ON THE MELTING BEHAVIOUR OF METALS EXTRACTED FROM LHS-1 REGOLITH SIMULANT BY ELECTROLYSIS IN MOLTEN SALT. T. Schild[1,4] , M. Conti[2], G. Aridon[3], D. Harries[1] and K. Hadler[1,4], [1]European Space Resources Innovation Centre, Luxembourg Institute of Science and Technology, 5 Avenue des Hauts-Fourneaux, L-4362, Esch-sur-Alzette, Luxembourg (timon.schild@esric.lu), [2]European Space Agency, ESTEC, Keplerlaan 1, PO Box 299, Noordwijk, 2200 AG, the Netherlands, [3]Airbus Defence and Space, 31 rue des Cosmonautes, 31400, Toulouse, France, [4]University of Luxembourg, 2 avenue de l'Université, L-4365, Esch-sur-Alzette, Luxembourg

Introduction: The extraction of metals from lunar regolith is a key area of interest in the field of space resources utilization. A recent system level analysis identified the FFC molten salt electrolysis process as one of the candidate technologies for the joint extraction of oxygen and metals from lunar regolith [1]. However, experimental studies have shown that the metallic products obtained from regolith simulants by this process are very heterogeneous and require post-processing before use [2], [3], [4]. In this paper, we present a first proof of concept for the thermal post-processing of such metallic products from the FFC molten salt electrolysis of LHS-1 regolith simulant.

Materials and Methods: The presented work has been conducted using solid products of the molten salt electrolysis of LHS-1 regolith simulant. The production process and final characteristics of these metallic products have been reported by the authors in a separate publication [4]. The present work uses a sample of the >600 µm fraction of the product from experiment "LHS-1 B".

The melting behaviour of the material sample has been investigated by combined differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), using a Netzsch STA409PC thermal analyser. A 30 mg material sample is placed in an MgO crucible and subjected to a thermal cycle under He 6.0 atmosphere. The material sample is heated to 1415 °C at a rate of 40 °C/min, held at that temperature for 30 minutes, and cooled down to room temperature at a rate of 5 °C/min. Images of the sample before and after the thermal processing are presented in Figure 1.

The morphology and composition of the material samples after thermal processing are assessed by Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectrometry (EDX), using a Hitachi SU-70 Field Emission SEM. Measurements are conducted at an acceleration voltage of 15 keV. The MgO crucible containing the sample is cast in epoxy resin and polished to obtain a vertical cross section of the sample.

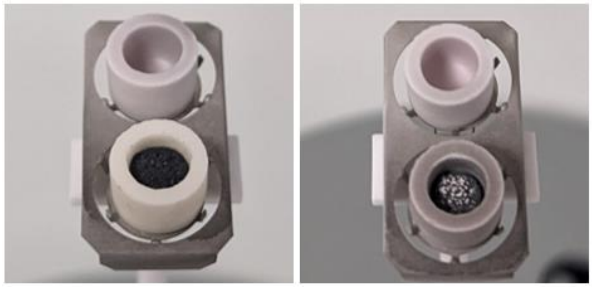

Figure 1: Images of the reduced LHS-1 sample before (left) and after (right) heating to 1415 °C.

Results & Discussion:

Thermal analysis: The TGA and DSC signals measured during the heating cycle are presented in Figure 2. During the heating phase, the DSC signal shows a wide endothermic bulge between 900 and 1030 °C but no clearly defined peak. This is attributed to the progressive melting of the heterogeneous starting material. Beyond 1030 °C, a significant fraction of the material is expected to be molten. During cooling, one main exothermic peak is seen at ca. 930 °C. This is attributed to the main (near-)isothermal crystallization process and is in line with the melting temperature range observed during heating. A second minor exothermic peak is seen at ca. 835 °C, which could indicate a second separate crystallization process. The TGA shows a progressive mass decrease during the heating and holding period, which is consistent with the Mg and Ca loss indicated by the EDX analysis below. The overall mass decrease measured between both passages at 400 °C is 7.6 % of the initial sample weight. Visual inspection of the sample after the heating process shows the formation of a solidified spherical "drop", with a white deposit at the surface.

Morphology & Composition: SE micrographs of the analysed material cross-section are shown in Figure 3. The average compositions of the outlined areas determined by EDX spectra quantification are given in Table 1. For comparison, the starting composition of the material before thermal processing reported previously is also included [4]. A map of the main recurring compositions identified in area 1 by EDX mapping

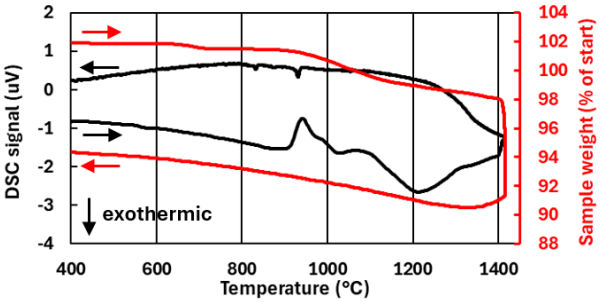

Figure 2: Measured DSC (black) and TGA (red) signal during thermal processing of reduced LHS-1.

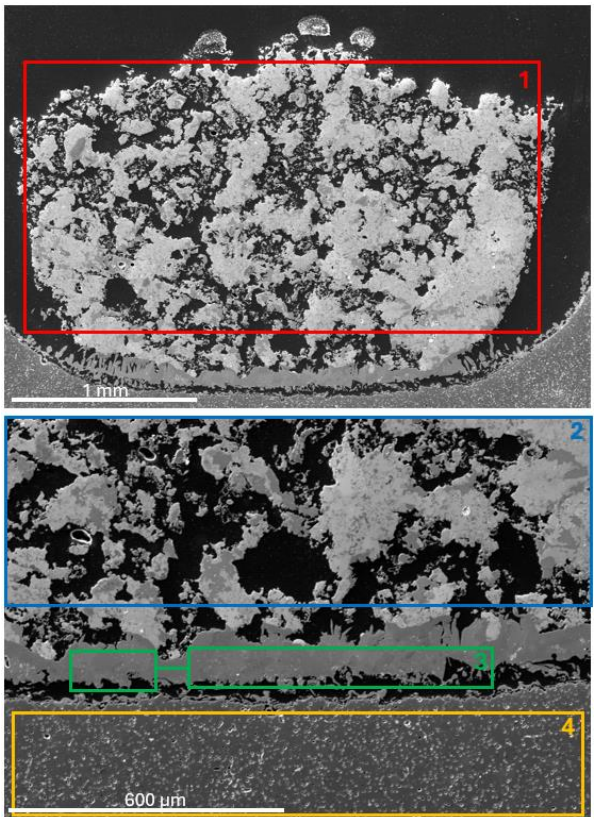

Figure 3: SE micrographs of the sample cross-section after thermal processing, at 40X (top) and 100X (bottom) mag., with EDX measurement areas.

followed by k-means clustering is presented in Figure 4. The corresponding cluster compositions are also listed in Table 1.

The metallic sample is consolidated but porous. Further consolidation has likely been inhibited by oxide films and, therefore, high interfacial energies. A distinct boundary layer exists between the main metallic body and the bottom of the crucible. The bulk material (areas 1 & 2) is significantly depleted of Mg and Ca when compared to the starting material. The boundary layer (area 3) is mostly composed of Al and Ca. The crucible still mostly contains Mg with minor traces of Al & Ca (area 4), indicating no uptake of material from the metallic charge.

The most abundant recurring compositions are Al-Si-Ca-Fe(-Ni) compounds (clusters 1 & 2). Cluster 1 approximately matches the composition of $Si_8Al_6Fe_4Ca$ [5], and cluster 2 could be a similar compound with Ni partially substituting for Fe. High Si content areas are also commonly found (cluster 4). However, those findings must be confirmed by complementary analyses (e.g. X-ray diffraction) before concluding on the nature of the phases being present.

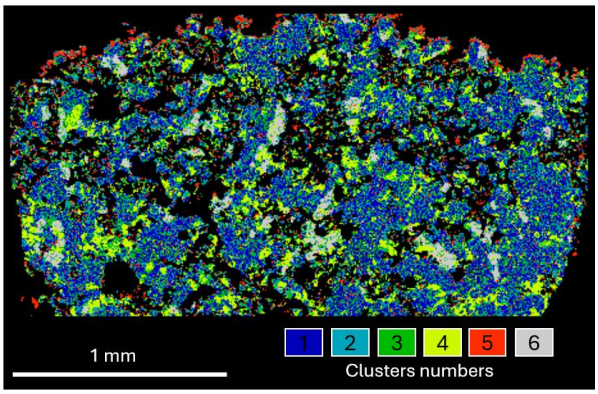

Figure 4: Map of main compositional clusters identified in area 1 by k-means clustering of EDX map.

	Composition (wt. %)										
	Start	Quantified areas				Composition clusters (area 1)					
		1	2	3	4	1	2	3	4	5	6
Mg	4	1	0	0	98	1	1	1	1	2	1
Al	22	22	21	50	1	22	30	21	7	44	9
Si	35	39	48	4	0	29	28	49	80	11	40
Ca	17	6	6	46	1	5	6	4	2	23	2
Ti	1	2	1	0	0	2	2	3	1	2	3
Cr	3	3	1	0	0	1	2	2	1	2	32
Mn	1	2	1	0	0	2	3	3	1	3	3
Fe	15	17	18	0	0	34	13	12	3	6	4
Ni	3	5	4	0	0	3	11	4	2	3	2
Mo	0	2	0	0	0	2	3	2	2	4	3
					Cluster pixel count :	21817	13159	12767	9203	5037	3716

Table 1: Average composition of starting material, areas of interest and composition clusters, determined by EDX spectra quantification (excl. O & C).

Conclusions: The present work is a first demonstration of thermal post-processing of metallic products from the molten salt electrolysis of regolith simulant. The consolidation of the obtained powdered material into a cohesive porous metallic ingot has been shown. The modification of the bulk metal content through partial evaporation of Ca and Mg and differentiated solidification of an Al-Ca rich compound has also been demonstrated. This is progress towards the refinement of the heterogeneous metallic mixtures produced by the reduction of lunar regolith, to obtain higher grade metallic alloys.

References: [1] Guerrero-Gonzalez F.J. & Zabel P. (2023) *Acta Astronautica*, 203, 187-201. [2] Lomax B.A., Conti M., Khan N., Bennett N. S., Ganin A. Y. and Symes M. D. (2020) *Planetary and Space Sciences*, 180, 104748. [3] Meurisse A., Lomax B.A., Selmeci A., Conti M., Lindner R., Makaya A., Symes M.D. and Carpenter J. (2022), *Planetary and Space Sciences*, 211, 105408. [4] Schild T., Lomax B.A., Conti M., Aridon G., Harries D. and Hadler K. (2025) *Acta Astronautica*, 232. 1-13. [5] Anglezio J.C., Servant C. and Ansara I. (1994) *Calphad*, 18, 311-318