Spinning Continuous Fibres from Lunar Regolith Simulants LHS-1 and LMS-1

Executive Summary

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Picture:

Fig. 1: LMS-1 – Bundles of LMS-1-M-1L fabricated with the single tip bushing. Size approx. 25 cm (left) and 10 cm (right).

Motivation:

The objective of this study is to investigate the processing capabilities of basaltic simulants into endless fibres from two distinct lunar regolith simulants using a hot melt extrusion method. Specifically, the test will be carried out in order to describe the spinning capabilities of both lunar simulants to form continuous fibre for reinforcing applications. The chemical composition of the simulants resemble lunar highland and mare rocks chemistry, and hence, allows an attempt to predict the fibre forming ability of comparable lunar regoliths.

Methodology:

The materials used in the study were supplied by ESA. The materials are known as LHS-1 and LMS-1. Both LHS-1 and LMS-1 are commercial lunar regolith simulants. The simulants LMS-1 and LHS-1 have a similar chemical composition to the average lunar regoliths of the Mare and Highland regions, respectively. All glasses needed for the spinning process were made from ESA simulants. Additional thermal treatments, such as annealing, tempering and water quenching were omitted to preserve their original physical properties. Since the viscosity is sensitive to water content, the melt was simply quenched in ambient air on a metal plate to avoid any risk of hydration and a resulting impact on the glass fibre spinning process. It is important to notice, that each glass specimen appears well melted, showing only minor unspecific small surface defects but no critical volume defects. Additionally, no effect on the subsequent fibre drawing process was observed.

1. Fibre extrusion:
   Two laboratory scale bushings were used to perform the fibre forming experiments with LMS-1 and LHS-1. The bushings are part of the laboratory induction fibre forming unit and consist of a manufactured cylinder made of PT/RH10 welded to a tip plate comprising one and seven tips. The volume of the bushings is about 100 ml. For the production of endless fibres, the vitrified material of the simulants was crushed and then filled into the heated bushing. The water cooling system is running before

the power generator is switched on. In the experiment, the crucible is suspended in the copper coil (inductor) and spatially and electrically separated by an insulation layer of a non-woven ceramic textile. The magnetic field generated in the copper coil induces eddy currents in the PT/RH10 bushing, which cause the crucible to heat up. The temperature control is achieved by adjusting the setting of the power input at the generator unit.

2. Fibre testing:
   For the mechanical analysis of the glass monofilaments, the testing machine Favimat made by Textechno21, is used. The filament to be tested is fixed by two clamps. A defined load is applied to the clamped sample before the measurement starts. The Favimat allows the fineness of a filament sample to be measured by a vibroscopic method. The fineness in dtex and the fibre diameter in µm is calculated from the physical relationships between the tensile force, the gauge length and the resonance frequency.
   The data about the physical behaviour of vitrified simulants is gathered through the differential scanning calorimeter method (DSC). A calibrated Netzsch Jupiter STA 449 F3 instrument was used for thermal analysis of LMS-1 and LHS-1. The samples were heated from room temperature to above 1400 °C at a rate of 10 K/min. The tests were performed in a synthetic air atmosphere with a constant gas flow using alumina crucibles. Additionally, a blank curve was run in parallel to account for buoyancy and the effects of the instrument for each scan.

Results:

The tensile strength of filaments represents the most relevant property, as filaments are supposed to be used as reinforcing fibres for lightweight materials or reinforced construction materials. A series of monolithic simulant specimens and continuous fibres were produced in several campaigns during this study for testing purposes

1. Results for LMS-1:

- Fibres of LMS-1 were produced from approx. 50 g of vitrified simulant material in each campaign using the single and seven tip bushing. For the single tip bushing, the fibre drawing process was stable and the formation of fibres was readily reproducible. The mean thickness of the fibres is 28 µm.
- The detected filament imperfections that occurred during the drawing process are critical for continuous fibre forming and prevent a stable process. The formation of small opaques like magnetite are assumed to be the nuclei of the dendritic crystal growth (olivine).
- Various issues were encountered drawing fibres with the seven tip bushing, e.g. the proneness for flooding of the bottom, the single tip bushing worked quite stable. In particular, the conical geometry instead of the flat bottom of the seven tip bushing contributes to a lower susceptibility to flooding, whereas drawing from single tip was more stable.
- As the drawing of fibres using the seven tip bushing was much more unstable, and essentially resulted in both coarser and more brittle filaments. The finest fibres were visually picked out and separated from the bulk sample to be tested, because the coarser fibres were too brittle to be tested mechanically with the single fibre tester. The microscopic examination revealed a more pronounced crystal growth of silicates and

opaques at the thickenings which represent regions of weak mechanical properties.The tensile strength of LMS-1 was determined for single filaments produced on the one and the seven tip bushing. The average value of tensile strength for single tip fibre is 0.84 GPa and the maximum is 2.33 GPa, compared to average tensile strength of 0.87 GPa and the maximum strength of 1.89 GPa for seven tip fibre.

2. Results for LHS-1:

- It was not possible with the present technical capabilities to draw continuous fibres from LHS-1 due to the fibre forming range, i.e. the temperature range between the upper fibre forming temperature and the liquidus temperature T_L, being almost inexistent. In addition, an exothermic effect is formed almost at the same temperature as the lower fibre forming temperature with first crystals being created when the melt starts to become spinnable. These factors translate into a highly unstable spinning process.

3. General:

- Adjusted and optimized geometric and material parameters are vital for the fibre forming ability of basaltic materials. In this context both bushings are not optimized regarding the chemical and physical behaviour of LMS-1 or LHS-1. The experienced build-up of a viscous layer of melt on the bottom plate that partially crystallised during the fibre drawing impaired the fibre formation markedly.
- The transformation temperatures of LMS-1 and LHS-1 occurred at 680 °C and 715 °C respectively. Their liquidus temperature, i.e. the temperature for which any crystal phase is in a molten state, appeared approximately at 1320 °C for LMS-1 and at 1410 °C for LHS-1. The melting reaction at 1215 °C and 1190 °C for LMS-1 as well as the melting reaction for LHS-1 at 1380 °C and 1190 °C are represented by endothermic peaks. LMS-1 displays three crystallisation peaks at 840 °C, 870 °C and 1096 °C, whereas LHS-1 has one exothermic peak at 937 °C.
- The oxidation state of iron as Fe2+ and Fe3+ has a decisive influence on the nucleation rate and the crystallisation behaviour, thus also on the mechanical properties.

Highlights:

As a supplement to the last part of this study, various trial settings were carried out to control the winding geometry of spun filaments. Usually, a feasibility study aiming at fiberizing basaltic materials consists of a series of tests about the glass forming ability, the fibre forming experiments including the mechanical evaluation of fabricated filaments. However, the spinning process of filaments from lunar simulants, thought being part of ISRU technology, has not only to demonstrate the capability of collecting filaments on a take-up wheel as raw filament bundles but also to be stably unwindable from the bobbin for further processing, i.e. producing non-wovens. As a result, it was achieved to store and wound filaments on a bobbin, ready for unwinding and potential post processing steps.