Material Processing Facilities in Space

Xiuhong Pan, Fei Ai and Yan Liu

Abstract Many researchers around the world have lots of interests in discovering what and how may happen when materials processing is conducted in space. Usually, for the investigation of space material sciences, experimental facilities are important and necessary. Up to now, more than one hundred of material experimental facilities have been developed in the world. They include many high-temperature heating furnaces, in situ observation and diagnosis equipments, as well as facilities for crystal growth from water solution in microgravity. Some of them are prepared by international cooperation among two or even more countries. In this chapter, we will give a brief summary for the material processing facilities in the world. The facilities developed in the recent ten years serving on International Space Station (ISS) are particularly focused. Furthermore, some material experimental devices built by China which have served in Chinese recoverable satellites and man-made space crafts are also discussed emphatically.

Keywords Microgravity material science · Facility · Heating furnace · In situ observation equipment

1 High-Temperature Heating Furnaces in Microgravity

For many kinds of materials, such as metal alloy, semiconductor, oxide crystal, ceramic, their experiments are generally performed under high temperature. So heating furnace is one important kind of experimental facilities for materials processing in microgravity. For such a furnace, its temperature can arrive up to

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several hundreds of centigrade or even higher than one thousand centigrade. Almost all the countries aiming at materials science research in space have developed their own heating furnace. The follows are some represents.

1.1 Russian POLIZON Facility

Russian/Soviet Union is one of the earliest countries to perform material experiments in microgravity [1]. More than ten material processing facilities have been built and launched on the Soyuz space capsule, Salyut space station, the MIR space station, or on the Photon satellite. Among these, POLIZON is the latest one. The POLIZON facility can perform material experiment using the PROGRESS transport spacecraft as a flying platform, which is aimed to deliver the payload on the international space station (ISS) [2].

The POLIZON facility includes an electrovacuum furnace with one 8-sectional heater, in which up to 12 different experiments on crystal growth can be performed in an automatic mode (Fig. 1). The furnace is equipped with the loading–unloading device in which 12 capsules with material samples are installed. The capsules are loaded by turn into the furnace, the heaters are switched on, and the melting process begins. Due to adjusting temperature on the separate sections of the heater, it is possible to obtain the optimal temperature profile along the capsule axis for each experiment. Crystal growth by Bridgman method is realized by transition of the capsule from the furnace at a given velocity.

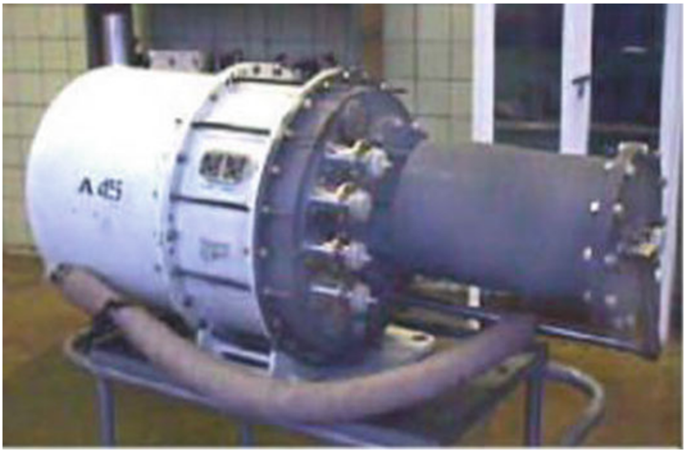


Fig. 1 The photo of POLIZON furnace, reprinted from reference [2]. Copyright 2018, with permission from Acta Astronautica

The furnace is equipped with a system of magnetic stirring of the melt that allows to heat control and mass transfer during crystal growth. The maximum temperature of POLIZON is 1200 °C.

1.2 Heating Furnace in Microgravity Developed by German

Some other European countries have also developed their heating furnace for material experiment in microgravity, among those German contributed much. In 1996, a multizone furnace including a magnetic damping array consisting of two radially magnetized permanent magnet rings was developed in Germany [3]. In this furnace, a three-zone heater has been designed which is suited to the utilization in the zone melting facility (ZMF). It is designed for crystal growth of semiconductors and provides standard subsystems like the heater translation mechanism, gas supply, and process chamber for the implementation of a specific multizone heater. The heater has been laid out for a maximum achievable temperature of the central heating zone of 1300 °C with a maximum total power of 1200 W. It has three heating zones, two isothermal heaters made of Kanthal with a length of 80 mm, and the central heater with a length of 22 mm. In order to compensate for the magnetic field arising from the dc current all heater wires are winded bifilarly.

Another heating furnace for solidification investigation of aluminum alloys had been prepared by German [4]. This facility named TITUS furnace has a 6-zone heater. It served onboard the Russian MIR Space Station. Al–Ti alloy experiments were performed in this furnace in 1999. The pulling rate of solidification can arrive at 1.9 μm/s. This furnace is used to study the transition from equiaxed to columnar morphologies.

1.3 Materials Heating Furnaces on ISS

NASA has sponsored the flight investigation on growing crystals with baffle under microgravity condition [5]. This project named SUBSA (Solidification Using a Baffle in Sealed Ampoules) was first conducted in the Microgravity Science Glovebox (MSG) on ISS.

The flight furnace located in the MSG had a transparent gradient section, and a video camera, sending images to the earth. The SUBSA furnace which designed for growing InSb is shown in Fig. 2. The axial temperature gradient in the transparent section was approximately 80 K/cm. The seed was placed about 10 mm from the furnace, so that seeding and the initial 10 mm of growth could be observed. The heater temperature is lower than 820 °C. Crystal growth was performed in this furnace by lowering the furnace temperature.

Several other heating furnaces can serve for materials experiments on ISS [6]. For example, NASA developed the Quench Module Insert (QMI) and the Diffusion

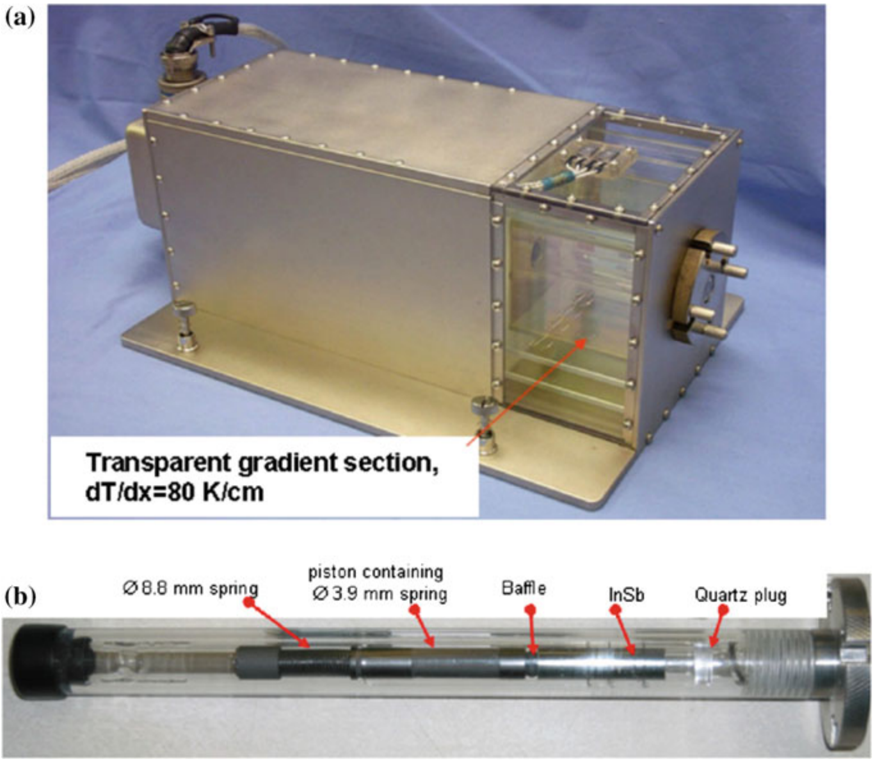


Fig. 2 SUBSA furnace with a transparent gradient region **a** and the ampoule with the baffle **b**, reprinted from reference [5]. Copyright 2018, with permission from Journal of Crystal Growth

Module Insert (DMI). ESA prepared a Low Gradient Furnace Module Insert (LGFMI) and the Solidification Quench Furnace Module Insert (SQFMI), which are both for material processing on ISS. JAXA built the Gradient Heating Furnace (GHF) which is in the Kobairo Rack (Kobairo) of ISS. Some of them are fixed in the Materials Science Research Facility (MSRF) of ISS, which is designed to accommodate materials science investigations selected to conduct research in the microgravity environment of ISS. The MSRF consists of modular autonomous Materials Science Research Racks (MSRRs).

The Quench Module Insert (QMI) operates inside the Materials Science Laboratory (MSL), which was launched at 2009 and installed in NASA's first Materials Science Research Rack (MSRR-1) [7]. This unique material processing furnace has been designed to create an extremely high temperature gradient for the directional solidification processing of metals and alloys. But at the same time it is flexible enough to process samples in either low-gradient or isothermal environments. QMI can heat samples up to 1400 °C, suitable for the processing of aluminum samples and its alloys. The QMI has also been designed with "quench capability." This allows

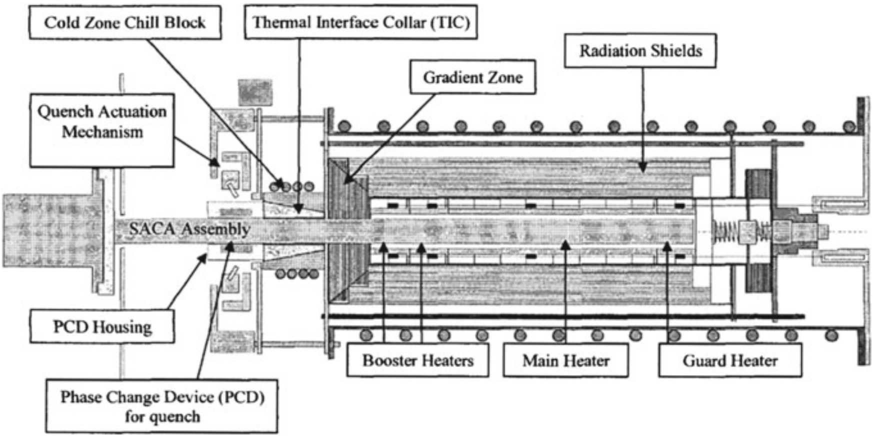


Fig. 3 Schematic view of the QMI furnace on ISS, reprinted from reference [7]. Copyright 2018, with permission from IEEE Aerospace Conference Proceedings

the furnace to rapidly freeze a sample at the solid-liquid interface. The design of the QMI can be broken down into three main parts: the hot zone responsible for melting the sample, the cold zone responsible for freezing the sample, and the quench zone which rapidly freezes the samples when desired to preserve the solid-liquid interface. A diagram of the furnace is shown in Fig. 3. The hot zone is constructed from four individually controlled heaters: the two booster heaters, the main heater and the guard heater. The main heater sets the overall sample temperature. The guard heater prevents the temperature at the end of the sample from falling off. The booster heaters keep the temperature of the sample from cooling off too far ahead of the chill block. The sample is quenched, or rapidly cooled, with the "Phase Change Device" located at the end of the "Thermal Interface Collar" (TIC). The samples are contained in "Sample Ampoule Cartridge Assemblies" (SACAs). The QMI works in vacuum.

The Diffusion Module Insert (DMI) is the second NASA-built insert for flight on the MSRR-1 [8]. DMI is a Bridgman type furnace, which is designed to accommodate processing temperatures up to 1600 °C. The requirements are for this Module Insert to have appropriate isothermality for Fickian diffusion measurements, but to support adequate temperature gradients for Soret experiments and Bridgman growth. Fickian and Soret diffusion investigations levy substantially different requirements on the furnace. Both processes focus on the study of diffusion. The Fickian experiment design calls for an isothermal furnace chamber at least 10 cm in length with temperature capabilities up to 1600 °C. Soret diffusion calls for a Bridgman-Stockbarger furnace that can provide an axial gradient of approximately 100 °C/cm. NASA's Advanced Pattern Formation and Coarsening Research Module (APFCRM) is an on-orbit replacement for an experiment module sponsored under NASA's Space Product Development program. It consists of a low temperature facility with a precisely controlled both for in situ observation of the solidification and growth of transparent model materials that simulate the behavior of metals and alloys.

With the ELIPS (European Program for Life and Physical Science) Program of the ESA, the MLS including furnace inserts have been developed to allow directional solidification and crystal growth processing on the ISS. ESA's Low Gradient Furnace Module Insert is a furnace for crystal growth capable of reaching 1400 °C. Samples can be translated at slow and precise rates within a temperature controlled environment. Magnetic field capabilities, both static and rotating are available to influence the liquid flow and improve the properties of the crystalline product. ESA's Solidification Quench Furnace Module Insert is a furnace designed primarily for metallurgical experiments capable of reaching 1400 °C, and including a quench capability. While initially designed to be used for ESA experiments, these latter two insert modules may be made available for NASA experimenters. By these facilities, samples of 8 mm diameter and 245 mm length have been processed and other sizes are possible. Up to 12 thermocouples can be accommodated in the crucible along the sample for temperature measurements.

The Gradient Heating Furnace (GHF) by JAXA is in the Kobairo Rack (Kobairo) ISS [9]. The GHF is a vacuum furnace that contains three heating blocks, and is used mainly to conduct high quality crystal growth experiments using unidirectional solidification. The three heater-units can generate the high temperature gradients needed to produce large scale pure crystals. Some preliminary experiments of $In_xGa_{1-x}Sb$ crystal growth in GHF have been performed recently [10, 11].

1.4 Materials Heating Furnaces for Microgravity Investigation Developed in China

The first heating furnace aimed at materials science research in space was designed and successfully carried out into orbit at the end of 1980s in China. Since then, a series of different materials processing experiments have been performed more than 10 times by virtue of piggyback opportunities onboard both the recoverable satellites and the manmade spacecrafts in China. Most of these furnaces had the ability of heating for material experiments at high temperature.

The DGW facility on Chinese Manmade Spacecraft. The temperature of Chinese early materials processing furnaces in microgravity could not be programmably controlled, until the occurrence of Duo Gong Wei (DGW) facility. The project of DGW materials processing facility began in 1993. This facility was designed mainly for crystal growth, solidification of metals and alloys, and the process of glasses, as the payloads of Chinese "SZ (ShenZhou)" series spacecrafts in space. DGW facility has been carried into orbits 3 times, as the payloads of SZ-1, SZ-2, SZ-3 Chinese unmanned spacecrafts from 1999 to 2002 [12, 13].

The DGW facility is a one-zone heater furnace. It consists of 3 main parts: the heating furnace, the sample management system and the controller. Both the heating furnace and the sample management system are enclosed inside a vacuum-tight housing. The heating furnace is constructed of an alumina tubular chamber of 24 mm

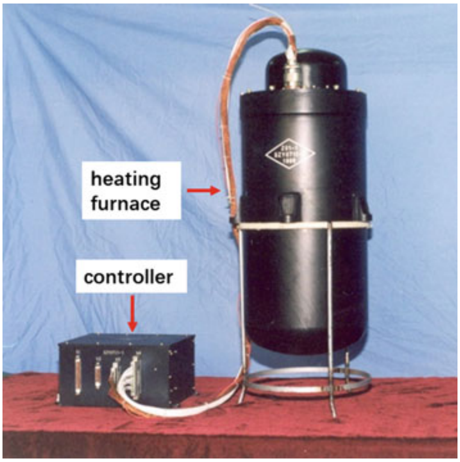


Fig. 4 The DGW facility for the Chinese SZ-3 flight mission

in diameter and 40 mm in length, a Ni–Cr filament resistance heating unit, multi-layer radiation reflectors, compound insulation structures and five S-type thermocouples. The Ni–Cr heating filament is enwound tightly adjacent to the inner bore of the alumina chamber to enable easy and direct heating to the sample cartridge with minimum heat loss. The furnace is able to maintain at 1000 °C. Totally six samples cylindrical cartridges can be installed in this furnace for microgravity experiments.

The controller is a programmable microprocessor system, which has the duel functions of process control and data acquisition. It controls the heating process of the furnace and the movements, including axial temperature-time profiles, the exact position of each cartridge, the rotating speed of the magazine and the translating speed of the cartridges. The PID mode controller of the facility has the ability of obtaining temperature stability better than ± 1.0°C/h. Figure 4 is the photo of DGW facility served in SZ-3 Chinese unmanned spacecraft.

The MMP facility on Chinese SJ-10 Recoverable Satellite. Recently, much more requirements proposed by the materials scientists greatly accelerate the developments in the field of space materials science in China. In other words, differing from before, more and more different materials including semiconductors, metal and alloys, single crystals, etc., are required to be processed in microgravity by taking advantage of very valuable opportunities given by recoverable satellite. Considering the varieties of materials and their experimental requirements, and also, the constraints of size, mass, energy given by the satellite, the Multi-functional Materials Processing (MMP) facility has being developed [14]. This heater furnace is designed for materials experiments planned to carry out in space by Chinese "SJ (Shijian)-10" recoverable satellite.

The design of MMP facility can be broken into four main parts as shown in Fig. 5: the vacuum house 1, the heating furnace 2, the sample arrangement assembly 3, and the controller 4. The later three parts are all in the vacuum house and fixed tightly with each other.

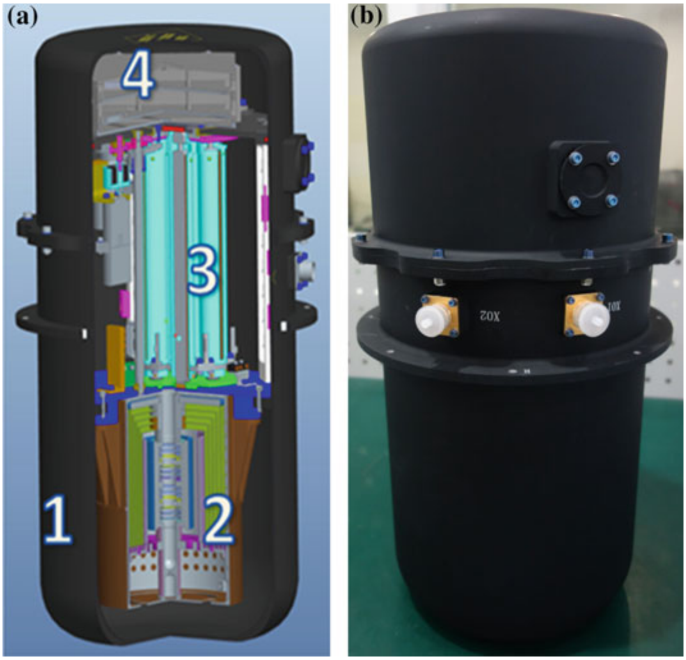


Fig. 5 The structure of MMP facility **a** and its photo **b** served on Chinese SJ-10 recoverable satellite

The vacuum house is a cylindrical house prepared with Al alloys. It can supply the vacuum environment when MMP facility works. This is benefit to the heat energy conservation as well as to preventing the sample cartridges being polluted at high temperature. Furthermore, all the mechanical and electrical interfaces of MMP facility with the satellite are designed on this vacuum house, including a flange ring and two electric connectors.

The heating furnace is designed with two adjacent heating zones, which can adjust the temperature file along the axis. It is a style of Bridgman method. The heater core of the furnace is constructed by two individual heaters, main brackets, four S-type thermocouples and high purity alumina pans. These two heaters are fabricated by resistance coils which enwound into a tube. The surface of the tube is a helical resistance coil. These tubes are encased along the circular grooves in pans whose symmetrical axes are in the same line. The cartridge of $\phi 16$ mm is structured by these pans. These two heaters can be controlled separately to achieve the various temperature profiles along the axes of the cartridge in accordance with each special experimental requirement. These two half main brackets shaping into a cylinder are able to fix these pans tightly through several grooves in themselves. The feature of the structure is efficient to focus the energy in the region needed and at the same time, enough long operation life. In fact, the average life of it is more than 1000 h under the maximum temperature of 950 °C validated by experimental tests on ground.

The sample arrangement assembly consists of two kinds of drive gears, two motors, several hall locators, and 6 sample magazines etc. Totally 6 sample cartridges with 231 mm in length are mounted in the 6 sample magazines individually which can be automatically exchanged by these drive gears. One group of the drive gears are used for displacing the cartridge in or out the heating region along the axes of the heating core. And the other group of drive gears can make the 6 sample magazines around the axes of the center and move them to the right place to do the next experiments located accurately. The maximum processing length of the sample is 178 mm with the translation rate ranging from 0.5 to 120 mm/h.

The sample translation and sample movement, as well as the heater process, are controlled by the controller. The controller is a programmable microprocessor system. The PID mode is used for temperature control with temperature stability of better than ±0.5 °C/h. The controller can communicate with the satellite by the two electric connectors on the vacuum house. It should be noted that, as for all of other Chinese materials processing facilities with high temperature, their electric controllers are fixed separately from the heating furnace. So there are at least two machines for each of these facilities. However, the MMP facility is only one machine since its controller is integrated into the vacuum house. This design makes the temperature controlling being more accurate due to the shorter cables between the heater and the controller.

In Table 1, the main technical specifications of MMP facility are summarized.

MMP facility was designed mainly for the project of “Research of Melt Growth in Space” (RMGS) on Chinese “SJ-10” recoverable satellite, which is supported by the Strategic Priority Research Program on Space Science from the Chinese Academy of Sciences. In this project totally 8 material sample experiments have been arranged in space. There are:

- Crystal growth of GaMnSb magnetic semiconductor in space;
- Growth and study of fluoride laser crystal with tunable ultraviolet wavelength;
- Growth and property study of new infrared detector material;
- Wetting of metallic molten and formation of composite;
- Solidification and defect controlling of high temperature alloy single crystal;

Table 1 The technical specifications of the MMP facility in Chinese SJ-10 recoverable satellite

Specifications	Values
Number of heating zones	2
Maximum temperature	950 °C
Maximum power consumption	Lower than 140 W
Translation rate range	0.5–120 mm/h
Max of axial temperature gradient	75 °C/cm
Number of magazines	6
Cartridge length	ϕ 16 mm × 231 mm
Temperature stability	Better than ±0.5 °C/h

Fig. 6 The returned sample cartridges in MMP facility served on the Chinese SJ-10 recoverable satellite

- Synthesis of metallic matrix composite by self-propagating high-temperature technique;
- Interface phenomena of Ti-based alloy during melting;
- Growth, simulation and property study of ternary InGaSb phototransistor crystal.

This MMP facility was carried into orbits by the “SJ-10” recoverable satellite launched at April 4, 2016, and automatically performed 8 material experiments in space lasting more than 210 h. It should be noted that, the 8 material samples were fixed in 6 cartridges, with the fourth and the fifth cartridges containing two samples, respectively. Figure 6 shows the sample cartridges returned to the ground after the experiments in space. Although two heating zones have been designed in the MMP facility, they were not allowed to work simultaneously in space due to the electrical current limitation. As a result, several samples in MMP had not got to their programmed maximum temperature during the microgravity experiments.

The Heating Furnace in Chinese TG-2 Space Lab. Another two-zone heaters furnace has been laughed in Chinese “TG (Tiangong)”-2 space laboratory in September 15, 2016. The structure of this heater furnace is familiar to that of MMP facility on Chinese “SJ-10” recoverable satellite. It also has two adjacent heating zones, which are fabricated by resistance coils enwound into a tube and the temperature of each heating zone can be controlled separately. The maximal temperature of this furnace can also arrive at about 950 °C. Furthermore, sample cartridges in this facility can be manned exchanged batchedly by the cosmonaut. Firstly, six sample cartridges with ϕ 16 mm × 260 mm were mounted in the magazines and finished their heating experiments in space automatically. Then these six sample cartridges were drawn out of the heating furnace by the cosmonaut and another group of six sample cartridges were put into the furnace for space experiments. At last, the third group of six sample cartridges were put into the furnace replacing the second group of six sample cartridges when the later finished their experiments. This is the first time that cosmonaut can participate in the material experiments in space in Chinese history. The heater furnace has performed 18 sample experiments in “TG-2” space laboratory in space.

In the future, Chinese Space Station (CSS) will be built, onboard which a High Temperature Experimental Rack (HTER) will be developed. This rack is a comprehensive equipment for materials science investigation in space. It will take on the functions of high temperature processing, in situ observation by X-ray or optical technique, and measurement of melting physical properties. Furthermore, this rack will be designed with "quench capability" as well as with a system of magnetic stirring of the melt during crystal growth.

2 In Situ Observation Facilities for Materials Processing in Space

2.1 In Situ Observation Facility by Optical Techniques

Optical observation with visible light is an important method to visualize the experimental process under microgravity conditions. Most of the optical observation systems are applied for the material experiment at room temperature or water solutions, so their hardware facilities can be much more comprehensive and sophisticated. They can be used in the investigation of fluid phenomenon as well as in the study of transparent materials, such as ice, succinonitrile, and protein crystal growth. Here, we will give several examples.

The PFMI on ISS. The Pore Formation and Mobility Investigation (PFMI) was conducted in the Microgravity Science Glovebox (MSG) onboard the ISS [15]. It was selected by the NASA Glovebox Investigation Panel and assigned to the Glovebox Program Office at the Marshall Space Flight Center (MSFC) on December 3, 1997. The PFMI hardware and samples were launched onboard Space Shuttle STS-111/UF-2 on June 5, 2002 and the first experiment was initiated on September 19, 2002.

The PFMI apparatus is a Bridgman type translation furnace with a main zone, a booster zone, and a cold zone. It is designed to process transparent samples such that the translating solid/liquid interface can be directly observed and recorded. Figure 7 shows a photograph of the PFMI ground test unit and the schematic of the PFMI sample installed in the MSG aboard the ISS. The maximum continuous operating temperature on the main and booster heaters is 130°C and the minimum temperature of the cold zone is 0 °C. The unique design allows the sample to be completely visible during all phases of processing. To view and record the sample processing, two cameras are mounted 90° apart on a translation system that is separate from the cold zone and electrode ring translation system.

The PFMI investigation utilizes an innovative approach for heating the sample. A thin, transparent layer of Indium Tin Oxide (ITO) is deposited on the exterior surface of the ampoule. The ITO coating is electrically conductive and acts as a resistance heater, effectively melting the sample, when current is applied. The PFMI is applied mainly to investigate the bubble formation from low-temperature melt materials, such as succinonitrile, a transparent material that solidifies in a manner analogous to metals [16].

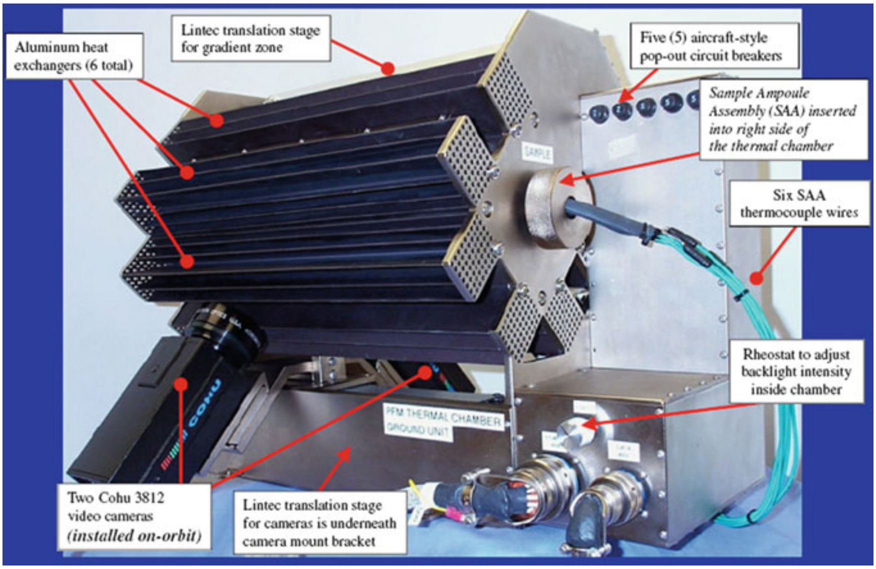


Fig. 7 Photograph of the PFMI ground unit, reprinted from reference [15]. Copyright 2018, with permission from Acta Astronautica

SCOF and EU on ISS. The Solution Crystallization Observation Facility (SCOF) was already set up to be used onboard ISS KIBO [17]. The facility was used for an ice crystal experiment and a phenyl salicylate crystallization experiment. For each experiment, the Experiment Unit (EU) was developed according to specific scientific requirements. The EU was connected to the SCOF, which provided electric power and communication lines to the EU. Cooling water (16–23 °C) was supplied to a cold plate on which the EU was installed.

The SCOF was equipped with an amplitude modulation microscope (AMT), a bright field microscope, a Mach-Zehnder type interference microscope with two wavelength light sources (532 and 780 nm), and two cameras (×2 and ×4). The field views were 2.4 × 3.2 mm and 1.2 ×1.6 mm. It enabled the in situ observation of the crystal growth process by using the Mach-Zehnder interference microscope. The EU that was developed specifically for this experiment was named "NanoStep".

DECLIC on ISS. DECLIC is a joint CNES/NASA research program implemented in the ISS. The facility can be used to study of materials science and the processing operations were performed in an ISS EXPRESS rack from 2006 to 2008 [18].

DECLIC comes in the form of two lockers, one electronic (ELL) and one experimental locker (EXL), accommodated in an EXPRESS rack inside the ISS. The EXL contains all the optics of DECLIC, which is divided in two sets: an optical emitter bench (OEB) and an optical receiver bench (ORB). In the OEB are all the light source, together with their optics and various devices. The OEB and ORB are mounted on a central titanium structure which gives the EXL mechanical and thermal stability.

The experiment is being led inside an insert located in the EXL. This insert contains the sample under study and all the associate scientific instrumentation in a given environment.

Two kinds of light source are available in the OEB: a He–Ne laser at 633 nm, and 6 red led (665 nm) with various beam shapes. Three cameras and one photodiode are available in the ORB. Cells containing the materials are placed together with their specific instrumentation (heaters, actuators, and scientific probes) in inserts that can be easily put in and removed from an experiment locker. This facility allows to study the directional solidification of transparent materials such as succinonitrile, too.

Chinese High-temperature In Situ Observation Instrument (HISOI). A High-temperature In Situ Observation Instrument (HISOI) is dedicated to visualize and record the whole oxide crystal growth with high temperature solution in space. This facility is developed by Jin et al. and served on the 17th Chinese recoverable satellite launched in the year 1996 [19] as well as served on “SZ-2” Chinese man-made spacecraft in the year 2001.

The HISOI is composed of three individual parts: (1) the space high-temperature microscope system, (2) the command and control system and (3) the video recorder system. The heart of it is a space optical microscope, by which both crystal surface and environmental melt flows can be observed. The Photo of HISOI and the experimental unit in it are shown in Fig. 8a. The test section is made by a heater chamber and a loop-like Pt wire heater. As shown in Fig. 8b, the Pt wire (ϕ 0.2 mm) is usually employed to heat and suspend the solution during the in situ observation experiment. A S-type thermocouple (ϕ 0.08 mm) is used to measure the temperature of the loop. The diameter of the loop is about 2 mm. Two V-typed electrodes are used to prevent the loop-like heater from deformation under high temperature. The maximal temperature of HISOI is up to above 1000 °C.

The video recorder system is a differential interference microscope coupled with Schlieren technique to visualize the crystal growth processes from the melt/solution. Figure 9 shows the schematic of the Schlieren optical system including two lens. The fore lens L1 is used to form parallel rays, and a knife edge is placed at the rear focal point of lens L2. Similarly, these parallel lights are also used to pass through the objective of the optical system of differential interference microscope. If a knife edge is installed at the rear focal point of the objective of the microscope, part of the light which has passed through the ununiform region of the object will be refracted and shielded, and the Schlieren effect can be obtained, i.e., the mass flow can be observed. With this method, the growth pattern and the mass transportation phenomenon can be visualized simultaneously. The video from the microscope is recorded and visualized by the video recorder system. Figure 10 shows the convection flow patterns observed by this facility in microgravity with temperature of 923 °C [20].

Chinese Optical Facilities for Materials Research at Low Temperature. To study the growth kinetics and structural transformation of colloidal crystals under microgravity condition, an experimental device with three crystallization cells, each with two working positions was designed [21]. It uses direct-space imaging with white light to monitor morphology of the crystals and reciprocal-space laser diffraction

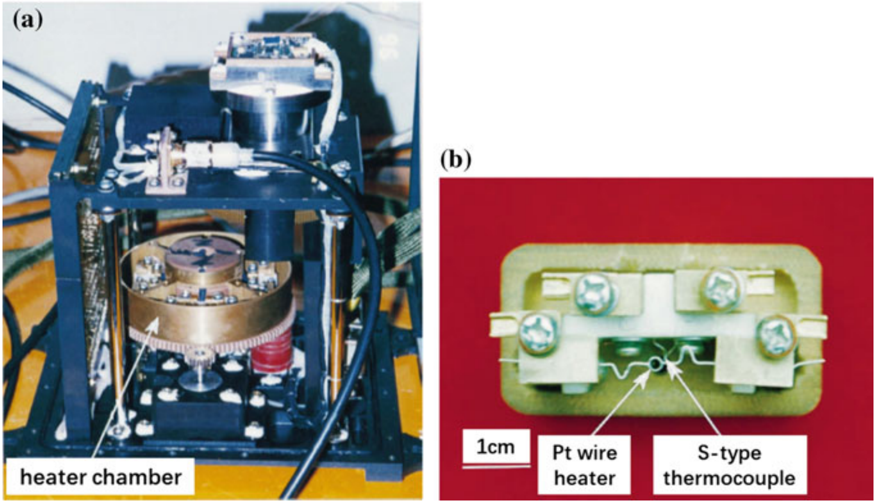


Fig. 8 Photo of Chinese HISOI **a** and the experimental unit in it **b**

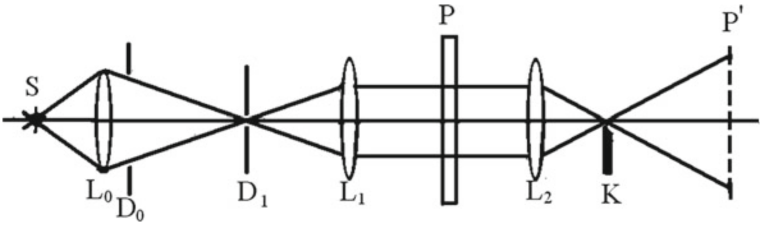
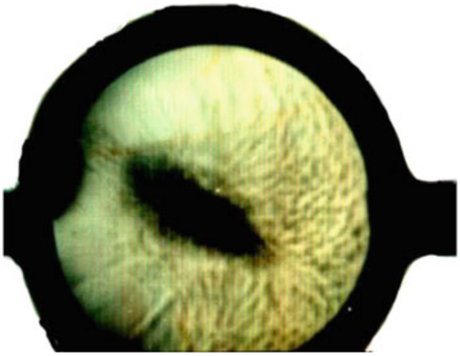


Fig. 9 Schematic figure of the Schlieren optical system for Chinese HISOI. S: light source; L0, L1, L2: lens; D0, D1: diagrams; P: specimen; K: knife edge; P′: image of P

Fig. 10 The convection photograph of the surface tension observed by Chinese HISOI in space



(Kossel lines) to reveal the lattice structure. The device has served for colloidal crystal growth on Chinese “TG-1” manmade spacecraft launched at September 29, 2011. Hundreds of images and diffraction patterns were collected via the on-ground data receiving station. The data showed that single crystalline samples were successfully grown on the orbit.

An optical in situ observation facility has been built to study the mass transfer process of water droplet in EAFP protein solution under microgravity condition provided by the Chinese “SJ-8” satellite [22]. This facility is based on the Mach-Zehnder Interferometer for imaging of liquid at room temperature. It can be applied to investigate the convective flows as well as the crystallization from water solution. So it is a convenient method for both fluid science and materials science.

2.2 In Situ Observation Facility by Other Techniques

Optical in situ observation technique can only applied to the investigation of transparent melt, which has much limitation. Some other diagnose techniques have been developed in microgravity for materials sciences investigation, such as X-ray radiography, infrared ray radiography, scanning probe microscopy, and diagnoses by Seebeck thermoelectric effect, and so forth. They are mostly designed for research of material processing especially for the non-transparent melt. Infrared ray radiography has been used in microgravity condition to obtain the temperature distribution of liquid and has not been applied to investigation of melt solidification. Scanning probe microscopy is used to observe the surface morphology of materials in nano scale. In addition, X-ray radiography and diagnoses by Seebeck effect have been used in space for investigation of alloy processing with high temperature.

In Situ Diagnose Technique by X-ray Penetration Method. X-ray penetration and image technique has been a very popular method on the ground for solidification research of metal or alloy. The solidi-liquid interface as well as the dendritic growth may be observed by this technique. However, in space it is much more difficult to observe the solidifying process of metal by X-ray image. In the frame of the XRMON (In situ X-ray monitoring of advanced metallurgical processes under microgravity and terrestrial conditions) project, which is a Microgravity Application Promotion (MAP) program of the European Space Agency (ESA), a facility dedicated to the study of directional solidification of aluminum-based alloys, with in situ X-ray radiography is developed [23, 24]. This facility, named XRMON-GF(GF for Gradient Furnace) device consists of a gradient furnace system for solidification of alloys and an attached high-resolution X-ray diagnostic system. The metallic samples had a sheet-like geometry with a length of 50 mm, a width of 5 mm and a constant thickness of less than 0.2 mm along the sample. Each sample was first mechanically polished down to the desired thickness (with a surface rugosity of 1 μm), and then spray coated with boron nitride (BN). Then it was sandwiched between two rectangular glass plates, welded together. The field of view

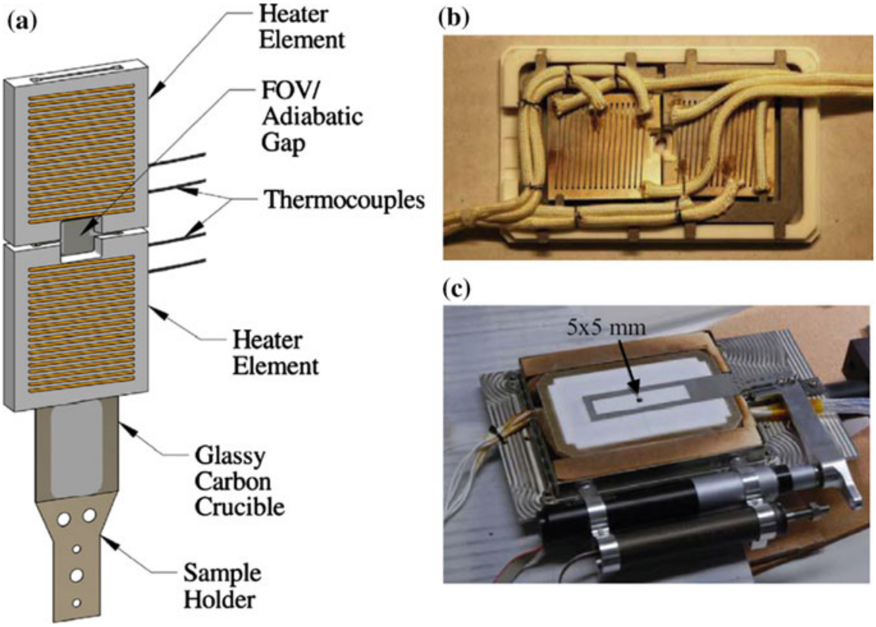


Fig. 11 **a** Schematic layout of the XRMON-GF furnace, **b** Pictures of the open furnace with the two heaters(top), **c** assembled furnace, with the hole for the X-ray radiation transmission, crucible frame placed on top to show sample placement, reprinted from reference [23]. Copyright 2018, with permission from Journal of Crystal Growth

for the X-ray diagnostics was about 5 × 5 mm^2 with a spatial resolution of 3–5 μm and a temporal resolution of 2–3 Hz. The X-ray source was based on a microfocus transmission target in molybdenum using polychromatic radiation with a peak at 17.5 keV. Figure 11 is the schematic layout of the XRMON-GF furnace.

This facility was successfully validated during the MASER-12 sounding rocket campaign, in spring 2012. Radiographs were successfully recorded during the entire experiment including the melting and solidification phases of the sample. The heaters are independently regulated by a PID-regulator implemented in software. The temperature of the facility can arrive above 500 °C.

In Situ Diagnose Technique by Seebeck Thermoelectric Effect. The interfacial morphology of alloys can be in situ diagnosed by the Seebeck thermoelectric effect [25]. This named Mephisto furnace is built under the auspices of the French nuclear energy commission CEA and the French space agency CNES. Mephisto furnace is a sophisticated Bridgman furnace with two heating and cooling sub-systems, where three separate samples are solidified in parallel. Sample 1 is dedicated to the measurement of Seebeck electric signal, which is used to obtain most of the experimental data presented here. A measurement of the electrical resistance is performed on sample 2. When the different resistivities of the base material between solid and liquid states are taken into account, this measurement can be translated in an estimation of the solidification velocity. In addition, sample 2 contains thermocouples for the measurements of the temperature gradients within the alloy. Sample 3 is for Peltier pulse marking and post-mortem analysis of the shape of the quenched growth interface. Sample 3 also contains thermocouples for temperature gradient measurements. Such experiments have been done on USMP1 and USMP3 flights.

Such Seebeck and resistance measurement device have also worked on board the ISS in 2004/2005 [26].

Scanning Tunneling Microscopy Technique. Scanning tunneling microscopy allows for atomic resolution imaging of surfaces and the observation of individual adsorbed molecules, as well as in situ studies of chemical reactions on an atomic scale. Tanja et al. built a scanning probe microscopy setup and it was carried out on a parabolic flight campaign in November 2001. This scanning probe microscopy setup is small, lightweight and do not require vacuum or high voltage supply. In addition, samples can be investigated directly without further preparation. By this facility, surface morphologies of graphite sample in nano scale were obtained in microgravity [27].

Facility for Electrical Resistivity Measurements of Melt. Some facilities are designed for the measurement of melt properties, such as diffusion coefficient, electrical resistivity. Here a facility for electrical resistivity measurement is introduced [28]. The experimental apparatus was developed for electrical resistivity measurements of liquid metals and alloys under microgravity due to the launch of rocket. Whole assembly, contained in the small space of 360 mm (length) × 360 mm (width) × 397 mm (height), was composed of the cell box, the electronic circuit box, and the battery box. The cell for electrical resistivity measurements due to the dc four probe method was made of Ti metal because of its strength and no reaction to corrosive liquid alloys at high temperatures. The application of this method was made successfully to the measurement of electrical resistivity for liquid Bi–Ga alloys with critical mixing both under gravity condition and under microgravity condition due to the launch of the rocket.

3 Facilities for Special Material Processing in Microgravity

Besides the high-temperature heating furnaces and the in situ observation facilities, which are the most popular two kinds of facilities for materials processing in microgravity, another kind of material experimental facilities are also typical. Such facilities are usually designed for crystal growth from low temperature solutions. For example, the α-$LiIO_3$ crystals were grown by the method of free evaporation of water solutions and evaporation with controlled condensation at a constant temperature. Experiments of α-$LiIO_3$ crystal growth were performed several times using the Chinese Scientific Recoverable-Satellites [29]. The zeolite crystal growth furnace unit is an integrated payload designed for low temperature crystal growth in solution on ISS [30]. These experiments are carried out mainly in solutions with temperature lower than 300 °C.

In addition, there are several other facilities that are developed for some special investigation of material experiments. Here are several examples in the following.

3.1 Containerless Processing Facility for Materials

To support several materials science experiments in microgravity, it is sometimes necessary to counter g-jitters and external forces imparted to bodies under study and to position accurately a sample in space. This requirement triggered the development of positioners or levitators that make use of different forces (e.g., electrostatic, aerodynamic, electromagnetic, acoustic).

Applying electromagnetic levitation under micro-gravity conditions the undercooled regime of electrically conductive materials becomes accessible which allows unique investigations of nucleation phenomena as well as the measurement of a range of thermo-physical properties both above the melting temperature and in the undercooled regime. Hence, based on a long and successful evolution of electromagnetic levitation facilities for microgravity applications (parabolic flights, sounding rocket missions and spacelab missions) the Electro Magnetic Levitator (EML) is developed by Astrium Space Transportation under contracts to ESA and DLR. EML is accommodated in the European Drawer Rack (EDR) within the European Module COLUMBUS on ISS. The design of the payload allows flexible experiment scenarios under ultra-high vacuum or ultra clean noble gas atmosphere individually targeted towards specific experimental needs and samples including live video control of the running experiments and automatic or interactive process control. EML can perform processing of samples with 5–8 mm diameter with temperature up to 2000 °C. The experiments by EML can be done under inert gas or ultrahigh vacuum possible.

However, the latest Electrostatic levitation furnace (ELF) for materials containerless processing is developed by JAXA, which is serving on ISS today. Before this ELF, a similar furnace was prepared hand worked under reduced gravity on-board a sounding rocket in 2000 by JAXA. The ELF on ISS by JAXA [31] consisted of a stainless steel chamber which was surrounded by diagnostics instruments (camera, pyrometers, etc.) and heating lasers. This furnace can handle evaporative materials over 3500 K. In JAXA's studies, samples with diameters ranging from 1 to 3 mm can be levitated. The ELF facility has launched onboard ISS in 2015 and is installed into the Work Volume (WV) of the Multi-purpose Small Payload Rack (MSPR) in the Japanese Experiment Module (JEM). And some experiments have been carried out [32]. Due to the limitation of space and electrical power, vacuum pumps necessary to get high vacuum environment have been omitted. This is one of the reasons why ISS-ELF will mainly process oxide sample.

3.2 Plasma Research Facility Under Microgravity

Complex plasma research under microgravity condition is one of the present key research topics in fundamental physics and materials science on ISS.

In 2001 the so-called PKE-Nefedov facility performed its first experiments in radiofrequency induced complex plasmas on ISS [33]. This bilateral German–Russian research facility operated successfully in over 13 missions nearly five years until its internal resources were consumed. The next generation experiment apparatus PK-3 Plus with refined and more powerful instruments and diagnostics had its on-orbit commissioning in January 2006. It is performing flawlessly since then and has already been successfully employed during various ISS increments including Russian cosmonauts and ESA astronaut Thomas Reiter.

Here PK-3 Plus is introduced (Fig. 12): the RF plasma chamber (1) is thermally improved to have a more evenly distributed temperature background in order to reduce thermo-phoretic effects; the RF generator (2) and the control possibilities for physical parameters are extended; the gas supply (3) was increased to two gases types: neon and argon; the number of cameras (4) was increased; three of the cameras (CCD, analog output) can be moved along a 2-axis translation table (5) allowing a much larger 3D analysis of the plasma; the particle injectors (6) are now mounted outside the electrode and their number was increased from 2 to 6, therefore allowing a larger variety of particle sizes; a small turbo molecular pump (7) is now integrated inside the experiment container.

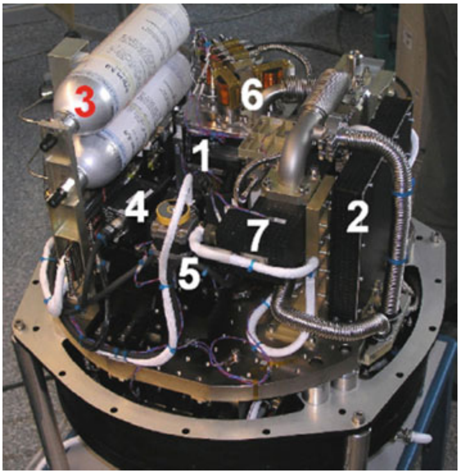


Fig. 12 PK-3 Plus experiment block integrated in experiment container, reprinted from reference [33]. Copyright 2018, with permission from Acta Astronautica

3.3 In-space Soldering Investigation (ISSI)

In-space Soldering Investigation (ISSI) was conducted in the microgravity environment aboard the ISS. The ISSI took place over four increments aboard the ISS in the maintenance work area (MWA). The investigation utilized commercially available soldering tools and materials to conduct a series of experiments and was conducted as "Saturday Science." The intent of the experiments was to look at joining techniques, shape equilibrium, wetting phenomena, and microstructural development in a microgravity environment. The desired zero up mass requirement a quick search of equipment and tools aboard the ISS found that a well-equipped soldering and pin kit was available. The components are all off-the-shelf items which facilitated ground testing. The ISS soldering iron was adapted for battery usage, reaching a maximum temperature of 600 °F [15].

3.4 Facilities for Thin-film Material Fabrication in Space

Materials development can also occur in space based on the presence of vacuum. The utilization of the vacuum of space for thin-film materials development has been pioneered by the Wake Shield Facility (WSF) [34]. The WSF is a 3.7 m diameter disc-shaped free-flying platform designed to generate an ultra-vacuum in low earth orbit space, and to utilize that ultra-vacuum for the fabrication of thin-film materials by epitaxial growth. The WSF was designed, built, deployed and operated by the Space Vacuum Epitaxy Center and its industry and government. The WSF is transported to orbit by the Shuttle in its payload bay mounted on a specifically designed cross-bay carrier. The carrier not only restrains the WSF in the payload bay, but also protects the central epitaxial growth region of the wake side from pre-launch payload processing environments, and payload bay launch environments through the incorporation of an integral vacuum chamber.

The thin-film growth apparatus on the WSF includes eight source cells (small furnaces) for the evaporation of elemental atoms for MBE thin-film growth, and two gas nozzles for the effusion of organometallic gaseous species for CBE thin-film growth. The WSF has flown several missions, STS-60 (February 3, 1994), STS-69 (October 11, 1995), and STS-80 (November 4, 1996). In the three flights of WSF, high-quality GaAs-based epitaxial thin-films were grown.

In references [35, 36], one experimental reactor cell designed and developed for the growth of films and crystals by closed cell physical vapor transport on the Space Shuttle Orbiter is discussed. The cell design has been proven by over 60 experiments, including 18 on two space flights, to be very satisfactory from the standpoints of minimal power use at operating temperatures of 400 °C, mechanical vibration resistance and approximation to ideal steady-state growth conditions.

4 Summary

Experimental facilities are very important for the research of materials science in space. Up to now several kinds of experimental facilities have been prepared, including many high temperature heating furnaces, in situ observation facilities and some specially designed facilities for materials science research.

Heating furnace is one presentative kind of facility for materials science research in space since many material experiments require high temperature environment. Almost all the countries or groups in the world aiming at microgravity materials science study have built their own heating furnaces serving in space, such as Russia, America, Japan, ESA, and China. Most of these furnaces work through heat generation from resistance when applied electricity due to its comparatively high stability. Generally, such heater furnaces are designed as Bridgman styles which allow the investigation of single crystal growth or directional solidification of metals possible for samples with large size. The maximum of working temperature is the most important parameter characterizing the ability of heating furnace. Most of the recent heating furnaces can heat up to more than 1000 °C. The largest value can even arrive at 1600 °C. The sample diameter usually is larger than 15 mm.

As for the in situ observation facilities in space for materials science research, optical imaging method by visible light is the most popular technique owing to its convenience. Several facilities for process visualizing by other techniques are also developed and used in space, such as X-ray penetration method, and diagnoses method by Seebeck thermoelectric effect. Most of the in situ observation facilities can only be used at low temperature, applying to the investigation of solidification or crystal growth from water solution. Only a little can work at temperature up to several hundred centigrade. So newly kind of in situ observation facilities for more higher temperature should be designed in the future.

The following features can be obtained for the materials science processing experiment furnace in microgravity developed recently: highly integrated synthesis experiment ability such as preparing materials, measuring, analyzing, with help of external force such as magnetic field; high precision for temperature control and sample movement control; ability of arriving higher temperature; stability and long lifetime; modularization, standardization and assembled, which allows the facility to be exchanged more conveniently.

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