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XRMON-SOL MICROGRAVITY EXPERIMENT MODULE ON MASER 13

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ABSTRACT

The XRMON-SOL microgravity experiment observed spatially isothermal equiaxed solidification of an Al–Cu alloy in microgravity on board the MASER 13 sounding rocket, launched in December 2015. It is the first time that isothermal equiaxed solidification of a metallic alloy has been observed in situ in space, providing unique benchmark experimental data.

The experiment used a newly developed isothermal solidification furnace in the re-used module of the MASER 12 experiment XRMON-GF. A grain-refined Al–20 wt%Cu sample was fully melted and solidified during 360 s of microgravity and the solidification sequence was recorded using time-resolved X-radiography. Equiaxed nucleation, dendritic growth, solutal impingement, and eutectic transformation were thus observed in a gravity-free environment.

This paper describes the technology development of the experiment module

1 BACKGROUND

1.1 XRMON project under MAP programme

The XRMON (<u>X-Ray Monitoring</u>) project under the ESA MAP (Microgravity Applications Promotion) programme was established to conceive and perform in situ X-ray radiography observations of metallurgical processes under microgravity and terrestrial conditions. Under the programme, a series of experiment modules and facilities have been developed and advanced experiments of metallurgical processes have been performed with X-ray radiography observations under microgravity conditions. These include:

- ✓ <u>XRMON-M</u>etal <u>F</u>oam: Study of metallic foams, MASER 11 2008, two parabolic flight campaigns in 2007 and 2009
- ✓ <u>XRMON-Diff</u>usion: MAXUS 8 sounding rocket in 2010
- ✓ <u>XRMON-G</u>radient <u>F</u>urnace: MASER 12 sounding rocket in 2012
- ✓ XRMON Laboratory Set-up in 2012

- <u>XRMON</u> <u>Parabolic</u> <u>Flight</u> <u>Facility</u>: Four flights during 2013-2016
- ✓ <u>XRMON-SOL</u>idification Furnace: MASER 13 sounding rocket in 2015
- ✓ <u>XRMON-Diff</u>usion <u>2</u>: MAXUS 9 sounding rocket in 2017
- <u>XRMON-G</u>radient <u>F</u>urnace 2: MASER 14 sounding rocket, under pre-study

1.2 XRMON-GF Experiment Module

Within the above activities, XRMON-GF was the first ever sounding rocket experiment with in situ X-ray monitoring of alloy solidification during flight [1]. The consequent XRMON Laboratory Set-up, XRMON-PFF and XRMON-SOL inherited the same overall system design of the XRMON-GF module. All of them use (with or without adaptation) the same compact X-ray imaging system developed under XRMON-GF project.

The XRMON-GF module was designed with the aim of being possible to be re-used. XRMON-SOL is the first re-fly of the XRMON-GF module, albeit with a very different experimental furnace design. The following chapter will focus on describing the adaption made to the module to accommodate the XRMON-SOL experiment and the design improvement implemented after the XRMON-GF project.

1.3 XRMON-GF Furnace

The XRMON-GF Furnace, developed under XRMON-GF project, is a gradient furnace of Bridgman type with two identical heaters for the "hot" and "cold" zones (Figure 1). It was designed for columnar solidification experiments [2].

The furnace has been accommodated in XRMON Laboratory Set-up and XRMON-PFF. Nearly isothermal solidification experiments have been performed by applying the same temperature to both heaters in XRMON-GF furnace. Experiments have been performed by using these 2 set-ups both on ground and in Parabolic Flight [3].



Figure 1. Schematic layout of the XRMON-GF furnace

However, a temperature gradient has been observed in the sample when attempting to perform isothermal solidification with the XRMON-GF furnace. The furnace design does not enable a temperature field which is isothermal in the Field of View (FoV); this is needed for equiaxed solidification [2].

2 XRMON-SOL EXPERIMENT MODULE

2.1 XRMON-SOL Furnace

The major activity in the XRMON-SOL project was to develop the XRMON-SOL furnace for equiaxed solidification experiment.

Based on the experience of XRMON-GF furnace, a completely new furnace, XRMON-SOL, was needed. It was to have rotational symmetry. It was designed to have one heater body (Ø70 mm) with eight heaters arranged so that 4 heaters form the inner ring and 4 heaters form the outer ring (Figure 2). Heating wire is wound as coils on the heater body. The inner ring heaters and the outer ring heaters are displaced by 45° so that the outer ring heater will guard the gap between inner ring heaters to minimize the heat leakage through the gap.



Figure 2. Cross-section of heater body showing placement of thermocouples



heater zone. Figure 2 shows also the placement of thermocouples.

After heater wires and thermocouples have been assembled (Figure 3), a sample holder with sample compartment is placed on top of the heater body. The alloy sample is placed between 2 glassy carbon crucible sheets and placed in the sample compartment (Figure 4). The sample has a diameter of 23 mm and the sample thickness is 0.2 mm. A sample lid is placed to cover the sample compartment and to hold the sample in place (Figure 5). Heater body, sample holder and sample lid are made from a special unidirectional sintered ceramic, to achieve good thermal conductivity and X-Ray transparency.



Figure 3. Heater has been assembled



Figure 4. Sample holder and the flight sample, photo was taken after payload retrieval



Figure 5. Sample lid in place



Figure 6. Part of cross-section of heater body, sample holder and sample lid showing placement of inner ring thermocouples

Efforts have been made to place the inner ring thermocouples as close as possible to the sample. Figure 6 shows part of the cross-section of heater body, sample compartment and sample lid. It shows that the inner zone thermocouples are placed less than 2 mm from the sample.

Figure 7 shows the complete assembled XRMON-SOL furnace. The furnace lid is made out of a tungsten alloy, with glassy carbon thermal shielding, in the X-ray window. It is insulated towards the furnace.



Figure 7. Flight furnace complete assembled

2.2 Furnace Temperature Regulation

Heaters and thermocouples are numbered according to Figure 8. Heaters 1 to 4 are inner ring heaters and heaters 5 to 8 are outer ring heaters.

For fine-tuning of solidification process, all 8 heaters are individually regulated. For the simplicity during operation, only two target temperatures (regulation setpoints) are used. The inner ring heaters use the inner ring set-point and the outer ring heaters use the outer ring set-point. Hence a temperature offset could be set for each heater. The offset could be used to compensate the eventual temperature difference caused by tolerance of the furnace or the sample. Offset of all heaters were set to zero during the flight, which means the furnace and the sample were manufactured very precisely to provide isothermal condition.



Figure 8. Heater and thermocouple numbering

A new design of sensor board and PWM control board has been implemented since XRMON Laboratory Setup.

The multiplexer before thermocouple amplifier on the sensor board was removed, as multiplexing the thermocouple amplifier limits the sampling frequency. Instead, each thermocouple gets a dedicated thermocouple amplifier. Microcontroller was also upgraded from an 8-bit MCU to a 32-bit MCU. The sampling frequency had been increased and a better software filter was used. The 24-bits temperature data from output of the software filter was used in the temperature control loop.

Also the 8-bit microcontroller on the PWM control board has been upgraded to 32-bit microcontroller. After the upgrade, PWM resolution was increased from 1000 in XRMON-GF module to 10000. The PWM frequency could be decreased from 1250 Hz to 25 Hz which dramatically reduces the switching loss of the output driver.

To adapt to this improvement, XRMON-GF sensor board was replaced by XRMON-PFF sensor board. XRMON-GF PWM board was replaced by a new PWM board that has 8 PWM outputs. The new PWM contains 2 microcontrollers to control 4 PWM outputs each. A general time synchronization mechanism via CAN bus has been implemented. The synchronization accuracy is less than +/- 2 μ s. Based on time synchronization, the 8 PWM outputs are divided into 4 groups with 2 outputs in each group. The groups has PWM phase shift at 0°, 90°, 180° and 270°. Each group drives 1 inner ring heater and 1 outer ring heater. In this way, the power consumption is more equally distributed.

Table 1 shows the temperature control error the first 50 s after lift-off when the furnace temperature was set to

540 °C. It shows that the temperature control is very accurate and stable for all heater zones.

	Mean (°C)	Std (°C)
TC1	-0.002	0.027
TC2	-0.001	0.026
TC3	-0.004	0.029
TC4	-0.003	0.031
TC5	-0.018	0.025
TC6	-0.010	0.030
TC7	-0.015	0.029
TC8	-0.013	0.030

Table 1. Furnace temperature control error, from liftoff to 50 s in flight, set temperature 540 °C

2.3 X-Ray Imaging

Although the same X-Ray and X-Ray camera were used, the sample placement for XRMON-SOL has been adapted to the FoV requirement of XRMON-SOL.



Figure 9. X-Ray image projection illustration

Figure 9 illustrates the relation between X-Ray source, the sample and the camera. The distance from the source to sample is 5.7 mm and the distance from the source to CCD sensor is 46.9 mm. This gives magnification factor 8.2. Consider that the effective pixel of the CCD sensor is 18 μ m (CCD size 24x36 mm, 2012x1340 pixels @ 2x2 binning), the virtual pixel size is 2.2 μ m in the sample. The actual spatial resolution [4], which is a function of the scintillator pitch (50 μ m) and the source size (3 μ m), was calculated as ~ 6.3 μ m.

2.4 XRMON-SOL module

After the above mentioned modifications (include the entirely new furnace design) and a few other improvements, the XRMON-GF module became XRMON-SOL module. It is worthwhile to mention that in order to meet requirements on general payload mass reduction, the total mass was reduced from 103.7 (XRMON-GF) to 99.3 kg. Extensive testing has been

performed to verify the performance of the furnace and the module [5].



Figure 10. XRMON-SOL Module

3 XRMON-SOL FLIGHT ON MASER 13

MASER 13 sounding rocket, with XRMON-SOL on board, was launched on Dec. the 1^{st} , 2015. It reached apogee of 261.7 km and provided good microgravity levels about 10^{-4} g for 359 seconds.

XRMON-SOL module was controlled by a preconfigured time-tagged tele-command queue. XRMON-SOL experiment was nominal during the count-down and the flight. No manual intervention was need.

Figure 11 shows the flight time line by using the actual furnace temperature data during the flight. Time 0 is the rocket lift-off time which was 2015-12-01 05:00:00 UTC. The microgravity time is from 89 s to 448 s. Because of the accurate furnace temperature control, time line could simply be defined by the inner ring and outer ring set-point. The alloy was melted and resolidified within the microgravity time window.

The experiment started at T-15 min, heating the sample to 540 °C at the rate of 2 K/s. At 60 s after lift-off (Figure 11), heating to 650 °C at 2 K/s was commenced. After an initial holding/stabilisation period of about 20 s, the sample was cooled to standby temperature of 625 °C at 0.9 K/s and 1.1 K/s on the inner respect the outer heater ring. After a second holding/stabilisation period, the first stage of the solidification experiment commenced at 180 s by applying cooling rate of -0.05 K/s to all eight heaters simultaneously. At 359 s, the second stage of solidification, i.e. the eutectic transformation, started by increasing the cooling rate to -1 K/s on the inner ring and the -1.5 K/s on the outer ring. All heaters were switched off at 435 s.



Figure 11. Experiment temperature profile / Time line

Because of the slow cooling rate of -0.05 K/s, it is important to identify the actual solidification temperature of the sample. Missing one degree means loss of 20 s of microgravity time. Therefore, sample conditioning was planned at the beginning of launch campaign. Sample conditioning uses a tele-command queue similar to the flight tele-command queue, but with bigger temperature and time margin. Sample solidification start temperature and eutectic transformation temperature (where all remaining liquid disappears) could be identified via the X-ray image. Then the new standby temperature and time to start the first and the second solidification stages could be calculated. The flight tele-command queue was updated accordingly.

It is important that nucleation of solid would not occur until the planned start of the first solidification stage. Therefore it is critical that the actual temperature should not drop below the standby temperature before the slow cooling of -0.05 K/s had been applied. The temperature regulation had been fine-tuned to avoid over/under shoot (Figure 12).



Figure 12. Standby temperature, commenced temperature and actual temperature

The uncompressed 12-bit X-Ray image was saved at 3 frames/s on board. Compressed image was down-liked live at 1 frame/s during the flight. The compression ratio is about 10 which is less than that of XRMON-GF. The down-linked images were already deducted by a reference image taken after sample melting. Figure 13 shows some examples of down-link images. Study [6] shows that observable solidification within the FOV occurred between 241 s and 425 s.





Figure 13. Example of live down-linked X-Ray image for monitoring durng the flight. a) & b) Solidification stage 1; c) start of Solidification stage 2; d) Solidification stage 2; e) eutectic transformation

Digitally enhanced images have been published [6] with open online access to the video sequence from the sounding rocket experiment. The XRMON-SOL project on equiaxed solidification was a scientific and technical success, and further quantitative examination of the results is being carried out by the UCD team.

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