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Ceramics from wet-processing of Martian soil simulant using slip casting or Additive Manufacturing for in-situ resource utilization on Mars

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Abstract

For future colonization of Planet Mars, the most realistic approach for the production of parts on site is in situ resource utilization (ISRU). In this study, we demonstrate the feasibility of this concept by producing objects of varying complexity exclusively using resources that can be found on the surface of Mars. The established production routes using slip casting and Additive Manufacturing via wet-processing of Mars simulant material are simple, robust and easily transferable even to the harsh conditions on Mars. After sintering our slip cast parts have mechanical properties comparable to commercially available porcelain, making them suitable for everyday use.

1. Introduction

A promising concept for the exploration and subsequent colonization of Moon and Mars is in-situ resource utilization (ISRU) - collecting, processing and storing of native materials that are encountered during human or robotic space exploration [1]. Early colonization scenarios suggest the direct use of rock-covering loose granular surface media (including dust, soil and rubble) consisting of various oxide minerals called lunar and Mars regoliths. The chemical composition of lunar and Mars regolith makes the extraction of metals and ceramics conceivable. The availability of ceramic tools is an important prerequisite for melting regolith in blast furnaces and bloomeries for the production of base metals. The ISRU approaches for ceramics include (a) dry consolidation [2-4], (b) melting [5,6], (c) self-propagating high temperature synthesis and geopolymerization [7,8]. For the first of these approaches, which represents the most realistic initial scenario of colonization, loose regolith sheets are pressed into bricks and fused by direct compression or sintering [2-4], such bricks could be used for masonry construction [9]. However, dry consolidation paths are often not suitable for ceramic components with complex shapes. Regarding Mars, it remains difficult to assess the feasibility of the ISRU approaches discussed above, especially as many of the proposed routes are not widely used on Earth.

Figure 1: Ceramics made by slip casting of JSC-Mars-1A slurries. The image shows the same geometry after different heat treatments - from left to right: directly after demolding, dried green body, sintered at 1000°C without dwell time, 1130°C without dwell time, 1130°C with 10 hours dwell time.

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It is surprising that the wet processing of minerals - the oldest and most universal processing method for stoneware ceramics, which was invented ~ 30,000 years ago - has not yet been discussed for ISRU. In traditional ceramics, mineral blends containing sheet silicates/phyllosilicates are mixed with water, then shaped by deformation or slip casting [10] and then dried and fired [11].
Here, we present a new versatile material system in the form of a slurry, that only uses two resources – both abundant on the Martian surface. This stable slurry system can be used for shaping green bodies with traditional slip casting and contemporary Additive Manufacturing routes such as layer-wise slurry deposition (LSD).
In this work, we followed a common approach to simulate extraterrestrial regolith on earth using regolith simulants – the first resource for the slurry [12]. Here, we employ JSC-Mars-1A, a natural glassy volcanic ash consisting of finely crystallized and glassy particles of Ca-rich plagioclase, Mg-rich olivine, Mg-rich pyroxene, Ti-magnetite and nanoparticulate iron oxides and oxyhydroxides known as npOx, which are also present on Mars and responsible for its reddish appearance [13-18]. We used JSC-Mars-1A as it is the best established Mars simulant and provides good comparability.

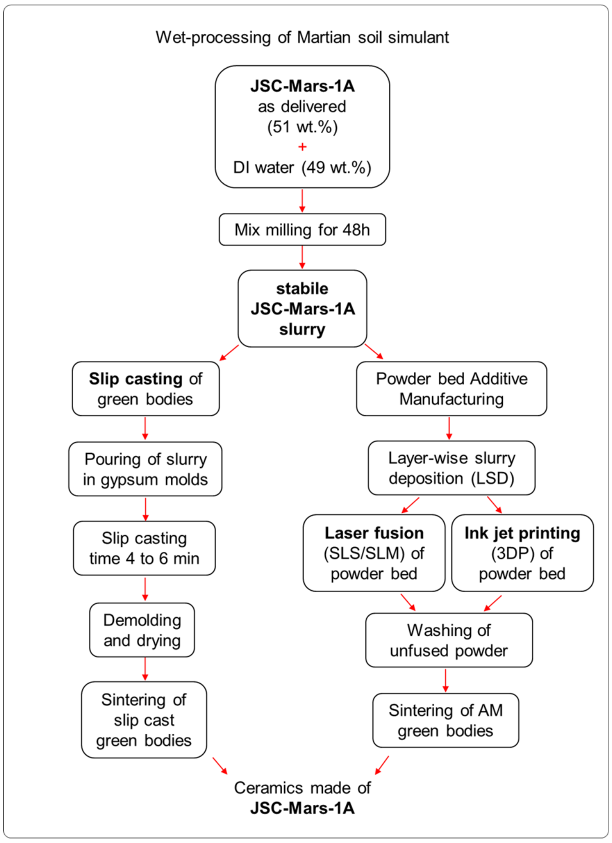

Figure 2: Possible wet-processing routes for JSC-Mars-1A slurry. This paper presents results for slip casting and LSD using laser fusion of the resulting powder bed.

The second resource for wet processing of minerals is water, which can be found in Mars' atmosphere, underground, in the polar caps, the vast majority of which is believed to be in the form of ice [19]. Recently, water ice deposits have been reported to be >100 meters thick [20]. Also, since the discovery of recurrent slope lines in 2013 [21], there has been a scientific discussion as to whether there is contemporary water activity in the form of liquid water brines in the shallow Martian soil.
Shaping via slip casting on Mars has a high ISRU potential, as the material for the gypsum molds can be found in the form of gypsum veins in Martain sedimentary rock at various locations [22,23]. This work not only presents the use of such a slip casting technology for the production of ceramic parts of varying complexity (rings and vases) using exclusively Martian resources, i.e. regolith, gypsum and water, but also investigates the use of this newly developed JSC-Mars-1A slurry system in conjunction with the layer-wise slurry deposition (LSD) Additive Manufacturing technology, which in the future could enable remotely controlled production on Mars.

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2. Materials and methods

The Martian regolith analog, JSC-Mars-1A with a size fraction below 1000 µm was obtained from Orbital Technologies Corporation (ORBITEC, USA). Allen et al. [13] developed this analog using visible and near-infrared spectra obtained by ground-based telescopic observations of Mars (from Mauna Kea Observatory) and Phobos-2 ISM spectra in the Olympus-Amazonis region of Mars [24]. JSC-Mars-1A is a well-characterized soil simulant and its chemical composition resembles Martian regoliths encountered during various Mars missions. Slurries with 51 wt.% JSC-Mars-1A solid load were produced by passing the simulant as delivered through a coarse 500 µm mesh screen. This powder was poured directly into deionized water (no careful/slow mixing was required due to the large grain size) and roll ball milled for 48 hours with 12 mm ZrO_2 grinding balls. Finally, the slurry was poured through a coarse sieve to remove the grinding balls and filled into bottles for later use. A standard porcelain slurry from the Royal Porcelain Factory in Berlin was used as a reference. The molds for slip casting rings with an inner diameter of 30 mm and a height of 18 mm were made by casting plaster with a water plaster content of 4 to 5. The ceramic rings for the mechanical test were cast by placing the molds on steel plates and filling them generously with liquid slurry. In order to obtain comparable wall thicknesses, the casting time for JSC-Mars-1A rings was set at 4 minutes and for porcelain slurries at 8 minutes. After casting, the remaining slurry and steel plate were removed and the ring mold with the wet ring rotated for 90 seconds to produce a homogeneous inner surface. Finally, a knife was used to cut casting overlaps from each side and the rings left to dry. To produce vases, a plaster mold for porcelain vases from the Royal Porcelain Factory in Berlin was generously filled with JSC-Mars-1A slurry and cast for 6 minutes (the longer casting time compared to the rings was chosen to accommodate the larger vase geometry), adding small amounts of slurry to maintain the liquid level. The mold was emptied and turned for 120 seconds and the casting overlap was cut off. After 15 minutes, the casts were demolded and small defects were retouched with a brush and fresh slurry. After the green body had dried, retouched areas and burrs were sanded with 2400 and 4000 grit sandpaper. Three different firing profiles were used for the sintering of slip cast green bodies: (a) heating at 1,7 K/min to 1000 °C, no dwell time, (b) heating at 1,7 K/min to 1130°C, no dwell time and (c) heating at 1,7 K/min to 1130°C with 10 h dwell time. The porcelain greens were sintered according to a different schedule, heated to 1440°C at 2 K/min. All sintering runs were carried out in a muffle furnace in standing air atmosphere, followed by furnace cooling. The weight loss during sintering was determined using a laboratory balance for weighing dried green bodies and sintered parts. A proof of concept study for the Additive Manufacturing of powder beds using the developed JSC-Mars-1A slurry was undertaken at BAM Berlin using layer-wise slurry deposition coupled with selective laser sintering as described previously [25,26].

Methods

A helium gas expansion pycnometer Pycnomatic ATC (Porotec, Germany) was employed to determine the powder particle density for the raw JSC-Mars-1A as delivered. The volume shrinkage and density of green and sintered parts were determined by measuring ring masses with a laboratory balance (RC210P, Sartorius, Germany) and ring volume by X-ray computed tomography CT 40 (Scanco Medical AG, Switzerland). These measurements were performed on one ring sample per sintering temperature before and after sintering for each sintering schedule. The accuracy of the obtained density values was verified by measuring dimensions of cast (and sintered) disks with a caliper and calculating the density using their weight. The linear shrinkage was calculated from the volume shrinkage, assuming isotropic shrinkage. Microstructural analysis was carried out with scanning electron microscopy (SEM) using a Gemini Leo 1530 (Zeiss, Germany) on fresh fracture surfaces of as-slip cast and sintered samples. Mechanical properties and Weibull analysis were assessed on 20 identical slip cast samples for each sintering schedule by brittle ring test in a RetroLine mechanical testing machine (Zwick/Roell, Germany) at a deformation rate of 100 µm/min as described in our previous article [27]. As the calculated tensile strength values are only to be compared with values for materials obtained from similar ring tests, as critically discussed by Hudson the rings made of commercial porcelain were characterized in comparison [28].

3. Results and discussion

To obtain a high-quality slurry from JSC-Mars-1A regolith simulants, neither a grinding step nor the addition of additives like binders were necessary. We found both the particle size distribution and the shear viscosity properties very similar to commercially available porcelain slurries (data not shown), which makes the develop slurry system ideal for ISRU. In the next step, we produced and characterized two sets of samples of different complexity and shape, (a) rings used to evaluate mechanical properties and Weibull statistics by brittle ring testing and scaling our production route by slip casting with a three-part gypsum mold to produce (b) a complex vase geometry (Figure 1). In the next step, the drying, demolding and sintering conditions for the JSC-Mars-1A slurries were investigated.

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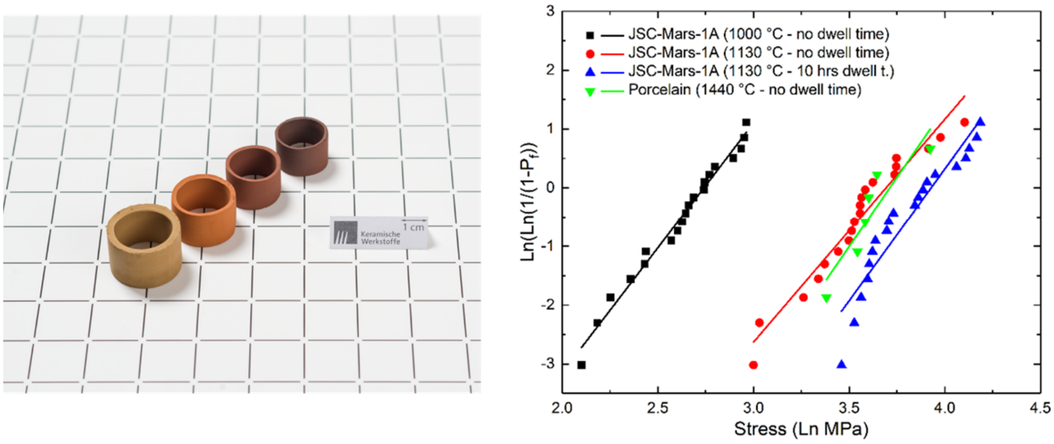

Figure 3: Slip cast JSC-Mars-1A ring test geometries and Weibull probability of failure plot for the three sintered rings and a porcelain reference.

JSC-Mars-1A casts differ from porcelain in their tendency to break, especially for parts with long planar surfaces. To understand this behavior, it is important to note that the regolith simulant does not contain sheet-silicates. These phyllosilicates or clay minerals swell during hydration and lead to the special plastic behavior of hydrated clays. This plasticity is the most important prerequisite for traditional ceramic processing and gives the cast porcelain bodies sufficient elasticity to prevent breakage during demolding. In contrast, JSC-Mars-1A casts show no plasticity when wet and could not be manipulated without fracture. The breakage of the slip casts during demolding could be alleviated by increasing the wall thickness and using molds with easily demoldable surfaces. For the slip cast parts, a study on the sintering behavior was undertaken using hot-stage microscopy [27]. For temperatures above 1250°C the study showed a tendency for the simulant green bodies to bloat - the release of gases during firing at high temperatures [29]. This expansion effect is clearly undesirable and therefore peak temperatures of not more than 1130°C were selected for the sintering schedules of JSC-Mars-1A specimens. The above-mentioned sintering profiles: (a) 1000°C, no residence time, (b) 1130°C, no residence time and (c) 1130°C with 10 h residence time, were selected according to these results. All three sintering schedules produced mechanically stable parts with significant differences in shrinkage, mechanical properties and colors (Figure 1, Figure 3 and Table 1). The best quality parts were obtained at 1130°C with 10h dwell time at volume and linear shrinkage of up to ~62% and ~17% respectively, which is significantly higher than the typical shrinkage of phyllosilicate-based ceramic material systems such as porcelain.

Table 1: Shrinkage (green to sintered), density, porosity and Weibull parameters of slip cast ring samples.

Sample	**Shrinkage (volume/linear), % ***	**Bulk density, g/cm³**	**Mass loss, % ***	**Porosity, %**	**Tensile strength, MPa**	**Weibull parameter m**	**Charac-teristic strength, MPa**
JSC-Mars-1A, green body	-	1.35	-	62.95	-	-	-
JSC-Mars-1A, sintered at 1000 °C	30.46 / 9.27	1.44	22.61	62.78	14 ± 3	4.2	15
JSC-Mars-1A, sintered at 1130 °C	52.36 / 15.07	2.23	22.68	40.35	36 ± 10	3.8	40
JSC-Mars-1A, sintered at 1130 °C with 10 h dwell t.	61.73 / 17.38	2.65	22.76	28.23	46 ± 11	4.5	51
Porcelain sintered at 1400 °C	-	-	-	-	43 ± 6	4.7	41

* from green body to sintered part

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SEM micrographs in Figure 1 show that larger grains are embedded in a voluminous but loose matrix of extremely fine particles. After sintering, the changes in the microstructure (b) are not very pronounced. With this temperature treatment, the total porosity (Table 1) is almost unchanged (62.95% to 62.78%), while an increase in density (1.35 g/cm^3 to 1.44 g/cm^3), as well as a significant volume shrinkage (30.46%) and mass loss (22.61%), occur. With increasing sintering temperature (1130°C, (c)), a portion of the material begins to melt, which in turn leads to fewer pores with increasing size. When the peak temperature of 1130°C is maintained for 10h (d), the resulting structure is a fully glazed matrix with some idiomorphic crystals.

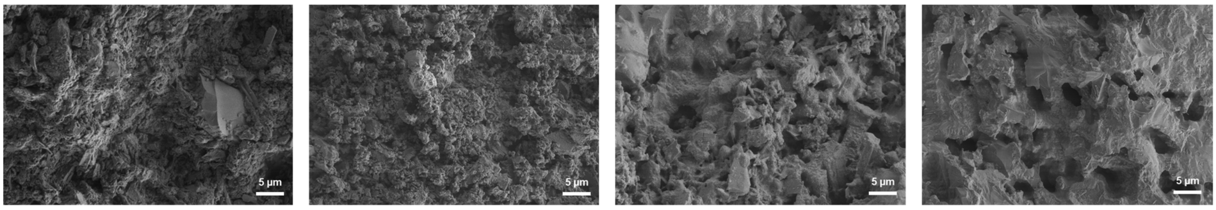

Figure 4: SEM micrography of obtained microstructure. From left to right: (a) green body, (b) 1000°C without dwell time, (c) 1130°C without dwell time, (d) 1130°C with 10 hours dwell time.

As the analysis of the microstructure showed vitrification of the matrix, we conclude that this is the result of a partial melting/liquid phase sintering of the samples, which leads to an increased sinter deformation. The main ferrous phases are hematite and maghemite, which are probably responsible for the reddish brown color of the sintered parts. The color change to red is associated with the oxidation state of iron in oxides typical of sintered earth (oxidation from Fe_3O_4 to Fe_2O_3), as described by Sherriff et al. for ancient Roman pottery [30]. Figure 3 shows the ring geometries and the Weibull diagram for the ring test. The relatively low Weibull parameters and the high standard deviation (Table 1) may be due to inhomogeneities in the geometry of the rings, on the one hand in the inner surfaces of the rings from the removal of excess sludge after the casting process and the sinter deformation. Overall, our slip cast ceramics from Mars soil simulators have exceptionally good mechanical properties compared to the porcelain reference. Of the three sintering schedules, only the JSC-Mars-1A samples sintered at 1000°C showed a characteristic compressive strength below that of porcelain (15 MPa), while the samples sintered at 1130°C without dwell time were similar to porcelain (40 MPa) and the 10 h samples sintered at 1130°C exceeded the reference with a value of 51 MPa. This is particularly noteworthy as these samples showed a high calculated porosity (28.23%) compared to standard porcelain, which has firing temperature dependent porosity values of only <5% [29,31], and the general rule for the flexural strength of ceramics is that the strength decreases exponentially with increasing porosity [29].

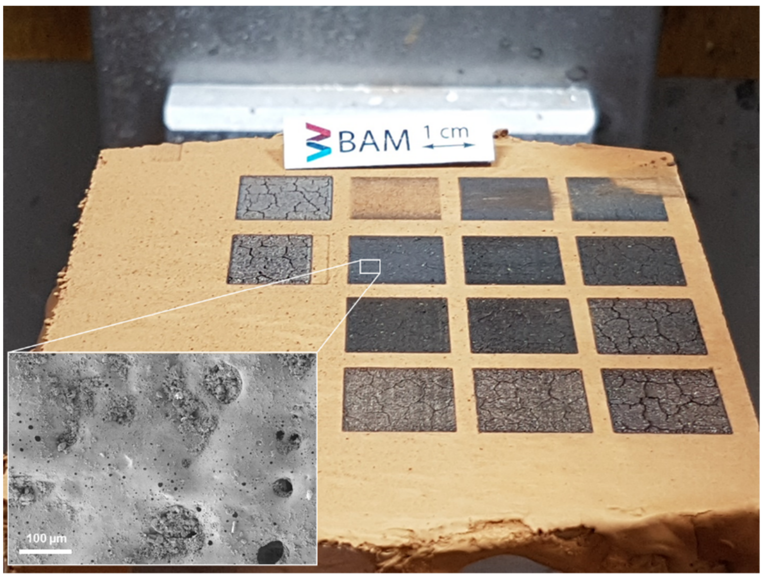

Figure 5: Layer-wise slurry deposition of JSC-Mars-1A. The powder bed was selectively fused using a laser.

Compared to established dry powder bed additive manufacturing technologies, layer-wise slurry deposition makes use of ceramic materials dispersed in water (slurry). The deposition with a doctor blade and its subsequent drying and consolidation allows powder layers with high particle packing and using submicron powders. This yields green body products with significantly higher densities compared to dry powder techniques. The technology is versatile as layer information can be inscribed either via laser or with binder jetting, producing green bodies that can afterward be sintered. As a proof of concept study (Figure 5) we used laser fusion of the powder bed, due to its ease of use in Martian conditions and no binder system that would have to be transported or manufactured on Mars. For our study, layer deposition in the LSD equipment produced cracked powder beds in the early stage of the experiments, which we attribute to the exceptionally fast slip casting of the slurry, described previously [27]. After several attempts and carefully adjusting pumping and deposition speeds, we found parameters that produced crack-free powder-beds. However, the LSD powder bed showed high porosity, similarly to the results for the slip cast green bodies described in Table 1. This was a challenge for the laser sintering of test block geometries using varying laser powders and scanning speeds shown in Figure 5. While most of the initial laser test parameters resulted in cracked green bodies, that had little mechanical integrity, two parameter sets produced test blocks with crack free surfaces, as can be seen in the SEM micrograph in Figure 5. The micrograph also makes obvious the high porosity of the LSD green bodies produced. This is in accordance with earlier observations on the microstructure of green bodies produced by laser fusion [25], which tend to be porous. As we expected the porosity to not decrease much further during sintering, we did not attempt a subsequent heat treatment of the produced parts. Even though dense parts could not be produced, there are possible applications for the presented AM process, for example as membranes or filter materials in ISRU scenarios on Mars.

4. Conclusions

In our work, we have successfully produced mechanically stable ceramic geometries from exclusively Martian resources using a newly developed slurry material system and the slip casting process. Our developed production method is simple, delivers ceramics that meet the requirements of daily use and could, therefore, serve as a starting point for future Mars colonization. We have shown that wet-processing of Martian simulants via slurries to solid ceramics is a promising alternative to the variety of dry consolidation approaches presented in literature. Furthermore, we report here for the first time on the layer-wise slurry deposition of simulant materials using laser fusion of the powder bed. Our results could pave the way for remotely controlled complex shape production with wet-processing of minerals even before humans reach the red planet.

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