Versatile compact X-ray radiography module for materials science under microgravity conditions.

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Abstract. A versatile compact microfocus X-ray radiography facility is presented. The facility serves as a technology demonstrator showing the applicability of X-ray radiography to experiments in space. It has been designed as an insert fully compatible with requirements of the Materials Science Laboratory aboard the International Space Station. The facility consists of a microfocus X-ray source delivering up to 20 W X-ray power at 100 kV acceleration voltage and a 49.2 × 49.3 mm RadEye2 sensor with a Scint-X scintillator at 48 μm per pixel resolution with a 14 bit dynamic range. The total device weight including sample chamber is 43 kg. The facility is classified as a fully protected radiography equipment according to German radiation safety laws. The capabilities of the facility for research in materials science are demonstrated in ground-based experiments.

1. Introduction

In recent years in-situ studying of processes became increasingly important for various fields of science. In materials science the in-situ study of materials and processes enables to discover not only new phenomena but also to revise previously obtained results sometimes subject to a larger error. In-situ methods hereby greatly benefit from the improved and novel experimental equipment developed in recent years.

In materials science and here in particular in the area of research on metal-based materials the advent of fast high resolution area detectors and highly brilliant synchrotron sources enabled to study of phenomena in the area of solidification on real samples [1, 2, 3] and not only on the previously used more easily accessible transparent analogue materials [4]. With the advent of microfocus X-ray sources in-situ studies are also possible in the laboratory as demonstrated for studying diffusion [5] and even under microgravity as shown for foaming of metals [6, 7, 8]. The latter studies are part of the X-ray monitoring (XRMON) project set-up as part of the ESA-MAP programme. The project focuses not only on the investigation of foaming but on a number of other processes in metallic melts that greatly benefit from in-situ analysis like e. g. diffusion and solidification.

In metal research experiments under microgravity are essential to study processes without being influenced by unwanted effects caused by the presence of the gravitational force as e. g. buoyancy-driven convective flow or sedimentation. Hereby, various possibilities exist to achieve
Figure 1. Left: Photograph of assembled fully MSL compatible X-ray radiography insert. The access port to the X-ray source compartment is opened with the turbomolecular pump visible at the top and the X-ray driving electronics at the bottom. Water cooling pipes are shown at the top cooling the X-ray front end. Cable feed through ports on the sample chamber are not mounted with the access holes covered by lead shielded blanks. Right: Photograph of the X-ray insert installed in the MSL equivalent processing chamber. Water cooling pipes connected to the detector are visible.

microgravity conditions. Amongst those are drop tower, parabolic flight, sounding rocket, and satellite experiments as well as experiment on the International Space Station (ISS). The order of the platforms is according to increasing microgravity time available which ranges from a few seconds to days.

Radiation safety requirements initially precluded the use of X-ray radiation in microgravity research due to bulkiness and weight constraints of the source as well as the heavy metal shielding required. However, as demonstrated in parabolic flight campaigns [9] but more recently also aboard sounding rockets (MASER-11 May 2008 [10], MAXUS-8 March 2010, and MASER-12 November 2011) microfocus X-ray sources are sufficiently compact to enable operation on these platforms. Sealed-tube microfocus X-ray sources have been used so far. However, these sealed-tube sources show a finite lifetime and a pre-defined target-substrate combination. Therefore, they might not be an optimal choice considering research in the MSL aboard the ISS, where long-time usability and flexibility of such a device is a key factor.

Therefore, the development of a compact but flexible microfocus X-ray facility that meets requirements of research under microgravity conditions and in particular demands by the MSL aboard ISS is mandatory to enable increased future use in in-situ studies of materials under microgravity conditions. Here, such a facility that meets not only scientific requirements in terms of source power, resolution, and image depth but also technical and safety requirements for operation aboard ISS is presented. The facility was developed by Astrium GmbH together with Viscom AG and partners. The previously mentioned key factors are met by using an actively pumped X-ray tube as part of the facility. The here discussed facility has a total weight of only 43 kg and can be safely operated in any laboratory. The facility is sufficiently flexible to allow for operation on different other microgravity platforms with minor adaptations.

2. Facility Parameters

Figure 1 left shows a photograph of the X-ray radiography insert. Figure 1 right shows the insert integrated into the MSL compatible processing chamber. The insert consists of three
Figure 2. 2D section view of the X-ray radiography insert. Cathode (red), X-ray focusing optics (blue) and target (green) on the right of the figure. The X-ray driving electronics is represented in light blue and the high voltage generator in light green, whereas the turbomolecular pump is represented in yellow. The sample compartment is surrounded by lead shielding (thin dark-red layer). The volume occupied by the sample environment is given by the semitransparent green area. The detector module is shown to the left with the detector being the dark/light grey structure close to the beam entrance window.

The X-ray source entity consists of an outer housing acting both as electrical as well as radiation protection. The housing is vacuum tight, thereby ensuring ambient conditions required for safe operation of the X-ray tube. Communication, power supply, and safety interlock cabling as well as a pre-vacuum line are fed through a vacuum flexible tube which can be connected via a standard KF25 flange to a flange adapter at the inside of the MSL chamber. Thereby the X-ray housing is protected from the MSL chamber atmosphere may it be under vacuum or partially pressurized. Operation outside the MSL chamber does not require this connection. The housing contains the X-ray tube, its high voltage generator, and the X-ray tube driving electronics. An adapted version of the standard X9100 tube by Viscom is used. The X-ray tube is fixed to the housing from the detector side. To the back of the tube the high voltage generator is connected. The only connection required is a communication cable and a power supplying cable delivering power at a voltage of 40V to the X-ray tube electronics board. The target currently in place consists of a 5 \( \mu \text{m} \) thin tungsten layer on a 200 \( \mu \text{m} \) thin aluminium window. It can be easily replaced by other target materials or by a Beryllium substrate depending on required source characteristics. The target is located behind a circular aperture matching the X-ray cone-beam size to the detector size. Thereby unwanted X-ray radiation disturbing either image quality or requiring more substantial shielding is avoided. Further shielding is included inside the housing at the radiation safety relevant positions. Thereby X-ray emission in backward direction through the housing is suppressed far below required limits. The tube is actively pumped via a compact turbomolecular pump in combination with a diaphragm pump. For continuous operation active cooling is required. This is guaranteed by thermal connection of the critical parts to the front plate of the X-ray housing which is actively cooled by chilled-water. The X-ray source of the insert delivers radiation at a maximum acceleration voltage of 100 kV and at a maximum total tube power of 20 W. Continuous operation of the source is possible up to 80 kV at maximum
tube power. At 100 kV and maximum tube power the source can be operated for up to 20 min before it is switched off required for cooling of the X-ray driving electronics. The electron-beam size ultimately defining the X-ray beam size is continuously adapted according to the X-ray power supplied by the source. For low power operation (< 4 W) the smallest beam-size is below 3 µm and therefore the best resolution can be achieved. At increasing power the electron-beam size is increasing at the expense of resolution. With the current source detector configuration power values beyond 15 W lead to a marked increase in electron beam size negatively impacting on image resolution. Increasing of the electron beam size with increasing power is required to not overheat and damage the target. Target deterioration over time can be accounted for by rotating the target around its surface normal. With the electron beam slightly off-center with respect to the target rotation axis fresh parts of the target can be brought into the electron beam. Two independent X-ray safety interlock switches are mounted besides the X-ray beam aperture in the X-ray tube housing front end. The matching interlock parts are installed to the inside of the sample environment chamber. Hereby, safe operation of the source is ensured with the beam path fully enclosed by the sample environment chamber and the detector module as shown in figure 5. Most importantly, the insert is according to German radiation safety laws classified as a fully protected radiography equipment, i.e. the insert can be safely operated in the lab without any further radiation shielding. Hereby, the acceleration voltage is limited to a maximum of 80 kV at maximum tube power. Within the MSL chamber operation is safely possible up to the maximum acceleration voltage of 100 kV at maximum tube power of 20 W. Any staff operating the facility is in both operation conditions, in the lab or in the MSL chamber, not required to wear any radiation badges.

The detector is housed inside a vacuum tight aluminium chamber. Vacuum tightness is achieved by mounting to the sample environment chamber. The detector chamber is clad by a thin layer of lead (3 mm thickness) at the backplate. The shielding absorbs the primary X-ray radiation transmitted into the chamber through the rectangular carbon-fibre window. The minimal selectable thickness of the carbon-fibre window is 0.3 mm. The detector consists of two RadEye\textsuperscript{TM} 100 image sensors mounted side by side in RadEye2 configuration. To the CMOS sensors a protective fibre optics face-plate (FOFP) is mounted. The FOFP serves as radiation protection for the sensor. Onto the FOFP a Scint-X scintillator is glued. The detector provides an active detection area of 49.2 x 49.3 mm at 48 µm pixel size resulting in a total of 1024x1024 pixels. The detector is actively cooled by a Peltier element. Active cooling to a temperature of 16 °C significantly reduces the detector noise. The hot-end of the Peltier element is actively cooled by connecting the detector chamber to the chilled-water circulation. The detector housing has two vacuum tight feed-throughs. One feed through is responsible for supplying the power for the Peltier elements and the detector. The other feed through is required for communication with the detector. Detector communication occurs via a USB 2.0 read-out interface connected to the detector and housed inside the same aluminium chamber. A continuous frame rate of 2 frames per second with a depth of 14bit is achieved with this connection. The detector housing is mounted to the sample environment chamber back-end with six nuts that can only be opened with a special tool. The detector housing whilst mounted to the sample environment chamber is vacuum tightly sealed by means of an O-ring.

Currently two sample environment chambers are available. Both chambers are made of stainless-steel and are shielded by a layer of lead of 3 mm thickness. Both chambers have a wide opening of diameter 190 mm at the front-end enabling easy integration of the sample environment. The chamber back-ends are sealed by a carbon-fibre window of 1 mm thickness. Currently available source to detector distances are 150 mm. This means that with 1 cm distance between sample and source a maximal magnification of 15 and therefore the resolution limit given by the minimal X-ray spot-size can be achieved. Correct positioning of the sample environment chambers to the X-ray tube housing is facilitated by two ground locating pins and bushings
made of quenched steel. The MSL sample environment chamber enables the installation of two lead-clad labyrinth like cable ducts at the back-end of the chamber. These cable-ducts are required to supply the respective sample environment installed inside of the chamber. The on-bench chamber is equipped with five welded ports equipped with KF flange connectors.

The total combined weight of the MSL-compatible version of the insert consisting of tube housing, MSL-compatible sample environment chamber, and the detector module amounts to only 43 kg.

The X-ray source is operated by the VxC software of Viscom. The software enables setting of acceleration voltage and beam current within the source operation limits. X-ray tube conditioning, filament warm-up, and beam-centering procedures can be carried out. For image recording the software XMC is used. This software allows in-situ correction of the recorded images for background and differences in pixel detection sensitivity. Recording time per image, averaging of images, and a number of other standard options for image manipulation and recording can be set.

3. Evaluation Experiments

Different test procedures were conducted to evaluate the performance of the equipment.

3.1. Modulation Transfer Function

The modulation transfer function shown in figure 3 of the detector with the Scint-X scintillator was evaluated by Viscom. Evaluation was carried out by using a tungsten edge. Hereby the testpattern was directly mounted to the detector conducting the test in magnification $M = 1$ configuration. Hence, the per pixel resolution of the detector rated to be about 10 linepairs per mm is probed. The X-ray source setting was chosen such that the X-ray beam size at the origin is less than the detector pixel size. The MTF for the present scintillator can be compared with the standard scintillator MTFs usually delivered with the RadEye2 sensor (cf. figure 3). It clearly shows that the present scintillator outperforms the standard scintillators.

To achieve such high resolution with high contrast is only possible due to the scintillator used. The scintillator consists of a honeycomb patterned silicon substrate filled with the scintillator material. Only light in forward direction within a relatively tight angle of acceptance is transmitted through the honeycomb pattern. Pixel-cross talk is thereby avoided to a large extent. The downside is the approximately 50% reduction in transmission. However, due to the short detector-source distance of 150 mm only, this is a minor problem compared with the major gain in image sharpness. Further improvement of the scintillator technology is expected over the years to come increasing transmission efficiency.
3.2. Sample Measurements

An interdiffusion experiment was carried out on a AlCu10at% vs. AlCu15at% diffusion couple at a processing temperature of 700 °C using the standard long-capillary diffusion X-ray radiography setup described elsewhere [5, 11]. The diffusion couple was mounted inside the graphite furnace. For this test detector and source were operated in separated configuration inside a lead box in order to be able to use the standard capillary setup. Hence, the source detector distance was increased from the nominal 150 mm in flight configuration to 390 mm for the test. The capillary was mounted such that a magnification factor of \( \sim 1.9 \) was achieved. Effective pixel resolution amounts therefore to \( \sim 26 \mu m \).

For the experiment the sample was pre-heated to 510°C in several steps. The sample was kept at pre-heat temperature for about 5 min. It was then heated to a temperature of 700°C with about 7 K s\(^{-1}\) at which the diffusion experiment was conducted. This temperature was stabilized. The sample was kept at this temperature for \( \sim 10 \) min followed by cooling to ambient temperature by switching of the heating power.

Recordings were taken from the start of heating to the diffusion temperature until the sample had fully solidified upon cooling. Images were taken with a frame rate of 1 Hz. The X-ray source was set to 80 kV and 170 \( \mu A \) beam current.

![Figure 4.](image)

**Figure 4.** Left: Cu concentration as a function of position in the diffusion couple at different times: \( t_1 \) solid and \( t_2 \) later stages of diffusion. Right: Recorded images of the diffusion capillary for the same times (top \( t_1 \) and bottom \( t_2 \)) and during melting (middle).

Images of the still solid diffusion couple, the diffusion couple during early stages, and during later stages of the diffusion anneal are shown in figure 4 on the right. The images were subjected to standard correction procedures consisting of background subtraction and pixel gain correction. From the recorded images grey value profiles were derived by integrating radially over the diffusion pair. The axial grey value distribution can be transformed to Cu concentration following procedures outlined in Ref. [5]. The concentration profiles corresponding to the recorded images for the solid sample and during diffusion annealing are shown in figure 4 left. Comparing the diffusion profiles with profiles obtained using a RadEye detector with standard scintillator and without cooling it turns out that though the X-ray intensity being detected is lower contrast resolution is increased by 50% and contrast to noise ratio is about similar.

Besides the described interdiffusion experiment monitored in real time images of two different solid samples have been recorded. A solidified Al-Cu alloy subjected to equiaxed growth was provided by N. Mangelinck-Noel and A. Bogno of the IM2NP-CNRS in Marseille. The specimen was 30x5x0.1 mm in dimension. Recordings with various source settings showed that images can be recorded in real time with 2 Hz repetition rate providing at the same time better resolution than recordings taken with a standard detector. In this sample the dendritic growth pattern was clearly visible. For the high magnification low tube power setup it might even be possible to follow Cu concentration from the dendrite into the surrounding liquid with an effective per pixel.
resolution of $\sim 4 \mu m$. Further, images were taken for a solidified Al-Cu foam sample provided by F. Garcia-Moreno from Helmholtz Center Berlin, Germany. Again it can be shown that similar image quality at a slightly better resolution than with a conventional detector can be achieved at 2 Hz image repetition rate. Edges of bubbles in the foam sample are clearly visible. The entire sample of dimensions of $20 \times 20 \times 1$ mm can be recorded at a magnification factor of about 2.5 resulting in an effective pixel size of between $20 \mu m$ and $24 \mu m$.

In future, for both, the solidified AlCu dendrite and the solidified foam sample, intensity and therefore the contrast could be increased by using a different target, as e. g. molybdenum on aluminium thereby increasing the X-ray intensity at lower X-ray energies. Direct-converting detectors might also be an option provided they reach image stability and quality currently achieved with scintillator-based detectors and a roughly comparable total detector area. In this case the signal would be increased by at least a factor five enabling either low-power high-resolution operation of the X-ray source at better image quality or faster imaging rates.

4. Conclusion
In conclusion a novel microfocus X-ray demonstrator insert for the Materials Science Laboratory aboard the International Space Station has been presented. Characteristic features of this facility were presented in detail and results of first tests of operation conditions and imaging capabilities of this device were discussed. The X-ray source can be operated at acceleration voltages up to 100 kV at a maximal power of up to 20 W. Images of $1024 \times 1024$ pixel at a pixel resolution of $48 \mu m$ and with a depth of 14 bit per pixel can be taken at up to two frames per second. The detector provides a field of view of $49.2 \times 49.3$ mm. The detector uses a RadEye2 image sensor in combination with a Scint-X scintillator. The CMOS sensor is shielded by a fibre optics face plate enabling irradiation with higher energy X-rays. Image resolution is increased compared with the available standard scintillators. First radiography results of an interdiffusion experiment on an Al-Cu alloy have been presented. The detector showed an increased contrast resolution at a decreased contrast to noise ratio. It has been demonstrated that this novel equipment is able to provide similar results as previously obtained in laboratory experiments using a less integrated piece of equipment. Improvements in detector technology over the coming years are expected to
enable faster recordings with higher intensity as currently encountered. The insert is currently slightly modified to enable its use aboard the DLR sounding rocket MAPHEUS in 2012 together with the recently designed and tested compact shear-cell furnace [12] for studying diffusion in metallic melts.

Acknowledgments

We thank the German Ministry for Economy and Technology (BMWi) for funding of this project through the Economic Stimulus Package I. We thank Nathalie Mangelinck-Noel and Abdul Aziz Bogno of the IM2NP-CNRS in Marseille for providing us with a solidified AlCu sample as well as Francisco Garcia-Moreno of the Helmholtz Center in Berlin for providing us with an Al-Cu metal foam sample. We thank Henning Weis for critically reading our manuscript.

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