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EFFECT OF VARYING THE PROPORTION OF CRYSTALLINE/AMORPHOUS PHASES IN A LUNAR REGOLITH ANALOG AND RESULTING PHYSICAL PROPERTIES FOR ISRU SELECTIVE LASER MELTING

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Introduction: For a decade the lunar regolith has been the subject of intensive research as a resource within the framework of ISRU studies [1].

Due to its formation and evolution mechanisms, the lunar regolith is composed of variable proportions of crystalline (original rocks) and amorphous (impact glasses) materials. To demonstrate the viability of ISRU technologies, the use of terrestrial rocks to produce lunar regolith substitutes called either simulants or analogs has become prevalent [2,3].

The Toulouse ISRU Taskforce [4], composed of laboratories having complementary expertise on the subject, has been part of this effort by producing a basaltic lunar regolith analog, known as the Basalt of Pic d'Ysson (BPY) analog. For studies for both additive manufacturing and electrolysis processes, the BPY (from Massif Central in France) has been extensively characterized regarding its grain size distribution, chemical composition (fig.1), phase identification, and thermal properties during a first thermal cycle.

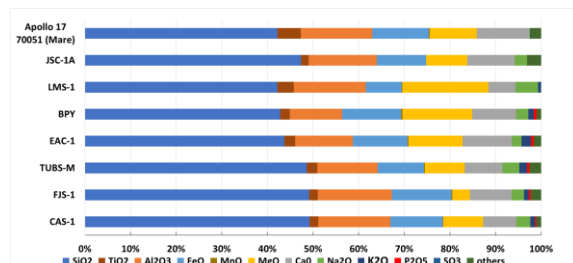


Fig 1: Chemical composition (wt.%) of Pic d'Ysson basalt (BPY) analog compared to well-known mare simulants and regolith samples from Apollo missions

Prepared from an unweathered basaltic lava flow, the BPY analog has the advantage of being naturally completely crystalline. By melting and quenching the initial rock, it is possible to create an amorphous material, used to 'dope' the initial crystalline-analog, thus controlling the crystalline/amorphous ratio, in order to span the range of situations expected to be found for the regolith at the surface of the Moon.

This presentation aims to present the main physical differences between the amorphous and crystalline materials, in particular in terms of thermal behavior, and then to study the impact of the crystalline vs amorphous phase on direct additive manufacturing techniques, such as Selective Laser Melting.

Comparison of main physical parameters:

Crystalline and amorphous powder mixtures (amorphous powder contents, wt.%: 0%, 30%, 50%, 70%, 100%) have been subjected to a first thermal cycle during a Differential Thermal Analysis (DTA) with a heating rate of 15°C/min, from ambient to 1300°C under argon atmosphere (fig.2).

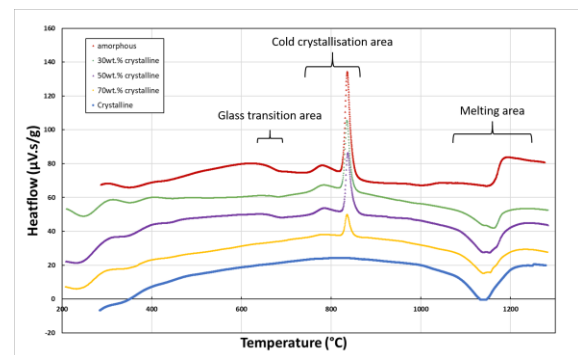


Fig 2: DTA curves of various crystalline and amorphous powder mixtures of Pic d'Ysson basalt (BPY) analog (wt.%), during first heating. To facilitate the readability of the graph, the curves have been translated vertically to avoid overlapping

Increasing the proportion of amorphous material induces the increase of the effect of specific phase transitions, especially for the glass transition at 660°C and the cold crystallization peaks at 790°C and 810°C. A melting domain is observed in all considered cases, centered at 1150°C.

To quantify the specific heat along temperature cycles as well as the enthalpies associated with those phase transitions, Differential Scanning Calorimetry is currently being investigated.

Focusing on the fully amorphous powder, annealing experiments performed at 900°C (just after the cold crystallization area), coupled with XRD analyses, have led to the conclusion that cold crystallization favors diopside formation. With a Vickers microhardness significantly higher than the original glass material microhardness (900HV for the diopside, 700HV for basaltic glass), the implementation of lasing conditions that allow annealing seems to be a suitable way for the optimization of the mechanical properties of manufactured objects.

A second interesting behavior of the amorphous material occurs just after the glass transition. Thermo-mechanical analyses (TMA) have been performed on 3mm high compressed pellets (φ8mm)

with both crystalline and amorphous BPY analog (fig.3), following these thermal cycle parameters: an argon atmosphere, a constant heating and cooling rate of 10°C per minute, a 20-hour isothermal dwell at 720°C. The behavior of the crystallized material is characterized by expansion on heating and contraction on cooling, characteristic of a stable material. In the case of amorphous material, shrinkage begins during heating at a temperature level close to that of its glass transition. This shrinkage continues during the isothermal dwell. A form of viscous flow sintering is so taking place in the amorphous material, which is a major behavior difference between the BPY analog in the crystalline and amorphous states. This notable feature could be exploited in more classical ceramic manufacturing methods and have a non-negligible impact on additive manufacturing processes.

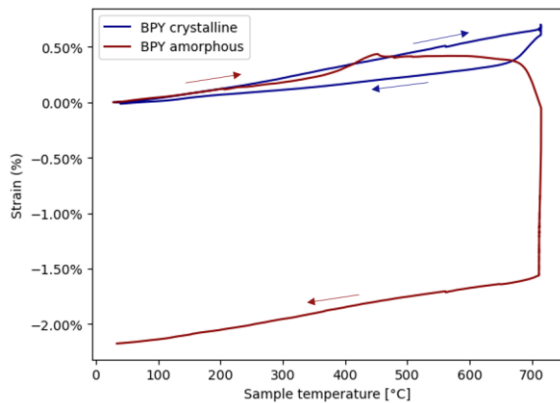


Fig 3: TMA curves of Pic d'Ysson basalt (BPY) analog during a first thermal cycle, with a 20 hr isothermal dwell at 720°C.

Selective Laser Melting tests :



Fig 4: BPY specimens on an amorphous silica substrate (50x50mm²) manufactured by selective laser melting

As of today, only the BPY raw material (ie with a crystalline state) has been tested for selective laser melting, with encouraging results (fig 4). Under a 50W laser power, a scanning speed of 225 mm/s, a spot size of 150µm and a hatching distance of 50µm, compression cylinders (φ3mmxh6mm) and bending parallelepipeds (26x5x2.5mm³) have been success-

fully produced as mechanical test specimens. For example, compressive strengths of 50MPa or more have been measured, showing at this stage the ability to make small objects with an interesting level of mechanical strength from the crystallized BPY analog. Further tests are underway to investigate the influence of varying the SLM process parameters and considering mixtures of crystalline and amorphous BPY.

Perspectives :

Three directions of research will be carried out in the near future:

- to carry out an experimental plan considering different values of the SLM process parameters to identify a process window allowing the best characteristics of the lasered material,
- to vary the crystalline/amorphous ratio of BPY analog in SLM tests, which should decrease the energy required to melt the material,
- to improve the SLM substrate performance to limit mismatch in thermal expansion coefficient and therefore the initiation of cracks in the final product.

In parallel with those additive manufacturing trials, the thermal conductivity and optical response (emissivity) of BPY analog powder over 1000°C will be determined to better understand the laser-powder interactions and to give the possibility of numerical modeling of the powder bed fusion process.

By the specific nature of the considered raw material, namely lunar regoliths or analogs, the objects obtained after SLM are necessarily ceramics. Therefore, they have a brittle mechanical behavior and a high sensitivity to the presence of defects like porosities, surface roughness, and cracks. Investigating the effect of adding controlled metallic powders to form ceramic-metal composites is a promising perspective that should allow keeping the hardness of ceramic-based materials with a more ductile behavior, by improving specific mechanical properties like fracture toughness.

References: [1] Reitz, B. et al. (2021) *Microgravity Sci. Technol.* **33**, 25. [2] Taylor L. et al. (2016) *Planet. Space Sci.* **126**, 1-7. [3] Engelschiøn, V. S. et al. (2020) *Sci Rep* **10**, 5473. [4] Granier J. et al. (2022), *ELS 2022 - In-Situ Resource Utilization*.