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Sintering of micro-trusses created by extrusion-3D-printing of lunar regolith inks

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ABSTRACT

The development of *in situ* fabrication methods for the infrastructure required to support human life on the Moon is necessary due to the prohibitive cost of transporting large quantities of materials from the Earth. Cellular structures, consisting of a regular network (truss) of micro-struts with ~500 μm diameters, suitable for bricks, blocks, panels, and other load-bearing structural elements for habitats and other infrastructure are created by direct-extrusion 3D-printing of liquid inks containing JSC-1A lunar regolith simulant powders, followed by sintering. The effects of sintering time, temperature, and atmosphere (air or hydrogen) on the microstructures, mechanical properties, and magnetic properties of the sintered lunar regolith micro-trusses are investigated. The air-sintered micro-trusses have higher relative densities, linear shrinkages, and peak compressive strengths, due to the improved sintering of the struts within the micro-trusses achieved by a liquid or glassy phase. Whereas the hydrogen-sintered micro-trusses show no liquid-phase sintering or glassy phase, they contain metallic iron 0.1–2 μm particles from the reduction of ilmenite, which allows them to be lifted with magnets.

1. Introduction

Due to the very high costs of transporting equipment and raw materials from Earth, the establishment of human bases on the Moon for scientific lunar investigations and further explorations of the solar system will require *in situ* fabrication using local, abundant materials, to create the infrastructure needed to support human life and laboratory equipment. Building materials must provide protection from the extreme thermal cycles and the vacuum environment of the Moon as well as solar and cosmic radiation [1–3]. Lunar regolith (lunar dust) is a promising material that could be utilized for the construction of the lunar infrastructure due to its abundance, ease of collection, ability to withstand the extreme lunar thermal cycles, and radiation shielding abilities [4,5]. The use of binder materials such as sulfur and the direct melting or sintering of the lunar regolith are two approaches that have been reported for the fabrication of lunar regolith objects. Numerous studies have focused on the use of lunar sulfur concrete, which uses sulfur as the binding agent for the lunar regolith rather than a water and cement mixture, as is used in hydraulic concrete on Earth. However, the use of this technology is limited by the narrow working temperature range for sulfur concrete (~130–140 °C), the sublimation of sulfur in lunar vacuum, the limited geometries and complexity of objects produced by the casting process, and the additional steps associated with sulfur supply (discovery, mining, transport, beneficiation, and reduction to elemental sulfur of sulfur-bearing ore) [6–8]. Recently, Khoshnevis et al. reported Contour Crafting, an additive manufacturing (AM) process based on extrusion, of lunar sulfur concrete to produce large scale domes and walls [9,10]. Although this method is capable of rapidly producing large scale structures on the Moon, sulfur concrete performs poorly when subjected to simulated lunar temperature cycles due to the large differences in the coefficients of thermal expansion between the lunar regolith and the sulfur [7]. Additions of fly ash or recycled aggregates have been reported to improve the thermal cycling capabilities of terrestrial sulfur concrete [11].

Additive manufacturing or 3D-printing (3DP) of objects from pure regolith powders, which are densified by sintering or fusing rather than by the addition of sulfur or water as in concrete or cement, is another viable manufacturing technique for the *in situ* fabrication of lunar regolith parts on the Moon [1,12–19]. Balla et al. first demonstrated AM of lunar regolith cylinders using Laser Engineering Net Shaping [12]. Cesaretti et al. reported a feasibility study of a lunar habitat using the

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D-shape process, which selectively sprays a liquid binder onto a layer of lunar regolith to bind the printed structures together *via* a chemical reaction of magnesium oxide with magnesium chloride, to produce large scale parts [13]. Blocks, gears, nuts, and mesh grids with high surface hardness values [14,15] and multi-layered structures and lattices [16–18] have been produced *via* selective laser melting of lunar regolith. Zhang and Khoshnevis proposed Selective Separation Sintering of JSC-1A simulant to produce interlocking parts using alumina as the separation coating [19]. The use of AM techniques allows the fabrication of a wide variety of both simple and complex part geometries without the use of molds. Additionally, AM techniques can be used to produce a variety of different ordered, truss-based microarchitectures within a part. Such micro-trusses are lightweight with high specific strengths and stiffnesses and high surface areas [20–26].

In this work, we characterize the microstructures, mechanical properties, and physical properties of lunar regolith micro-trusses produced *via* a combination of extrusion-based 3DP of liquid inks containing JSC-1A lunar regolith simulant powders, followed by sintering in either air or hydrogen atmospheres (Fig. 1). The as-printed micro-trusses are thermally processed to remove the polymer binder and to sinter the lunar regolith particles to form oxide struts. Two atmospheres were considered for sintering: air and hydrogen. Several studies, including current NASA projects, have proposed the reduction of the ilmenite ($FeTiO_3$) mineral in lunar regolith using hydrogen to produce the water, or subsequently oxygen, required for human survival on the Moon [27–33]. For the micro-trusses sintered in hydrogen, the iron-oxide-containing minerals present in the lunar regolith are reduced to metallic iron, creating micro-trusses whose struts show a ceramic-metal composite structure, which could have an increased toughness or strength compared to the purely ceramic counterpart [34]. Thus, sintering in a hydrogen atmosphere may be used not only to create stronger, tougher building materials, but also for oxygen extraction. Although robotic fabrication in high vacuum outside a lunar habitat may occur, we consider here the scenario where 3DP and post-printing reduction and sintering of parts is done by humans within the habitat. Thus, an air atmosphere was chosen for comparison. Similar 3D printing platforms involving the extrusion of liquid particle-containing inks can also be used to produce metals and alloys [35–40], ceramics and ceramic-based composites [41–44], electronic materials [45,46], materials for bone regeneration and tissue engineering [45,47,48], and graphene for tissue engineering and electronics [45,49], making extrusion 3D-printing a suitable option for the production of additional materials on the Moon with the same printer. Flexible ceramic-loaded elastomer composites have also been fabricated using the same extrusion-based 3DP of liquid inks containing lunar or Martian regolith particles that is used in the present work [50]. A complete description of this 3D-printing process and a discussion of its suitability for a lunar environment are provided by Jakus et al. [50]. Moreover, the lactic acid and the glycolic acid monomers used to prepare the poly(lactic-co-glycolic acid) copolymer binder in the inks may be derived from urine [51,52] and the solvents used in the ink synthesis can be recovered after thermal processing, and distilled, making the present processing method particularly appropriate for lunar outposts with limited *ex situ* resources.

2. Methods

Fig. 1 shows a schematic of the 3D fabrication process. Lunar regolith inks were synthesized through physical mixing of (i) poly(lactic-co-glycolic acid) copolymer (PLGA, 85:15 PLA-PLG (poly-lactic acid – poly-glycolic acid) by mass, from Boehringer Ingelheim, Germany), (ii) JSC-1A bulk lunar mare regolith simulant (sieved to <50 μm, from Orbitec, Madison, WI, USA) and (iii) a 15:2:1 by mass mixture of dichloromethane (DCM), ethylene glycol butyl ether (EGBE), and dibutyl phthalate (DBP) (all from Sigma-Aldrich, St. Louis, MO, USA) using the methods described previously [35,50,53]. The lunar regolith simulant is composed of olivine, plagioclase, Ca-pyroxene, glass, and ilmenite ($FeTiO_3$) mineral phases [54,55]. The oxide compositions of each mineral as well as the overall composition from the technical specification sheet are shown in Table 1 [56,57]. The bulk density of the lunar simulant determined *via* helium pycnometry is 2.95 g/cm^3. The solid component of the ink contains 74% JSC-1A simulant and 26% PLGA by volume. For every cm^3 (corresponding to 2.95 g) of JSC-1A powder, 16.2 g of solvent mixture were used. The PLGA was dissolved in approximately half of the solvent mixture in the ink cartridge (Nordson EFD 30 cc fluid dispensing system). The JSC-1A powder was added to the remaining solvent mixture in a separate 50 mL Falcon tube. After the PLGA dissolved, the powder suspension was poured into the ink cartridge and mixed using a mini vortex mixer. The ink was thickened under ambient conditions *via* DCM evaporation, with occasional hand stirring, until its viscosity reached 30–35 Pa s.

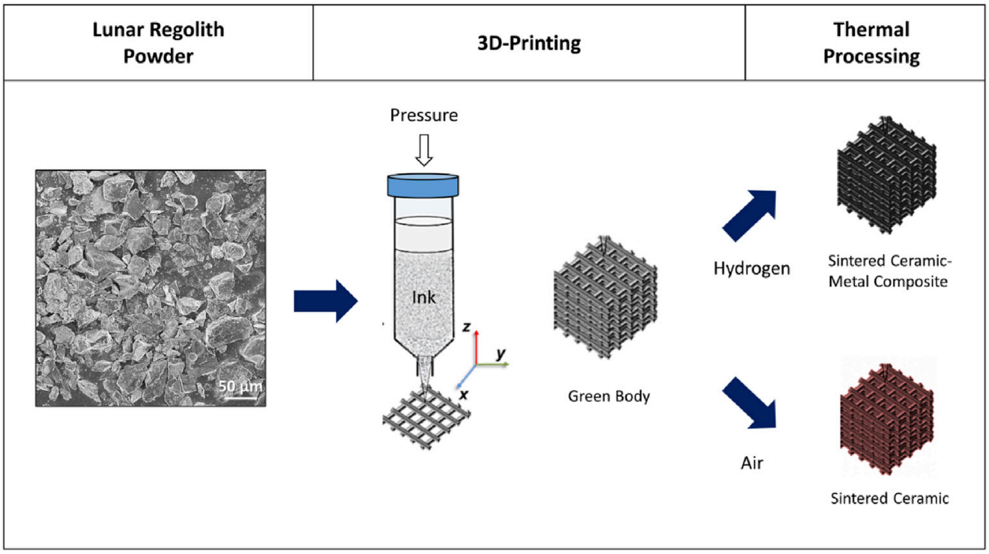

Fig. 1. Schematic of the 3D fabrication process for lunar regolith cellular structures. Inks are comprised of lunar regolith powder, PLGA, and three solvents (DCM, EGBE, and DBP). The liquid ink is direct-extrusion 3D-printed at room temperature into green bodies consisting of layers of parallel struts. The green bodies are then sintered in air or in hydrogen to produce a ceramic (air) or a ceramic-metal composite (hydrogen) micro-truss. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)