̃ the ampule slowly from bottom to top, cooking salt is dissolved at the upper warmer side of the aqueous zone so that the solution is continuously enriched and the seedling grows at the colder side of the aqueous zone.

The mentioned semiconductor materials are usually grown in the solution zone with an excess of the lower melting component. The solution of the raw material at the solution front and the separation at the crystallization front require that the solution temperature lies only very little above the crystallization temperature. At the same time the concentration of the growing material is greater at the solution front than at the crystallization front. The components dissolved at the solution front migrate by diffusion and, if existing, by convection to the crystallization front.

Here it is important that the temperature in the solution zone is everywhere higher than the crystallization temperature corresponding to the particular concentration (figure 12 center). Otherwise - one then speaks of constitutional supercooling - undesired seeds can be formed at any time (figure 12 right).

While constitutional supercooling can be eliminated by slow growth, only insufficient experience and concepts are available concerning the second essential disturbance factor, the production of bands of different composition (figure 13). Such so-called striations can be formed by periodic fluctuations of the free convection and the boundary surface convection, by an interaction between convection and crystal growth or even by self-dynamics of the crystal growth rate.

Since the solution zone is heated from the outside, there develops under terrestrial conditions a natural convection which is directed on the inside downward and on the outside upward. During growth without ampoule (figure 13d) the boundary surface convection also comes into play which, for the given temperature distribution, triggers two stream rolls from the center of the solution zone to the raw crystal and to the pure crystal. For one the convection contributes to the material transport from a solution front to the crystallization front and to the temperature equalization in the solution zone. For another it produces a trend toward conversion into a periodic behavior as soon as it has an adverse /42 effect on the temperature profile to be generated.

Periodic fluctuations of convection and temperature bring about periodic fluctuations of the crystallization rate. Corresponding inhomogeneities of the crystal composition result. If the bands are triggered in this way flow-mechanically, then they should no longer appear under microgravity conditions when working with a closed ampule and thus without free surfaces.

The same is true in case the bands are produced as the result of interaction between convection and crystal growth. The interaction can be based among others on the fact that incidental fluctuations of the convection first stabilize themselves and become periodic by the fact that they bring about corresponding fluctuations of the crystallization rate and the volume of the solution zone.
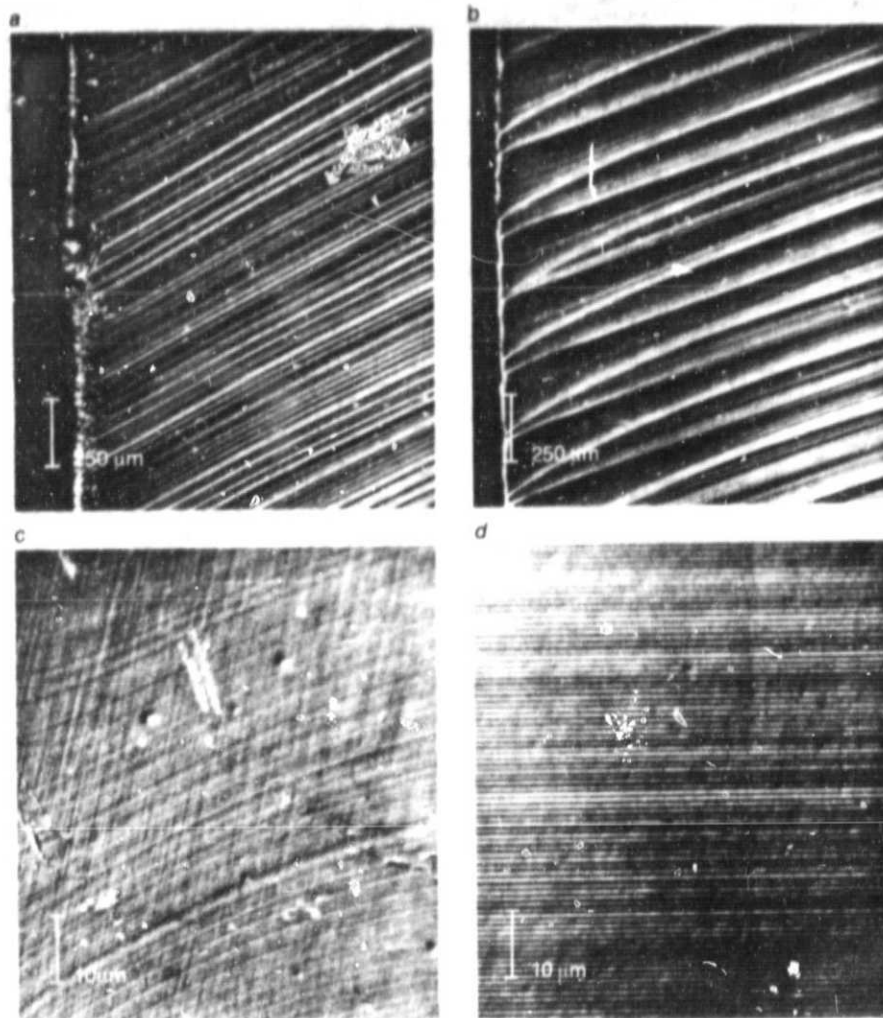
Figure 13: Crystal bands of various origins developing in the
growing of single crystals. On a silicon crystal grown from the
melt at a rate of 2 millimeters per minute doping bands have formed
at irregular intervals between 10 and 30 microns       (a). The
growing silicon crystal was rotated to equalize the unsymmetrical
heating at eight revolutions per minute; in this way rotation-
and doping bands were formed at distances of 0.25 millimeters (b).
On a gallium arsenide crystal, doped with tellurium and grown from
the solution, bands at irregular intervals between 2 and 5
 microns     and growth defects can be seen (c). In a comparison
experiment conducted without ampule and with a free surface doping
bands also developed (d); since in addition to natural convection
boundary surface convection also was present, they have very regular
distances between them. The photographs a and b were made by
Rudolf Nitsche (University of Freiburg), the photographs c and d
by Klaus-W. Benz (University of Stuttgart).

In both cases one must be able to establish a correlation between the temperature fluctuation in the solution zone and the distance between the bands.

The great decrease of the distance between striations suggests a substantial participation of convection in the striation formation during the growth with free surface (figure 13d) as compared to the growth in a closed ampuole (figure 13c). The boundary surface convection occurring in the first case increases the flow velocity in the solution zone considerably and thus brings about more rapid oscillations and shorter distances between striations.

Finally one cannot exclude that striations are also produced as the result of self-dynamics of a crystal growth. A perfect crystal is generally more stable than an imperfect one, that is a crystal fi..led with structural defects or an impure crystal; therefore its crystallization temperature is higher. For the case that the heater travels faster than the crystal can grow perfectly, the crystallization front lags behind the crystallization temperature; the temperature at the crystallization front drops, and an imperfect crystal section is formed at increased crystallization rate until the crystallization front has again caught up with the crystallization temperature. This produces a growth jump.

Also, or exactly because of this effect experiments under microgravity conditions are appropriate. Without convection in the solution zone the atoms of the growing material can pre-arrange themselves at the crystallization front. Then the growth rate of the perfect crystal increases considerably, and jumpy growth is avoided.

A pre-arrangement of the atoms is suggested particularly by the results of Walter Littke of the University of Freiburg during the growth of enzyme crystals in the mission TEXUS 5. Here he was dealing with crystals of molecules which have a chain length of

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several ten-thousand atoms. The crystal grown under microgravity
conditions was by far greater than all crystals grown till now in
earth laboratories. The results available till now of the first
Spacelab flight point in the same direction. Although the disturbance
of the crystal growth rates by convection is particularly noticeable
for long chain molecules, it cannot be excluded that even in the
crystallization of atoms the crystallization rate and the homogeneity
of the produced crystal is disturbed by convection.

The experiments conducted till now under microgravity conditions
have produced surprising  results, but have also uncovered avoidable
mistakes. Many effects were considerably more pronounced than one
could expect as the result of terrestrial pretests. As the result,
some complicated clarification tests became unnecessary. Other
experiments gave rise to new theories.

This trend will also continue during the continuing Spacelab
missions, which make possible microgravity  for periods of days:  Some
of the hypotheses postulated here will be disproved, others will
be confirmed and new ones will appear.