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MATERIAL SCIENCES EXPERIMENTS UNDER MICROGRAVITY CONDITIONS WITH M*A*U*S

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REDUCED GRAVITY
GRAVITATION EFFECTS
SEDIMENTS
CONVECTION
SPACE SHUTTLE PAYLOADS
DISPERSION
TEMP. GRADIENT
X RAY
RADIOGRAPHY
LIQUID-SOLID INTERFACE SLIP CASTING

Abstract

Project MAUS is a part of the German material sciences program and provides autonomous payloads for the Space Shuttle. These payloads are housed in canisters which are identical with those of NASA's Get-Away-Special program. The main components of the hardware are: a standard system consisting of power supply, experiment control, data acquisition and the experiment modules containing experiment specific hardware. So far, three MAUS modules with experiments from the area of material sciences have been flown as GAS payloads.

Introduction

NASA's announcement to provide flight opportunities in the Space Shuttle on a low-cost, space-available basis induced the German government, represented by the Minister for Research and Technology (BMFT), to book 25 options in the Get-Away-Special Program. For optimal utilization of these flight opportunities project MAUS (Materialwissenschaftliche autonome Experimente unter Schwerelosigkeit) was initiated with project management assigned to DFVLR and MBB/ERNO company selected as the main contractor. This project is part of the German material science program supporting experiment development for the German Spacelab Missions (D1 and D2), a sounding rocket project (TEXUS) and ground based research.

As a result of a phase A study it was decided that a MAUS payload should consist of a standard system, developed and manufactured by a main contractor and an experiment module containing experiment specific hardware. In general this hardware is supplied by the principal investigator with support being also provided by the main contractor.

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In addition, a MAUS payload should not only be compatible with the requirements of the GAS program but also should be designed for use in dedicated Shuttle Missions as payload complement of partial payloads. In the past years ten MAUS experiments have been flown among others with the structure OSTA-2 (1983) and the sattelite SPAS-01 (1983, 1984).

In December of 1979 a contract was plced with MBB/ERNO for development and fabrication of ten MAUS standard systems and preparation of flight experiments was started at several institutions.

MAUS Standard System

A MAUS Payload consists of the experiment mounting structure (EMS), the batteries, the standard electronics for experiment control and data acquisition, the house-keeping systems, and the experiment hardware.

The experiment mounting structure is built of an adapter ring, 6 posts and 2 experiment platforms with brackets. Three different batteries are providing power for experiments and electronics. The total capacity of the experiment battery is 1.8 kWh. The data acquisition system consists of a microprocessor controlled multiplexer unit with digital and analog inputs. The data are stored via an intermediate memory on tape. The capacity is 10 Mbits. To allow for long measurement phases a data reduction system is provided. A detailed description of the standard service system can be found in Ref. 1, a photo is given in Fig. 1.

Experiment Interface

For the experiments to be accomodated in a MAUS module two platforms are available one of which is adjustable in height by 25 mm-steps. The maximum height for the experiments is 400 mm and the maximum mass about 20 kg. Space for 6 cards is available in the standard electronic boxes for the experiment dedicated electronics.

Experiments

It is a policy of the project to assign experiments to a MAUS mission rather late in order to maintain a high degree of flexibility. About one year is con-

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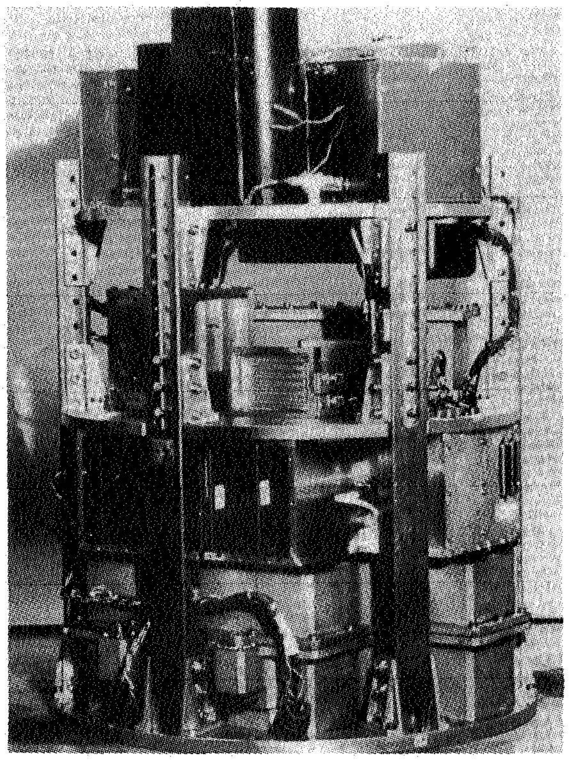

Fig. 1: Total view of MAUS Standard System with dispersion experiment at top during integration. The battery housing can be seen at the lower part of the photo, above the electronics for experiment control and data acquisition.

sidered for the development of the experiment specific flight hardware by the experimenter. Preliminary flight assignments are made from a pool of experiments consisting of microgravity relevant proposals which fit into the limitations set by the MAUS project with regard to power-consumption, volume, weight etc.

The objectives of the different proposals are generally of a scientific nature and do not attempt manufacturing processes in space. It has been one of the results of earlier experiments that "secondary" phenomena become important which are normally masked by Earth's gravity. These effects should be understood first in order to perform meaningful future experiments and to be able to exploit the benefits of microgravity. In this regard MAUS experiments from the area of material sciences so far dealt with the effects of reduced sedimentation, convection phenomena caused by temperature gradients and the behaviour of dispersed particles ahead of a solid/liquid interface.

The first flight of a MAUS payload occured within the GAS program on Shuttle STS-5 (1982). The objectives were twofold: to test the function of the MAUS standard system and to perform a scientific metallurgy experiment using X-ray radiography as a diagnostic tool. A short summary of objectives follows:

"Stability of Metallic Dispersions"
Investigator: Dr. G. Otto, DFVLR Köln-Porz

There are several combinations of two different metals which show solubility in their liquid state above a certain temperature (consolute temperature) and immiscibility below this temperature. Such a combination consisting of gallium and mercury was used to investigate the dissolution process above the consolute temperature and the time-dependent stability of the resulting dispersion, composed of mercury droplets in gallium. For the first time, this experiment employed X-rays to penetrate the metallic sample and to provide a series of real-time data during different states of the experiment sequence. The sample could be recycled into its starting conditions by repeated thermal treatment. The experiment was planned to run for a duration of 3 days.

After return of the payload to the MAUS team it was found that the experiment had not worked. A failure analysis yielded that a leak in a silver-zinc electronic battery had developed during the several weeks of waiting time on ground. Because of no voltage conditions the electronics of the standard system could not be activated by the "on" signal given by the crew.

The cause of the leak, a simple O-ring seal, was corrected and the same payload successfully reflown on the NASA structure OSTA-2 with STS-7. This experiment then yielded the first X-ray photos from a metallic dispersion by cooling a homogeneous solution into the miscibility gap. Some of the observations made are the following:

- Homogenisation appears to be completed after 4 hours at 190°C. This can be concluded from the constant grey scale value of the sample when measuring across the X-ray film. In the laboratory at least 8 hours are needed for worst case conditions when the heavier mercury is on the bottom of the container.
- When cooling the sample into the miscibility gap with a rate of 30 K/min the precipitation of Hg-droplets occurs rapidly. However, no finely dispersed state with a particle size of about 0.3 mm diameter (resolution limit of the X-ray photos) can be observed. Hg-droplets seem to be generated by heteroge-

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neous nucleation at the gallium surface. Droplets seem to be stationary once they achieve the visibility limit and do not show any blurring by movement despite an exposure time of 20 s.

- Supercooling of the melt appears small and if present should be less than 20 °C.

- When cooling into the gap the growth of precipitated droplets is rather fast (Fig. 2). Within one minute (30 K into the gap) the particles have already grown to an average diameter of 0.8 mm. Anticipating growth by diffusion only, the diameters increase too fast by at least a factor of five. Other processes like convective material transport or coalescence are likely to contribute to growth. A detailed discussion of the data is given in Ref. 2

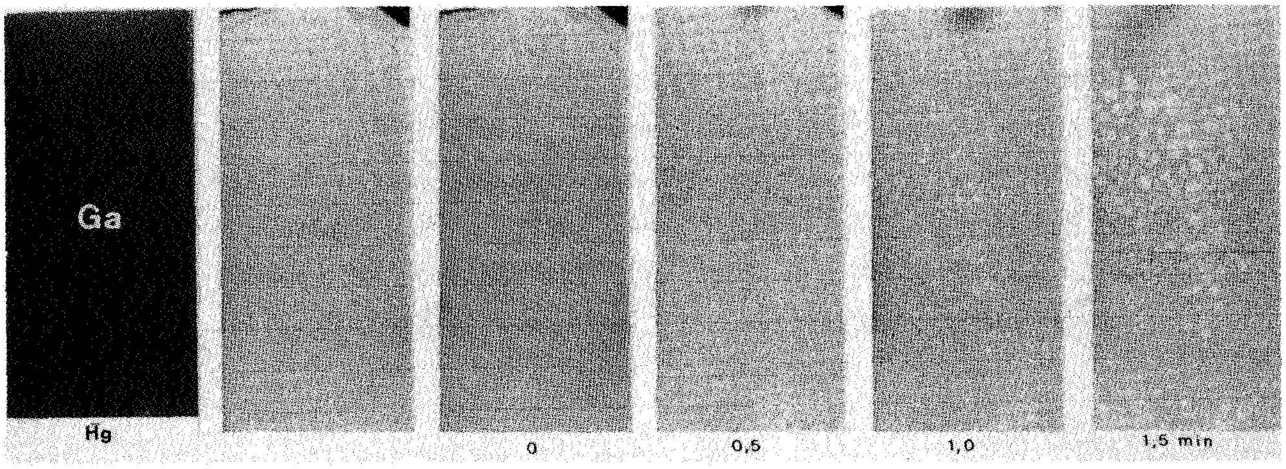

Fig. 2: Precipitation of mercury during cooling into the miscibility gap with a rate of 30 K/min. Shown are from left to right the ground based state, the appearance of the sample after 8 hours at 190°C in microgravity and the cooling sequence. Time interval between each of the last four photographs is 30 sec. Color representation: Ga - Dark; Hg - Light.

The housekeeping systems also provided information about the payload from which the acceleration data taken over a period of three days are the most interesting (Ref. 3). Crew activities and activation of the robotic arm can be seen clearly on the record. It should be stated that g-sensitive runs of the X-ray experiment were programmed to happen during the sleeping time of the crew.

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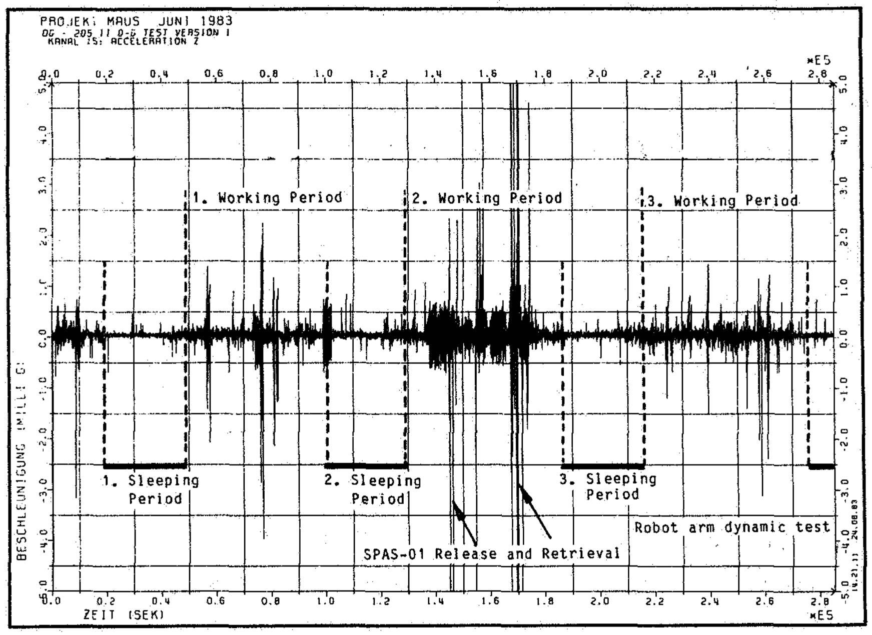

Fig. 3: Accleration data acquired during the run of the X-ray experiment for a period of three days during mission STS-7. The generation of g-jitter by different crew activities and the activation of the robotic arm during this mission can be seen clearly. Saturation of the g-sensor occurs at 5 x 10^{-3} g. Each MAUS payload carries its own acceleration sensor which is activated with the "on" signal.

Two additional MAUS payloads to be flown aboard STS 51-G in the GAS program were turned over to NASA on April 26, 1985. A short description of the experiments and their scientific objectives are given below.

"Fundamental Studies in the System Manganese-Bismuth"
Investigator: P. Pant, Krupp Research Institute, Essen

The main objective of this experiment is the synthesis of the intermetallic compound MnBi in an isothermal furnace. The compound forms by a peritectic reaction at 450°C involving liquid bismuth and solid manganese. This kind of reaction is diffusion controlled and requires a longer time for completion as when both components were in a liquid state. On Earth, such reactions are incomplete when both components exhibit different densities and become separated by sedimentation and buoyancy. The compound MnBi has promising applications as a magnet material because of its high theoretical coercitive strength which so far could not be achieved with ground based specimens.

First experiments on TEXUS (Technology Experiments und Reduced Gravity, a German program using sounding rockets) showed that during melting and solidification under microgravity separation of the components did not occur and forces promoting segregation, e.g. surface tension gradients did not become effective. The MnBi-phase in the flight samples was found in form of micron-sized particles uniformly distributed. The small size is thought to be responsible for the good magnetic properties of the flight samples when compared with the ground base samples.

During the course of this MAUS experiment 8 MnBi samples will be processed in a two-chamber isothermal furnace. Sequentially, each furnace chamber containing 2 samples will follow a pre-programmed temperature profile of heating and cooling. Since the processing time under reduced gravity in MAUS is about 3 hours for each sample the question may be answered weather the peritectic reaction to form MnBi can run its full course. This hasn't been possible with the TEXUS experiments which were performed during only 6 minutes of microgravity conditions.

"Slip Casting Under Microgravity Conditions"
Investigator: Dr. K. Schweitzer, Motoren und Turbinen Union (MTU), München

The process of slip casting employs a ceramic slurry to form complicated shapes of hollow bodies. On Earth, this process is limited in applications because of gravitational influences on the dispersed particles in the slurry. Sedimentation can only be avoided by the use of materials with equal densities or by the utilization of stabilizing additives. However, the latter may be harmful to the desired properties of the slip cast product. Goal of this experiment is to demonstrate with model materials that slip casting is possible in microgravity even with unstabilized suspensions. Using mixtures of powders with different density, grain-size and concentration. For this reason ceramic and/or metal powders are homogeneously mixed in solid paraffin by kneading. Rods of these solid slurries are pressed into cartridges against the ends of porous ceramic rods which are mounted in the lower halves of these cartridges.

During weightlessness thirteen samples of these solid slurries will be melted by heating the upper part of the cartridge in a furnace. Then the slip casting process will be started by additionally heating the lower part of the cartridge containing the suction bodies made of porous ceramic. These will slowly absorb paraffin but not the dispersed particles. The casting process will be stopped by turning off the furnace and cooling of the samples. Solidification of the paraffin will preserve the slip cast layers as well as the residual slurries for later examination on Earth in respect to their structure and particle distribution. This experiment will lead to a better understanding of the slip cast process and evaluate the possibilities for the casting of delicate shapes under microgravity conditions.

Future Planning

MAUS missions will continue with flights of dual payloads using the remaining 22 GAS options. Launch Services Agreements with NASA have been signed. In princple, international participation in this project is possible. The MAUS standard system is also available by MBB/ERNO on a commercial basis.

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