RAMMEC
Recycling enhanced additive manufacturing processes under Martian environmental conditions

Executive summary
Early technology development

OSIP Idea ID: I-2022-01589

Affiliation(s): FOTEC Forschungs- und Technologietransfer GmbH (Prime), University of Innsbruck (Sub 1)

Activity summary:
In-situ resource utilization (ISRU) is a reasonable approach for future exploration missions to build structures on Moon or Mars with on-site resources such as regolith or Sulfur. Such structures can be habitats for astronauts, landing pads or spare parts needed during such missions. The idea is to save payload by using the regolith for radiation shielding, protection against micrometeorite impacts or similar applications. The aim of this project was to develop an additive manufacturing setup capable of demonstrating the processing of Martian regolith simulant under ambient and simulated Martian environmental conditions (atmosphere composition and pressure at room temperature) and to produce a first set of material data, to prepare in-situ manufacturing capability for future exploration missions.

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CONTENTS

1. SCOPE OF THE DOCUMENT

This document is the Executive Summary Report of the RAMMEC project, performed under ESA contract no. 4000139887/22/NL/GLC/ov (OSIP Discovery Ideas Open Channel ETD Activities Evaluation Session (Idea I-2022-02896) by FOTEC Forschungs- und Technologietransfer GmbH, Austria.

A compendium of the technical reports is available in the form of a Final Report (Doc. no. FTC2024-094-01-01). The Final Presentation will be held on December 9th, 2024, via an online meeting.

1.1. Applicable Documents

Unless specified otherwise, the following documents in their latest revision shall be considered as an integral part of this document.

Ref.	Document Number	Title	Issue/ Rev.	Date
[AD-01]	I-2022-028696	RAMMEC – Recycling enhanced additive manufacturing processes under Martian environmental conditions – Full proposal	01-00	25-09-2022
[AD-2]	FTC2023-048-01-00	Concept design and material research	01-02	11-08-2023
[AD-3]	FTC2023-097-01-00	Test report of non-AM samples (TR1 incl. TP1)	01-00	10-10-2023
[AD-4]	FTC2024-001-01-01	Test plan for AM samples (TP2)	01-01	21-03-2024
[AD-5]	FTC2024-052-01-00	Test report for AM samples (TR2)	01-00	08-07-2024

Table 1-1. Applicable Documents

1.2. Reference Documents

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1.3. Acronyms and Abbreviations

In the scope of this document, the following abbreviated terms are defined and used:

3D	3-dimensional
ABS	Acrylonitrile-butadiene-styrene copolymer
AD	Applicable Document
ALM	Additive Layer Manufacturing
CAD	Computer Aided Design
CNC	Computerized Numerical Control
COTS	Components Off the Shelf
DAQ	Data Acquisition
DC	Direct Current
DDL	Deliverable Documents List
DLP	Digital Light Processing
DSC	Differential Scanning Calorimetry
EDX	Energy Dispersive X-Ray
FDM	Fused Deposition Modeling
MC	Material Composition
MGS	Mars Global Simulant
MS	MileStone
PA	Polyamide
PC	Polycarbonate
PE	Polyethylene
PLA	Polylactide
RD	Reference Document
SCCC	Sulfur Concrete Contour Crafting
SEM	Scanning Electron Microscope
SLA	Stereolithography
TBC	To Be Confirmed
TBD	To Be Defined
TG	Glass Transition Temperature
UIBK	University of Innsbruck
WP	Work Package

2. BACKGROUND AND OBJECTIVES

2.1. Background

In-situ resource utilization (ISRU) is a reasonable approach for future exploration missions to build structures on Moon or Mars with on-site resources such as regolith or Sulfur. Such structures can be habitats for astronauts, landing pads or spare parts needed during such missions. The idea is to save payload by using the regolith for radiation shielding, protection against micrometeorite impacts or similar applications.

FOTEC has shown in a precursor activity ("Limited resources manufacturing technologies", ESA Contract No. 4000114191/NL/SFe) successfully that Phosphoric acid as a binding agent for Martian regolith simulant (JSC-Mars-1A) is working. However, only ambient environmental conditions (RT and ambient pressure) during extrusion-based additive manufacturing were considered. Furthermore, the Phosphoric acid still needs to be transported as part of the payload to the destination (e.g. Mars).

Based on that within this project further aspects were taken into account, for example the use of Sulfur as binding agent (instead of Phosphoric acid), 3D-printing under simulated Martian environmental conditions and the impact of recycled polymer food packaging foil as reinforcement on the mechanical and thermal properties of Martian regolith samples.

2.2. Project Objectives

Herewith a novel approach to process Martian regolith simulant in relevant environmental conditions and investigate recycling aspects of packaging material commonly used during space missions.

The project objectives were to establish a simulated Martian environment with reduced atmospheric pressure and high CO_2 concentration, design and manufacture an additive layer manufacturing device capable of processing dry regolith feedstocks and perform material testing including the comparison between ambient and Martian environmental conditions.

The material data base and lessons learned shall contribute to follow-on activities and future exploration missions including permanent stations on Moon and Mars.

3. MAIN ACHIEVEMENTS AND FINDINGS

3.1. Sample Manufacturing Results

The first key aspect of this project was the determination of suitable process parameters to additively manufacture Sulfur/regolith samples under both ambient standard laboratory and Martian environmental conditions while studying the impact of those parameters on the samples. It was possible to produce bending samples which exhibited a homogeneous material and pore distribution by adapting the printing temperature and volume rate during extrusion. Volume rates of approx. 23 mm^3/s and a print temperature of 135°C were the optimized parameters for printing in ambient laboratory conditions. When printing under CO_2 atmosphere and 6 mbar pressure, it was necessary to increase the temperature to 145°C to prevent clogging in the extruder, see Figure 3-1. The need of an increased temperature under Martian atmospheric condition is an important aspect to keep in mind when investigating AM processes to be conducted on Mars.

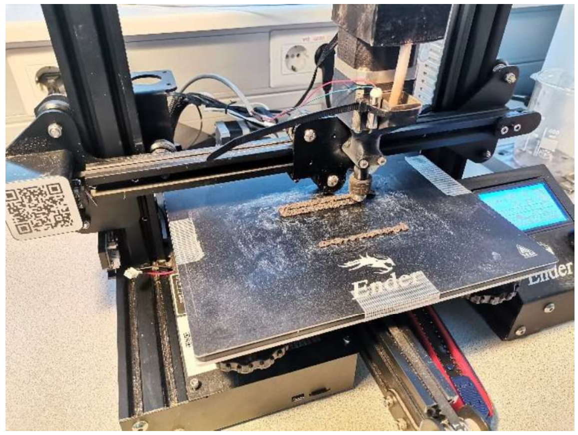


Figure 3-1. Print trial of bending test samples (12x12x80 mm³) with MGS-1/Sulfur mixture

To produce non-AM samples with defined dimensions a stainless steel mould was used, see Figure 3-2. Different feedstocks (mixtures of regolith, Sulfur and polymer foil flakes) were premixed and filled into the mould and cured at increased temperature.

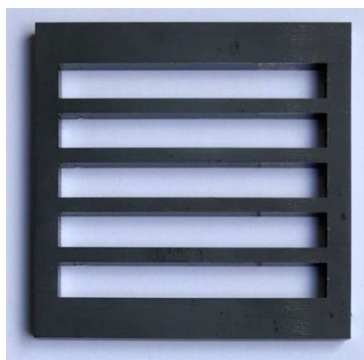


Figure 3-2. Mold made from stainless steel used for casting to manufacture the samples

Examples for non-AM samples manufactured with the mould are shown in Figure 3-3.

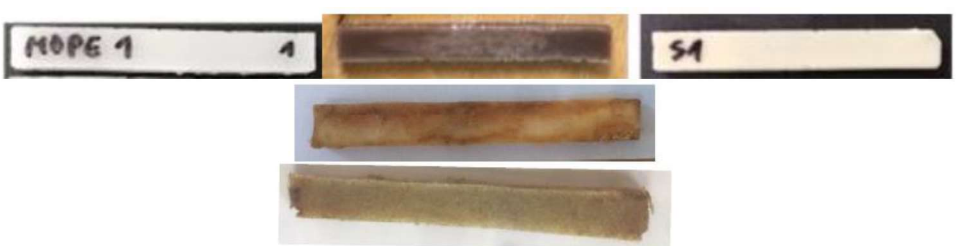


Figure 3-3. Examples for three-point bending samples with different chemical composition (pure MDPE, with Sulfur, with MGS-1 regolith)

The approach of using such a mould for sample manufacturing was working properly. However, sample extraction after curing was challenging as, depending on the type of mixture, the adhesion between the mould and the sample was strong.

To verify the quality of sample manufacturing and to assess the impact of simulated Martian environmental conditions, micro-CT (computer tomography) measurements were done. The results can be seen in Table 3-1. It can be concluded that low-pressure environment leads to foaming, although as mitigation action the MGS-1 regolith has been dried beforehand. For future activities, further feedstock preconditioning steps might be relevant, especially for the used Sulfur (no drying step applied within this activity).

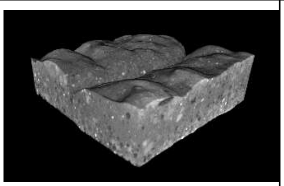
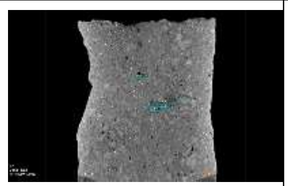
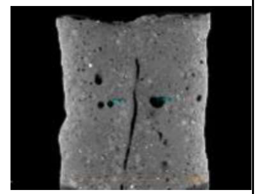
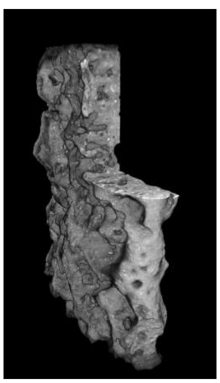
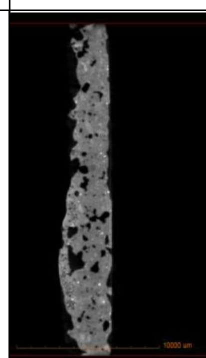
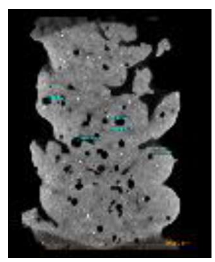
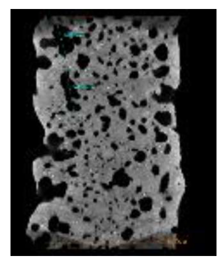
Batch ID	**Overview (3D reconstruction)**	**Side view (YZ-plane print bed on the right side)**	**Top view of layer close to print bed (XZ-plane)**	**Top view close to uppermost layer (XZ-plane)**
#1				
M4				

Table 3-1. Reconstructed 3D volume and 2D slices of micro-CT measurements from halved samples were used for three-point bending tests

3.2. Material Extrusion Prototype

Based on a technology trade-off material extrusion was chosen for sample manufacturing in ambient and simulated Martian environmental conditions. The main challenge of the development was the material feeding of the regolith/Sulfur mixture to the heated nozzle.

Several design iterations were done to achieve continuous feeding without any clogging issues. All structural components of the print head (mounted on the gantry robot system of the COTS printer) were manufactured out of Polycarbonate (PC) with Fused Deposition Modeling (FDM) technology (see Figure 3-4). This approach allows full design flexibility combined with low costs and short lead times.

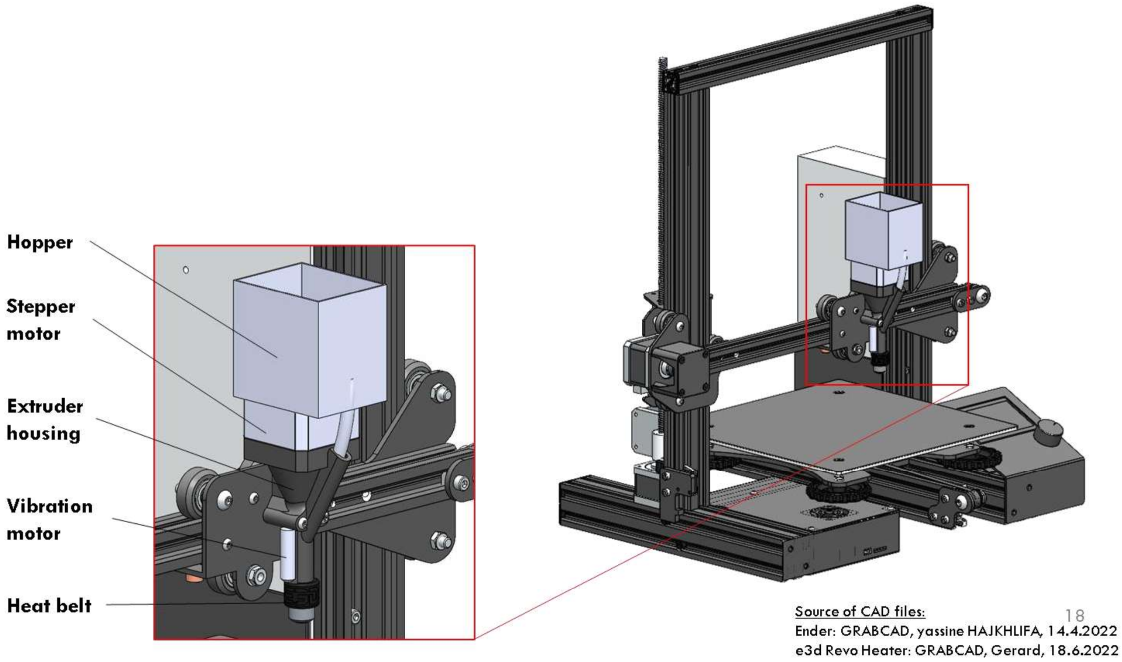


Figure 3-4. Design of the material extrusion prototype

The assembled prototype during material extrusion is shown in Figure 3-5.

Figure 3-5. Assembled material extrusion prototype to integrated in the environmental chamber

Lessons learned:

Material extrusion setup must be combined with a shaping blade to overcome the significant irregularities of the sample surface due to inhomogeneous extrusion. The test campaign showed that irregularities of the sample surface cause increased standard deviations combined with a generally low level of measured values (due to mechanical stress concentration).

3.3. Main Results of Material Testing

The flexural and compressive strength of the AM samples tested yield a lower flexural strength than the thermally uncycled samples made from Mars regolith simulant and phosphoric acid "JSC Mars-1A" [RD-57]. This might be due to less pore formation by using phosphoric acid as a binder. The thermally cycled samples, however, have less compressive strength than the samples produced for the test campaign presented in this project.

Casted samples produced at UIBK serve as another comparison for the performance of the AM samples and the material in general and further allow for comparison to other studies investigating Sulfur concrete. The cast samples (160x40x40 mm³) were tested according to DIN EN 196-1. The flexural and compressive strength of the cast samples is 7.98 ±0.20 MPa and 47.38 ±4.99 MPa respectively and therefore higher than those of the smaller AM samples. The flexural strength agrees well with the result of 7.24 ±0.73 MPa as does the compressive strength of 48 MPa, both obtained from samples containing 50 wt.-% Sulfur and 50 wt.-% Mars regolith simulant JSC Mars-1A [RD-58]. The authors in [RD-58] further argue that recasting and compressing while casting can increase the compressive strength up to 63 MPa, since the applied force during casting reduces the formation of pores. Which also confirms the trend observed that the increased pore formation for the samples printed under Martian atmospheric conditions is the main factor influencing the mechanical properties.

In conclusion, the AM samples produced under Martian atmospheric conditions exhibit reduced mechanical properties, caused by the significantly increased pore formation. It remains open at which point of the AM process exactly the pore formation takes place (e.g. extrusion or hardening). While the larger pores at first seem to be a disadvantage due to the, for instance, reduced flexural strength, it could serve as a starting point for investigating the potential use of Sulfur/ regolith mixtures as an insulating material.

As an example, in Figure 3-6 and Figure 3-7 the comparison between ambient and simulated Martian environmental conditions in terms of flexural modulus and strength is shown.

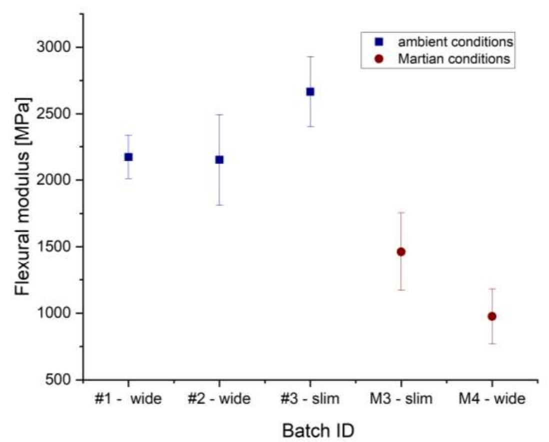


Figure 3-6. Flexural modulus obtained from three-point bending tests; AM sample batches printed in ambient conditions marked in blue squares and batches printed in Martian conditions marked in red dots

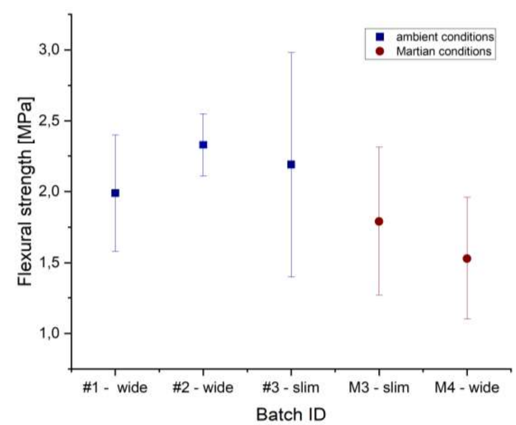


Figure 3-7. Flexural strength obtained from three-point bending tests; AM sample batches printed in ambient conditions marked in blue squares and batches printed in Martian conditions marked in red dots

The results on the thermo-mechanical properties of AM samples made from recycled Combitherm xx15 foil and various percentages of MGS-1 Mars regolith simulant revealed a change in the glass transition, melting and crystallization temperatures of PA6,12 as well as in the melting temperature of PE for samples containing MGS-1. Further measurements are required to determine the statistical significance of the observed changes. However, this test campaign serves as a starting point for further investigations on the potential to manipulate the (thermo-)mechanical properties of AM samples with recycled polymers by adding Mars regolith simulant to also extend the possible applications on Mars.

Lessons learned:

Within this project only single layer samples were tested, in future activities it is recommended to use multi-layer samples for mechanical properties characterization such as three-point bending or flexural strength tests. Furthermore, considering the sample density and normalizing the results (e.g. compression strength) accordingly to cover the pore formation due to reduced atmospheric pressure (6 mbar).

3.4. Main Results of Packaging Foil Recycling

Differential Scanning Calorimetry (DSC) was performed on a Netzsch DSC 214 Polyma, see Figure 3-8. Measurements of the foil were conducted at a heating-/ cooling rate of 10 K/min and the temperature program as follows: i) heating from 25°C to 230°C, ii) isothermal for 3 min, iii) cooling to 25°C, iv) heating to 230°C and v) cooling to 25°C. Nitrogen protective gas flow was set to 60 ml/min. The samples were inserted into pierced aluminum crucibles together with "Thermal Grizzly" heat transfer agent to allow for a more precise measurement due to improved contact of the sample with the crucible [RD-50].

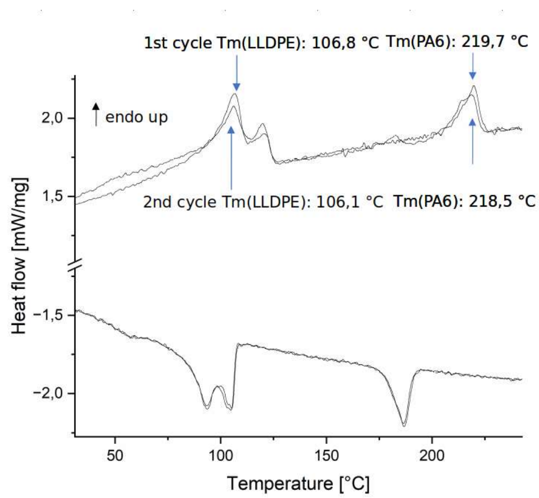
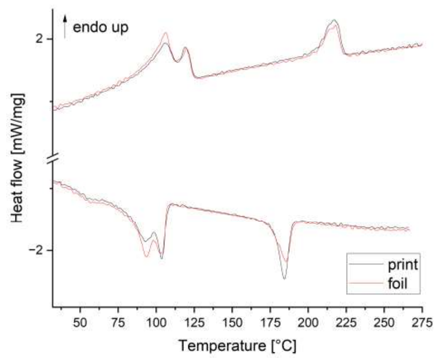


Figure 3-8. Differential Scanning Calorimetry results for the Combitherm xx115 foil. Two heating cycles were performed to eliminate the thermal history of the material. In the second heating cycle, the melting temperatures for LLDPE and PA6 are reduced

The DSC results in Figure 3-8 display the thermal behavior of the Combitherm xx115 foil between 25°C and 230°C. In particular, the melting peaks of Linear Low-Density Polyethylene (LLDPE) at 106.1°C [RD-51] and Polyamide 6 (PA6) at 218.5°C [RD-52] at the second heating cycle are prevalent. This result yields the minimal temperature required to fully meld the composite material. At the same time, it serves as a starting point for future studies investigating the impact of several recycling cycles on the thermal signature of the composite material. Figure 3-9 shows a first comparison of DSC measurements of the Combitherm foil and a printed sample respectively, which was also measured at 10 K/min from 20 to 275°C and shall serve as a comparison to the DSC measurements emulating the recycling process (see Figure 3-9 right).



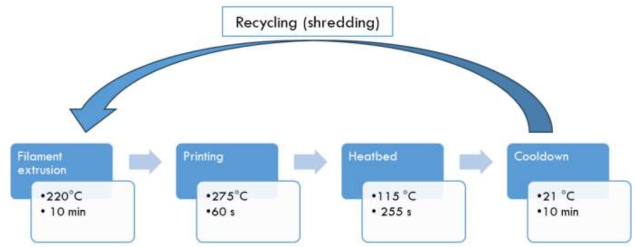


Figure 3-9. Left: Differential Scanning Calorimetry (DSC) results for the Combitherm xx115 foil and print from 20 to 275°C, second heating cycle for each measurement is displayed. Right: Combitherm xx115 foil recycling process steps which shall be emulated via DSC measurements

3.5. Results Summary

- A material data base of Martian regolith simulant samples with Sulfur and recycled polymer food packaging foil processed in ambient and simulated Martian environmental conditions has been established
- An extrusion-based additive manufacturing device has been developed to allow the manufacturing under simulated Martian environmental conditions
- Durability testing of operating an additive manufacturing device at reduced pressure (6 mbar) and CO_2 atmosphere has proven that no overheating issues occur, and the device has no impact on the chamber pressure level
- The setup in its current stage considers only pressure and atmosphere, further functionalities such as temperature cycling, radiation and dust are reasonable add-ons for a future Martian environmental test chamber
- The results show that regolith/Sulfur is a promising candidate for any future ISRU activity
- Implementation of recycling polymer food packaging foil still needs further R&D efforts, within this project only basic investigations were done

During the test campaign, valuable insights regarding potential AM applications on Mars could be obtained. Of particular interest is the formation of large pores ("foaming") which was encountered in samples printed in CO_2 atmosphere and at 6 mbar pressure. This behavior must be considered when aiming to implement AM of Sulfur/ regolith composites to manufacture structures on Mars (e.g. habitats). Further research is necessary to investigate whether it is possible to produce layers which are gas-impermeable and/or can also act as insulation to contain the heat which would be required inside a habitat for astronauts.

The feasibility of using recycled Combitherm xx15 foil for AM was also confirmed. Furthermore, the possibility of adding MGS-1 Mars regolith simulant was demonstrated together with its impact on thermo-mechanical properties of AM samples. The impact on mechanical properties of Mars regolith simulant on AM samples made from recycled polymers is subject to further studies. Furthermore, aspects like the applicability of repeated usage cycles (recycling) and correlated material degradation should be considered in further research activities.