Slag Chemistry on the Moon

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Keywords: Lunar Metal, Ferrosilicon alloys, Regolith, Lunar Metallurgy, Slag Chemistry, ISRU

ABSTRACT

The field of astrometallurgy is a growing area of study. Metal production on the Moon is likely to begin in less than a decade as major space agencies such as NASA and CNSA are preparing to construct permanent lunar bases. The utilisation of regolith to generate oxygen and metals is expected to be important in the development of a sustainable lunar habitat. This paper provides an overview of potential metallurgical processing methods for lunar conditions, emphasising the impact of lunar factors on slag chemistry. The interaction between regolith and the extracted metals, as well as their impact on slag composition and refractory performance, are critical considerations in metal production on the lunar surface. The initial research on lunar metallurgical technologies is focused on oxygen extraction and Fe-Si alloys are reported as the by-products of these processes. This paper delves into the fundamental thermodynamics associated with the carbothermic reduction of lunar regolith. Thermodynamic analysis using the FactSage software indicates that the Fe-Si alloy can be produced as a metallic product from the lunar regolith at temperatures ranging from 1400 ºC to 1600 ºC under terrestrial conditions. However, under ultra-high vacuum conditions (3×10^{-15} bar) on the Moon, the required operating temperature for producing ferrosilicon would be significantly reduced. Thermodynamic modelling results indicate that under lunar vacuum conditions, Fe-Si alloys only exist as solid phases at lower temperatures below 650 ºC. At higher temperatures, both Fe and Si are expected to be present in the gas phase. In this work, the carbothermic reduction of the Lunar Mare Simulant (LMS-1) is conducted under terrestrial conditions. The findings indicate that the process can readily yield Fe-Si alloys with 7 to 10 wt.% Si at 1600 ºC. Additionally, EDS analysis of the resulting Fe-Si alloy indicates the presence of phosphorus up to 1 wt.%.

INTRODUCTION

Metallurgical engineering technologies have played a crucial role in the development of societies on Earth. Expanding to the Moon is now the intended stated goal for several nations and space agencies, such as NASA, ISRO, ESA and CNSA, have already made their plans for the sustainable lunar exploration (Carpenter 2019; Mahoney 2020; *NASA: Artemis* 2020). NASA's Artemis missions, China's Change missions, Russia's Luna missions and India's Chandrayaan missions represent some of the ongoing major programs in the lunar exploration series. Moreover, several commercial ventures, such as SpaceX, Blue Origin, ORBITEC, Cislunar Industries, Lunar Resources Inc., and Shimizu Construction Company, are actively engaged in lunar exploration efforts (Ignatiev et al.

2018; Mackwell, Neal & Pestak 2019). For this kind of settlement, new metallurgical processes need to be developed that can work under extreme lunar conditions such as ultra-high vacuum of the order of 3×10^{-15} bar, low gravity (1/6th of that of the Earth) and large temperature fluctuations of 121 ºC to -133 ºC between lunar day and night near the Moon's equator. The knowledge of metallurgical processes on Earth acquired over many years could help in the development of the lunar metallurgical processes. In particular, the vast knowledge and experience of steelmaking on Earth can be leveraged to inform lunar steel production. Recovery of iron from the lunar regolith and processing into steel can potentially contribute to the establishment of a permanent lunar base.

A permanent lunar base would require a sustainable supply of metals and alloys; In-Situ Resource Utilization (ISRU) of lunar resources can provide the necessary metals for the lunar base. The metal resources on the Moon can be grouped into three categories: (a) metal produced from lunar regolith using ISRU, (b) recycled end-of-life products on the lunar surface, such as metals from the landers and rovers, and (c) outsourced metals from the Earth. Lunar regolith and rocks are source of oxygen and metals. Lunar regolith is primarily composed of metal oxides close in chemistry to mineral deposits found on Earth but with a relatively large content of fine glassy material formed from meteorite impacts. The composition of bulk lunar regolith varies across the lunar surface. The chemical composition of the lunar regolith suggests the presence of average 44.6 wt.% oxygen and several metal(loid)s like Si, Fe, Ca and Al (Duke et al. 2006). Extraction of the metals from lunar regolith using ISRU technologies can minimize the dependency on Earth for material supplies. Another source of metals on the Moon could be the end-of-life products on lunar surface. In 2012, NASA prepared a catalogue of human-made objects left on the lunar surface (NASA 2012). It was estimated that about 200,000 kg of such human-made materials are available on the lunar surface. The amount of these end-of-life products like landers, rovers on the lunar surface will continue to grow with the future missions unless their raw materials are recuperated.

Existing ISRU techniques like Molten Regolith Electrolysis (MRE), solar pyrometallurgical reduction and vacuum pyrolysis have shown their potential for oxygen and metal production (Sargeant et al. 2020; Schreiner 2015; Shaw et al. 2021; Sirk, Sadoway & Sibille 2019). These technologies are still in the development stage, with some prototypes having been tested under simulated lunar conditions (Shaw et al. 2021; Sirk, Sadoway & Sibille 2019). Alongside these metal extraction processes, the technologies for mining and beneficiation need to be proven to operate effectively in the lunar environment.

The literature published on ISRU technologies is primarily focused on extracting oxygen from the lunar soil as it is vital for human survival on the moon(Schlüter & Cowley 2020). Ferrosilicon (Fe-Si) alloys are reported as the major by-product of these ISRU technologies (Grossman et al. 2019; Samouhos et al. 2022). In the MRE process, Fe and Si are the first elements to be reduced from FeO and SiO_2 due to their lower dissociation potential compared to other elements like Al and Ca (Schreiner 2015). Carbothermal reduction is another potential ISRU technology to reduce lunar regolith(Gustafson et al. 2010; Kaur et al. 2024; Samouhos et al. 2022). This approach has been proposed for other extra-terrestrial application, such as iron production on Mars, where carbon is sourced from the CO_2 atmosphere (Nababan et al. 2022). Carbothermal reduction uses carbon sources such as C, CO, CH_4, etc. to reduce oxide species of lunar regolith, forming metal and CO/CO_2 according to the following reactions.

$$MeO_{(S)} + CO_{(g)} \rightarrow Me_{(S/l)} + CO_{2(g)} \quad \text{(Eq.1)}$$

$$MeO_{(S)} + C_{(s)} \rightarrow Me_{(S/l)} + CO_{(g)} \quad \text{(Eq.2)}$$

On the moon the carbon needs to be supplied from the earth or it can be recycled from the carbonaceous lunar waste. Gustafson et al. (2010) suggested that carbothermal reduction of molten regolith requires a temperature of over 1625 °C. At this temperature oxides of both Fe and Si can be reduced, and an oxygen yield of 28 kg per 100 kg regolith can be achieved. A recent study by Samouhos et al. (2022) synthesized granulated Fe-Si alloy with an average of 23.78 wt.% Si along with minor Al and Ti at 1300 °C using BP-1 lunar regolith simulant and carbon. Kaur et al. (2024) performed thermodynamic modelling study on carbothermal reduction of JSC-1A lunar simulant and predicted the favourable operating temperatures at three different pressures (10^{-1},10^{-8} and $3\text{x}10^{-15}$ bar) for Fe and Si production. They reported complete conversion of Fe_2O_3 to Fe gas above 850 °C and about 90 % conversion of SiO_2 to Si gas above 1000 °C under lunar conditions of $3\text{x}10^{-15}$ bar.

In this study, thermodynamic calculations have been performed to understand the primary products and by-products of carbothermal reduction of lunar regolith under earth and lunar conditions. The report discusses the effect of various carbon percentages, temperature and lunar conditions on the product chemistry and recommends the optimum processing condition for producing Fe-Si alloys using ISRU. Additionally, preliminary experimental results on the carbothermal reduction of lunar regolith simulant, LMS-1, are presented, indicating the formation of ferro-silicon alloys.

THERMODYNAMIC PREDICTIONS

Thermodynamic modelling is a critical tool to predict the equilibrium compositions based on Gibbs free energy minimization (Bale et al. 2016). In this study, FactSage 8.2 thermodynamic software was used to predict the composition of the products from the carbothermic reduction of lunar regolith under different pressures. FactPS, FSsteel and FToxid databases were used for the calculations. Thermodynamic modelling was conducted in three stages using the Equilibrium module. The first stage was to predict the optimum equilibrium conditions for the carbothermic reduction at Earth's conditions, i.e. at 1 bar pressure. These calculations were performed to design the experimental process on Earth and develop an understanding of the equilibrium phases and the possible metallic alloy extraction. These findings will also be potentially applicable for regolith processing inside a pressure-controlled human habitat on the Moon. The second stage of equilibrium calculations was performed on the lunar vacuum condition (3×10^{-15} bar). These calculations were aimed at predicting the slag composition, metallic alloy chemistry and off-gas composition over a range of temperatures and carbon content. The third stage of calculations was performed at an intermediate vacuum condition of 3×10^{-8} bar. The reason behind these calculations was that during a lunar carbothermal reduction process, the off-gas generated from the chemical reactions would potentially increase the pressure inside the reaction chamber. It is important to note that for such ultra-low pressures, the extrapolated thermodynamic data may not give the accurate results, as the experimental validation data becomes scarcer. However, qualitatively It will provide useful trends and insights of the process.

The composition of the lunar regolith varies across the lunar surface (Shaw et al. 2021). The composition of the regolith obtained from different lunar missions is shown in Table 1. It is clear from the table that FeO content significantly varies from below 5 wt.% to over 20 wt.% in all these samples. For this study, the composition utilized was that of LMS-1 regolith simulant provided by Exolith lab, which contains 8.6 wt.% FeO and is commonly used in studies related to regolith processing.

TABLE 1. Compositions of Lunar Regolith samples (Duke et al. 2006; Shaw et al. 2021)

Constituents (wt.%)	**Apollo 16 (Sample ID 64501)**	**Luna 24 (Sample ID 24999)**	**Apollo 17 (Sample ID 76501)**	**This study uses LMS-1 Simulant**
FeO	4.4	20.3	11.1	8.6
Al_2O_3	27.6	11.1	18.2	12.4
SiO_2	45.2	45.9	42.8	46.9
TiO_2	0.4	1	3.3	3.6
MgO	4.7	10.4	11.9	16.8
MnO	0.1	0.3	0.1	0.2
CaO	16.6	10.7	12.3	7
Na_2O	0.4	0.3	0.4	1.7
K_2O	0.1	0.0	0.1	0.7
P_2O_5	-	-	-	0.2

Processing on the Earth

The equilibrium calculations for carbothermal processing of lunar regolith on the earth were conducted for 100 g of regolith quantity and 1 bar pressure at different reduction temperatures and carbon content. Primary products obtained at different conditions were liquid alloy, liquid slag, gas and, in some cases, solid silicate phases. The percentage of these phases varied with carbon content and temperature. Figures 1a and 1b show the predicted quantity of liquid alloy and slag as the carbon and temperature vary.

Figure 1 indicates that at 800 °C, no liquid alloy or liquid slag formation is expected. The prediction suggests that liquid phases are anticipated to begin forming above 1000 °C. As the temperature increased to 1200 °C, a sharp rise in the quantities of both liquid alloy and liquid slag were observed, although the quantities remained unchanged with increasing carbon addition. As the temperature further increased to 1400 °C, the amount of liquid alloy was predicted to increase slightly, while the quantity of slag more than doubled. A slight increase in the liquid alloy quantity of 0.1 g can also be observed in Figure 1a when the carbon increased from 2 g to 4 g. Figure 1b shows the slag quantity increased from 39 g to almost 88 g across all the carbon content. As the temperature increased further to 1600 °C, the liquid alloy quantity is predicted to increase, however the slag quantity decreased. A clear declining trend can be seen for slag with increasing carbon content. This trend remains consistent at 1800 °C. The liquid alloy on the other hand jumped to its highest values at 1800 °C and at 10 g carbon. After 10 g carbon, the decline in liquid alloy quantity is attributed to the formation of carbide phases such as TiC and SiC. The predicted results also indicated that there was excess carbon remaining in the form of graphite.

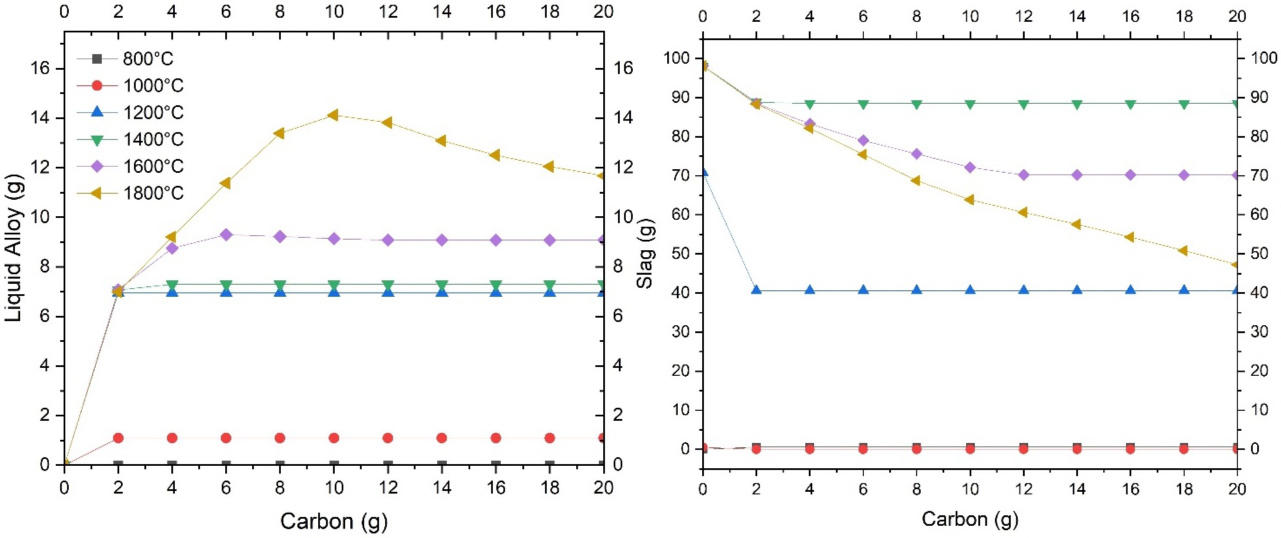

FIG 1 – (a) Predicted amount of liquid alloy and (b) liquid slag from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperature from 800 °C to 1800 °C at 1 bar.

To understand these trends further, the compositions of both liquid alloy and liquid slag were analysed. Figure 2 shows the predicted primary elements of the liquid alloy (Fe, Si and Ti) at four different temperatures, 1200 °C, 1400 °C, 1600 °C and 1800 °C. As can be seen from Figure 2, iron is the only major component of the liquid alloy at 1200 °C. Other elements such as C and P are also present in the liquid alloy, for e.g. reduction at 2 g C, results in a liquid alloy with 3.96 wt.% C and 1.26 wt.% P, equivalent to 0.275 g C and 0.088 g P. These elements are not shown in Figure 2 due to their smaller quantity. As the temperature increases to 1400 °C, silicon is predicted to start forming. With further rise in temperature to 1600 °C and 1800 °C, silicon quantity in Fe-Si alloy is predicted to increase. Silicon content in the Fe-Si alloy is also predicted to rise with increasing carbon content, reaching a maximum at about 6g carbon for 1600 °C and 10 g carbon for 1800 °C. Titanium is also predicted to be present in Fe-Si alloy at 1600 °C, and its quantity starts to rise with temperature. The graphs in Figure 2 illustrate that at temperatures exceeding 1600 °C, liquid alloys are anticipated to contain higher quantities of titanium and silicon.

These calculations indicate that the liquid alloys formed during the carbothermal reduction process of lunar regolith simulant LMS-1 consist of various compositions of Fe-Si alloy. Although the highest amount of liquid alloy is predicted to form at the highest reduction temperature of 1800 ºC, it contains higher percentages of silicon and titanium compared to lower temperatures. As the lower Si-containing Fe-Si alloys have a better balance between strength and ductility, lower temperatures of 1200 to 1400 ºC seem like an ideal temperature range. However, it is also important to look at other phases present in the products to determine the optimum temperature.

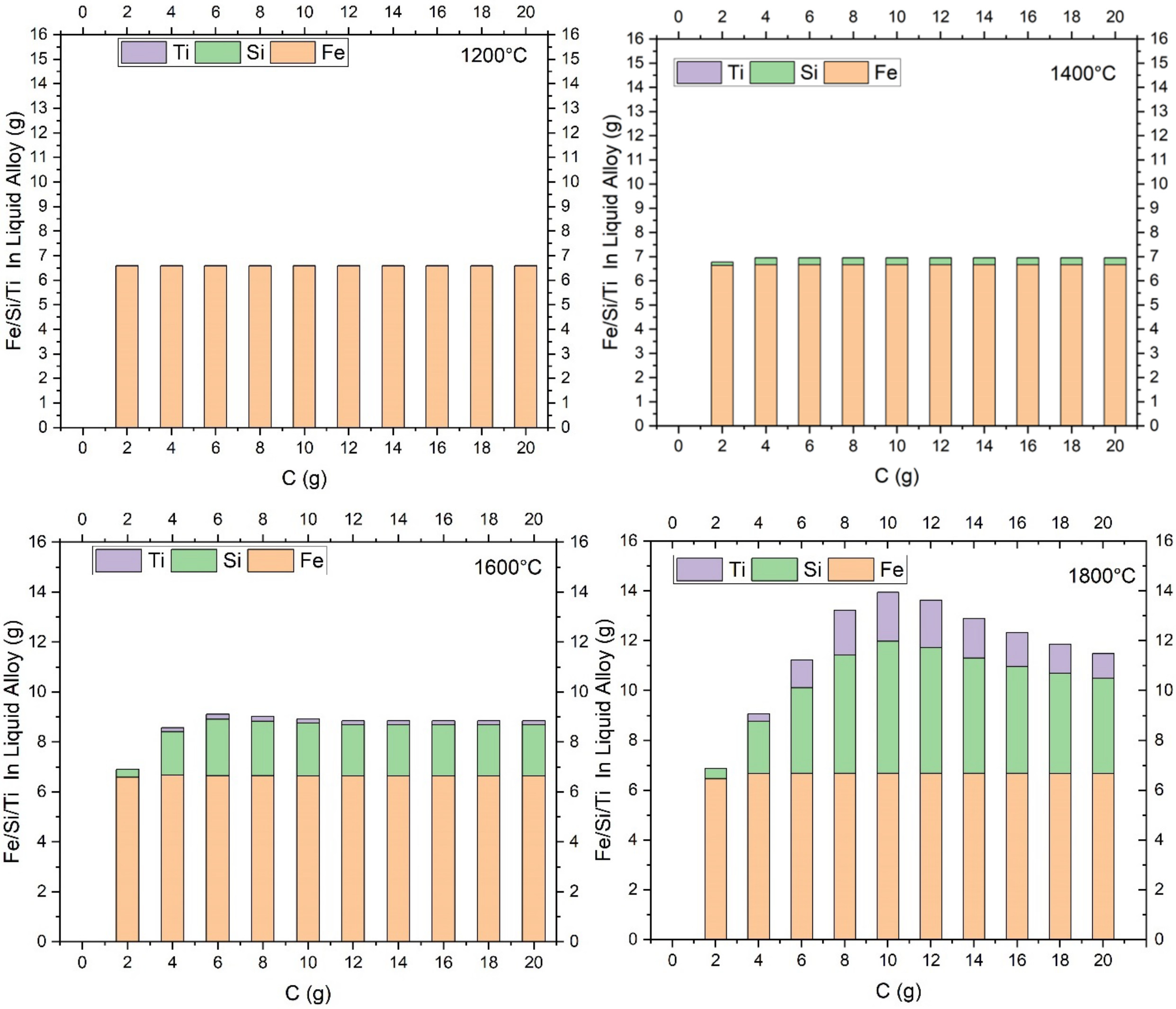

FIG 2 – Composition of liquid alloy predicted to be produced from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1200 ºC, 1400 ºC, 1600 ºC and 1800 ºC at 1 bar pressure.

Figure 3 shows the predicted composition of liquid slag at 1400 ºC and 1800 ºC. As can be seen in Figure 3, at 0 g carbon, FeO is present in the slag. With carbon addition, FeO is the only component that is fully reduced. CaO and MgO are present at all conditions but are not reduced to metal and, therefore, were not observed in the liquid alloy. At 1800 ºC, the notable difference in slag composition lies in the reduction of SiO_2 and TiO_2 levels with carbon, compared to 1400 ºC. This is because at higher temperatures, both SiO_2 and TiO_2 are reduced by carbon, forming Si and Ti that go into the liquid alloy. In addition, the metallic Si and Ti reacts with excess carbon and forms carbide phases. From Figures 2 and 3, 1400 ºC and 4 g, carbon appears to be sufficient for producing Fe-rich ferrosilicon alloys. However, separating and extracting the liquid alloy from the other products is also crucial in the effective recovery of the Fe-Si alloy. Therefore, it is crucial to understand the other products during the reduction.

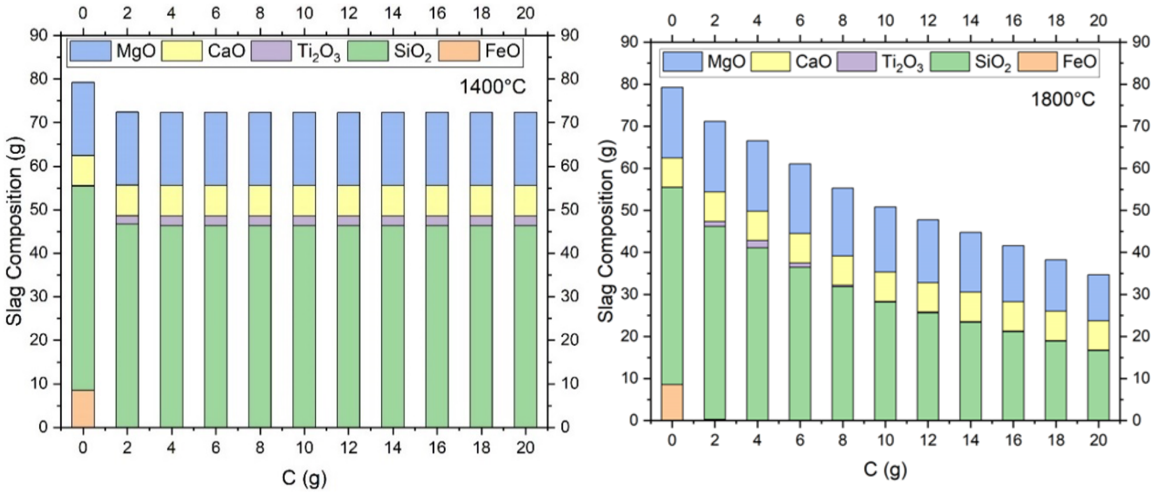

FIG 3 – Composition of liquid slag predicted to be produced from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1400 ºC and 1800 ºC at 1 bar pressure.

Figure 4 shows the stability of different phases that form during the carbothermal reduction of lunar regolith at 1200 ºC and 1400 ºC at 1 bar. At 1200 ºC, different silicate phases such as olivine, pyroxenes and feldspar are predicted to be present in the products in the solid state. These silicate phases coexist with liquid alloy, liquid slag and gas phases at all carbon percentages, as shown in Figure 4a. At 1400 ºC, these silicate phases are predicted to disappear. The liquid alloy phase will only coexist with liquid slag, and due to density difference, it can be separated from the slag. At higher temperatures, the quantity of slag is predicted to decrease, as shown in Figure 1b. This will increase both the quantity of liquid alloy and the gas phase. Therefore, 1400 ºC is the ideal temperature for processing the lunar regolith under the terrestrial conditions. In addition, the amount of slag, liquid alloy and gas plateaus after 4 g carbon at 1400 ºC.

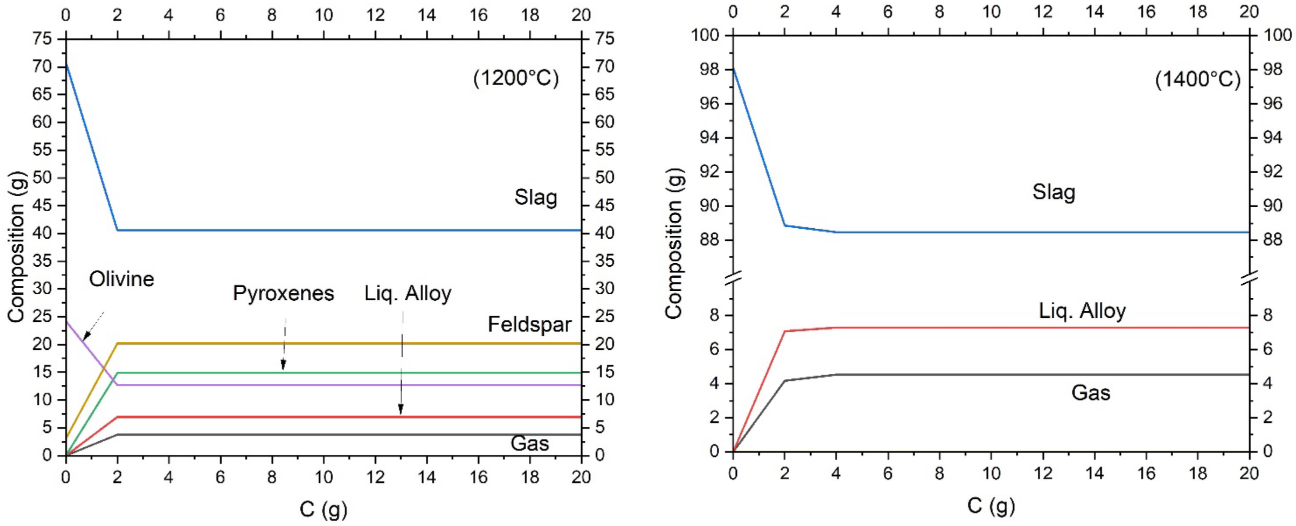

FIG 4 – Predicted equilibrium compositions from the carbothermal reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1200 ºC and 1400 ºC at 1 bar.

It is important to note that the primary aim of the ISRU technologies on the moon is to produce oxygen. Therefore, the aim of the carbothermal reduction process is to generate as much gas as possible to recover the oxygen. Figure 5 shows the variation in equilibrium compositions with reduction temperature at fixed carbon amounts of 4 g and 6 g. The graph indicates that the amount of gas increases with temperature and carbon. Figures 5 shows that after about 1350 ºC, the primary constituents of the products are gas, liquid alloy, and liquid slag. A small amount of carbide phases such as TiC and SiC are also predicted to form after 1450 ºC.

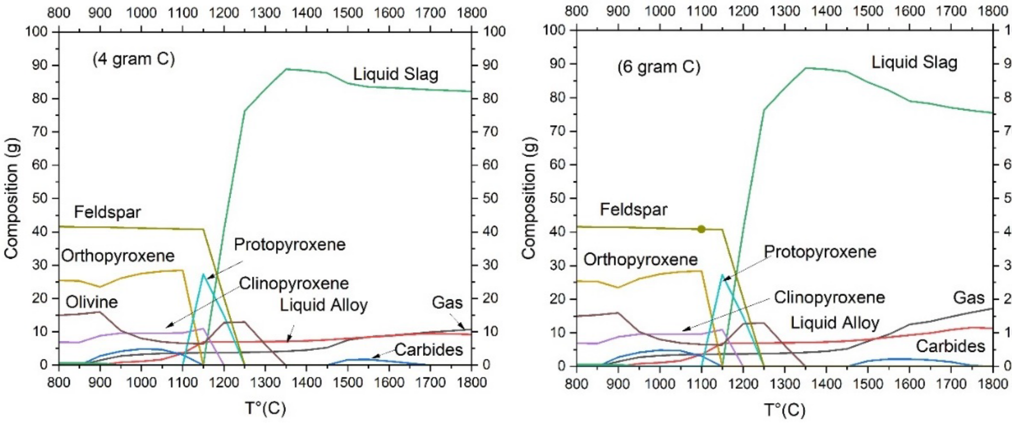

FIG 5 – Predicted equilibrium compositions of the products from carbothermal reduction of 100 g lunar regolith at various reduction temperatures at 4 g and 6 g carbon.

Processing on the Moon

The equilibrium calculations for carbothermal processing of 100 g lunar regolith simulant (composition shown in Table 1) were conducted at 3x10^{-15} bar pressure and 6 g carbon, and the equilibrium product composition under lunar vacuum conditions is shown in Figure 6. Few minor phases have been removed from the phase diagram to avoid clutter. As can be seen in Figure 6a, there is no liquid slag or liquid alloy phase in the product. Gas is the major phase. The products include various silicate phases, such as different types of pyroxenes and feldspar, at temperatures below 1000 ºC. Corundum forms between 1000 ºC to 1150 ºC. The quantities of Fe-containing phases are very small compared to the other phases. The magnified phase diagram shown in Figure 6b shows the presence of four different solid phases of Fe: BCC phase, FCC phase, Fe-Si and Fe_5Si_3 alloys. At lower temperatures of below 350 ºC, the majority of Fe is present in the BCC phase. After 350 ºC, the FCC phase starts to form. Both FeSi and Fe_5Si_3 alloys start to form at 400 ºC. Initially, the quantity of the FeSi phase increases with an increase in temperature, reaching its peak at 500 ºC, and then decreases, finally disappearing at 600 ºC. Similarly, the Fe_5Si_3 intermetallic compound initially increases until 450 ºC and disappears after 500 ºC. Beyond 600 ºC, no solid phases containing Fe are observed in the phase diagram, indicating that all the Fe is predicted to have evaporated. After 900 ºC Feldspar is predicted to vaporize into different gas components, few minor-concentration phases such as $Mg_4Al_{10}Si_2O_{23}$, $CaTiO_3$, $CaAl_4O_7$ have been omitted from the phase diagram to mitigate visual complexity In Figure 6.

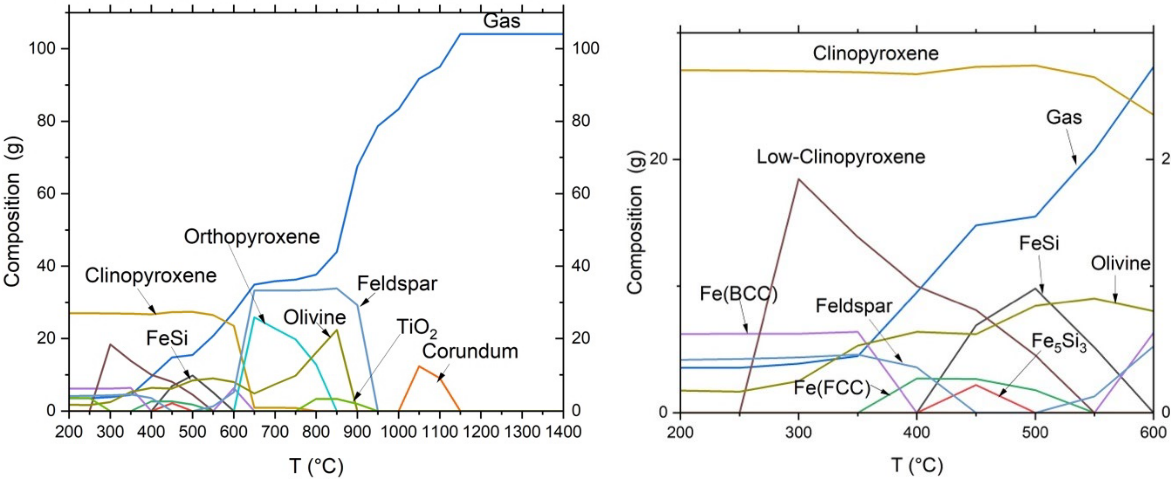

FIG 6 – (a) Predicted equilibrium compositions of the products from the carbothermal reduction of 100 g lunar mare simulant (LMS-1) at 6 g carbon and 3×10^{-15} bar pressure, (b) Magnified diagram

As gas is the major product of the carbothermal reduction of regolith under lunar vacuum conditions, the predicted composition of the gas phase with temperature is shown in Figure 7. Figure 7a shows that the gas phase is predicted to start forming from as low as 200 ºC reduction temperature with CO gas as the major constituent. With the rise in temperature to over 350 ºC, Na and K are predicted to appear in the gas phase. The formation of SiO gas starts at about 500 ºC and increases as the temperature rises. Upon comparing Figures 6 and 7, it is evident that initially, the SiO gas forms from the Si present in the FeSi alloy. Subsequently, between 600 ºC to 950 ºC, the SiO gas is produced from all the silicate phases containing SiO_2. Figure 7b shows the magnified image of Figure 7a at higher temperatures. The figure suggests that Fe is predicted to vaporise at about 600 ºC, and the quantity of Fe gas reaches a plateau after about 700 ºC. With further rise in temperature, Mg evaporates, followed by Ca and Al. The quantity of gas phase reaches its maximum at 1150 ºC and plateaus afterwards. Vacuum distillation process can be used to recover these metallic vapour phases although it has its own technical challenges. In order to recover Fe metal and especially the FeSi alloys, a reduction temperature of 400 ºC to 600 ºC could be used, and the resulting product may undergo grinding and magnetic separation process to potentially eliminate silicate phases. It is crucial to highlight that these predictions have not taken reaction kinetics into consideration. Hence, experimental research is necessary to confirm these predictions and gain insights into the kinetics of the reaction.

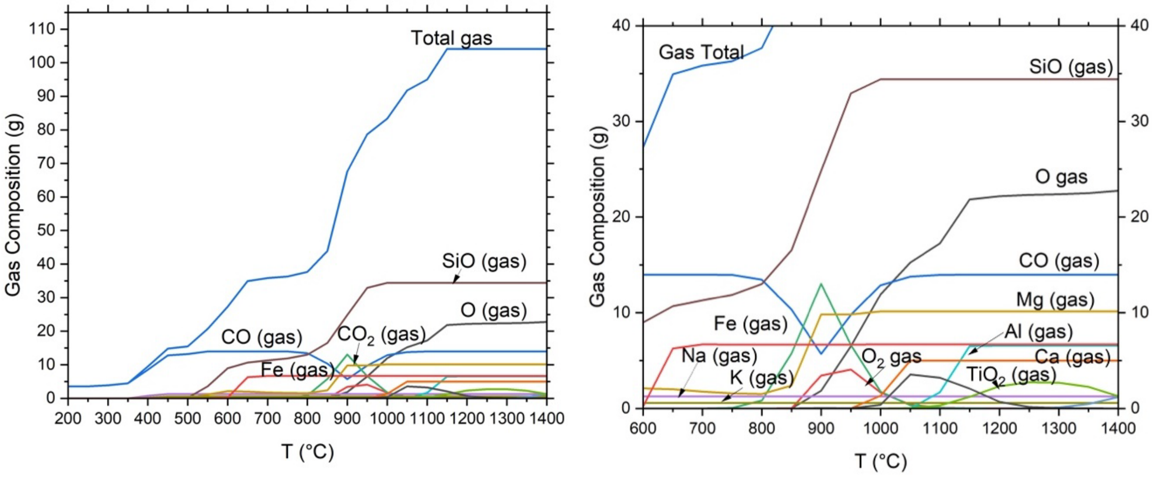

FIG 7 – (a) Equilibrium compositions of the products from the carbothermal reduction of 100 g lunar regolith at 6 g carbon and 3×10^{-15} bar pressure, (b) Magnified diagram

Processing at Intermediate Vacuum Condition

As mentioned earlier, the gases produced during the carbothermal reduction will increase the vacuum pressure inside the reaction chamber. Kaur et al. (2024) assumed a pressure of 10^{-8} bar in their study to account for the pressure increases due to the evolving gases. In this study, equilibrium calculations were performed at 3×10^{-8} bar pressure to understand the products of carbothermic reduction. Like the previous equilibrium calculations, 100 g regolith was reduced using 6 g carbon and the equilibrium phases at different temperatures are plotted, as shown in Figure 8. Under this intermediate vacuum condition, although the liquid slag phase is forming in the temperature range of 1150 ºC to 1500 ºC, metals stay in the solid or gaseous form. As with the previous diagrams, different types of silicate phases are shown to form at different temperatures. The FeSi alloy is stable in the temperature range of 650 ºC to 900 ºC. The magnified image shown in Figure 8b suggests the presence of Fe_5Si_3 alloy until 950 ºC. The key takeaway from this figure is that even under this intermediate vacuum condition, the Fe and FeSi alloys are still in solid form and need to be ground and magnetically separated to extract the Fe/Fe-Si alloys from the silicate phases.

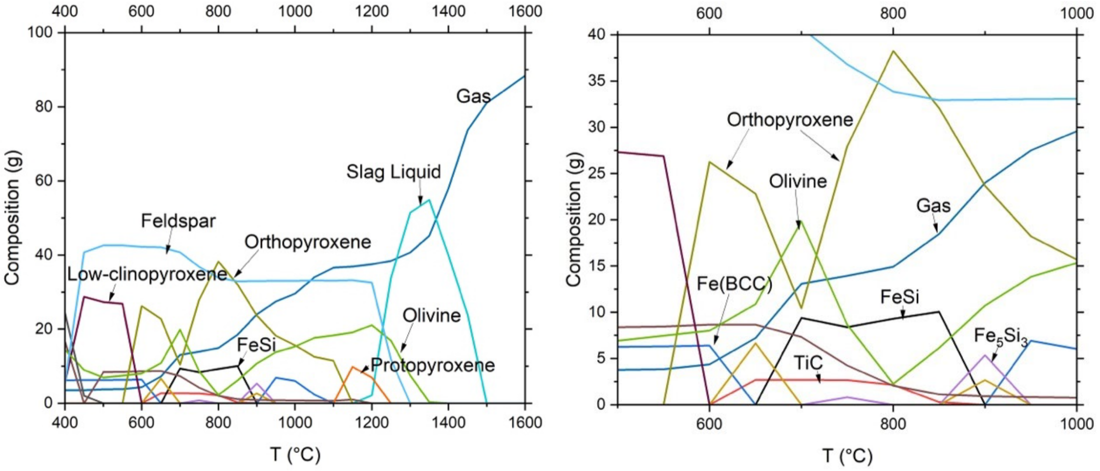

FIG 8 – (a) Equilibrium compositions of the products from the carbothermal reduction of 100 g lunar regolith at 6 g carbon and 3×10^{-8} bar pressure, (b) Magnified diagram.

In order to estimate the optimum vacuum pressure to recover liquid Fe-Si alloys, equilibrium calculations were performed at different pressures ranging from 1 bar to 10^{-15} bar, and the results are shown in Figure 9. The figures suggest that at both 1200 ºC and 1400 ºC, the liquid alloy coexists with liquid slag in the pressure range of 1 to 10^{-5} bar. The amount of both liquid slag and liquid alloy is predicted to be higher at 1400 ºC. For both cases, the liquid slag is present until 10^{-9} bar. From these graphs, it is evident that the pressure inside the reaction chamber should be more than 10^{-5} bar to recover liquid Fe-Si alloys. The density difference between the liquid slag and liquid alloy should ensure easy separation. At higher vacuum conditions, FeSi alloys need to be extracted in solid form by grinding and magnetic separation. Fe vapour can also be extracted from the gas phase using vacuum distillation.

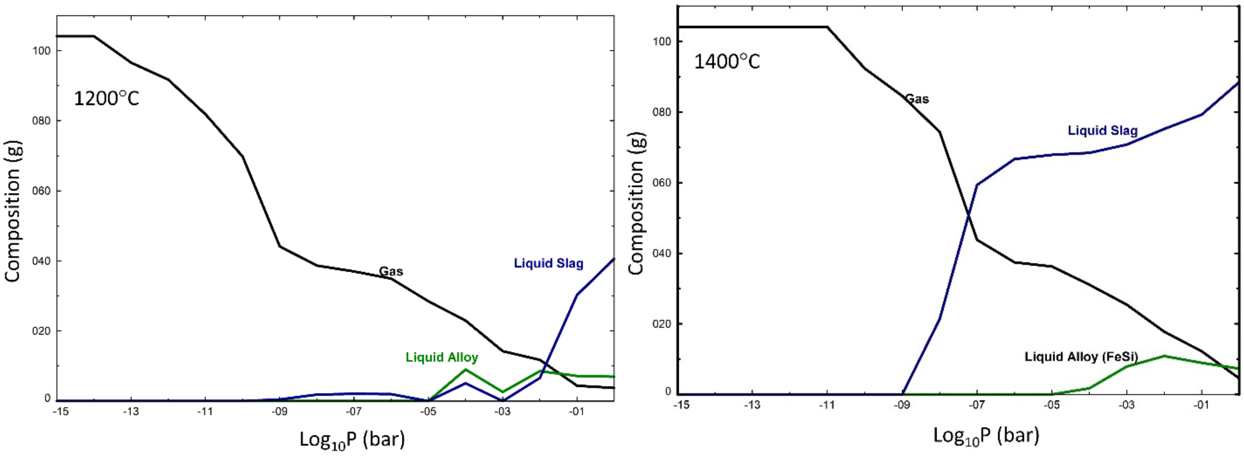

FIG 9 – (a) Predicted products of carbothermic reduction of 100 g LMS-1 simulant with 6 g carbon at different pressures at (a) 1200 ºC and (b) 1400 ºC.

Figure 10 presents a schematic summary of the predicted product phases resulting from carbothermal regolith reduction under three distinct pressure conditions at 1400 ºC. As explained earlier, under earth's conditions of 1 bar pressure, both liquid Fe-Si alloy and liquid slag phases forms, while at pristine lunar vacuum conditions and intermediate pressure of 3×10^{-8} bar, Fe and Si (in the form of SiO) joins the gas phase.

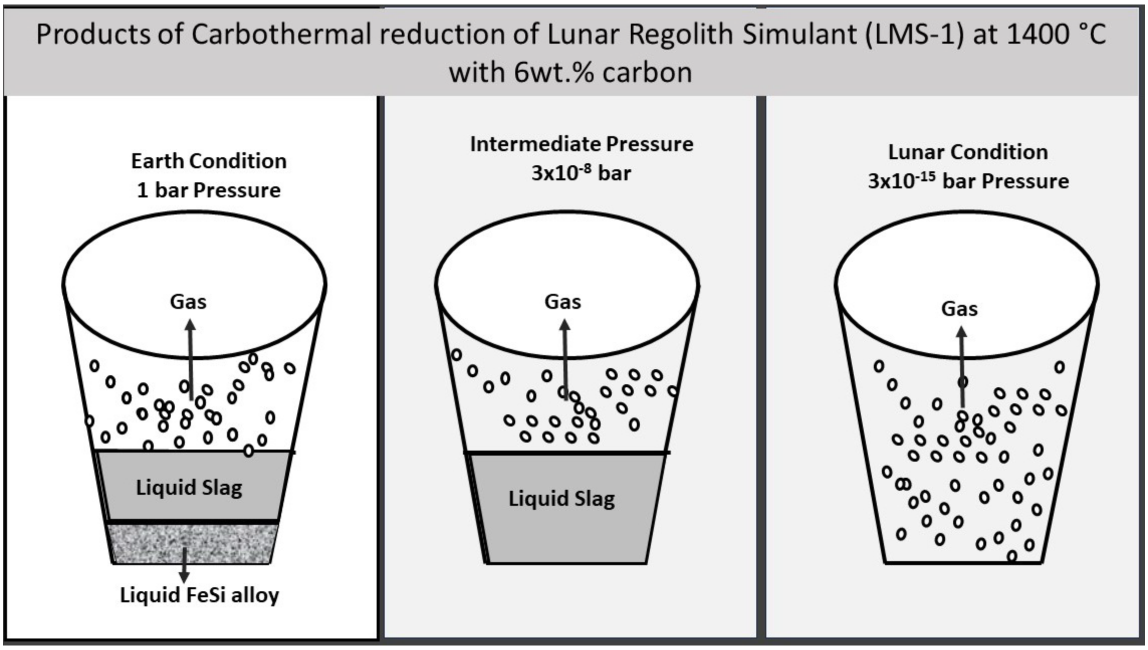

FIG 10 – Schematic diagram showing predicted results of carbothermal reduction of 100 g LMS-1 at 1400 ºC and 6 g C at different pressures.

EXPERIMENTAL FINDINGS

The thermodynamic calculations at Earth conditions suggests a minimum of 4 g carbon (i.e. 4 wt.% of the regolith) and 1400 ºC temperature as the optimum condition to produce liquid Fe-Si alloy with less impurities. At higher reduction temperatures, higher amounts of Si and other elements are present in the Fe-Si alloy and increase the quantity of liquid alloy. For the experimental investigation presented in this study, the aim was to prove that a liquid FeSi alloy can be produced from the lunar regolith. Therefore, a higher reduction temperature of 1600 ºC and a higher carbon amount of 10 wt.% was chosen to ensure liquid Fe-Si alloy forms during the experiment.

The experiments were conducted using a resistance-heated horizontal tube furnace from Nabertherm. 30 g regolith sample was mixed with 10 wt.% (i.e. 3 g) graphite using mortar and pestle, followed by loading the mixture in an alumina crucible. The crucible was then loaded into the hot zone of the furnace. The furnace was then heated at 200 ºC/hr until the desired 1600 ºC was achieved at the hot zone. The experiments were conducted under an inert atmosphere of argon gas, and the sample was cooled inside the furnace. The next day, the sample was taken out in a cold condition. Figure 11 shows the sample before and after the experiment.

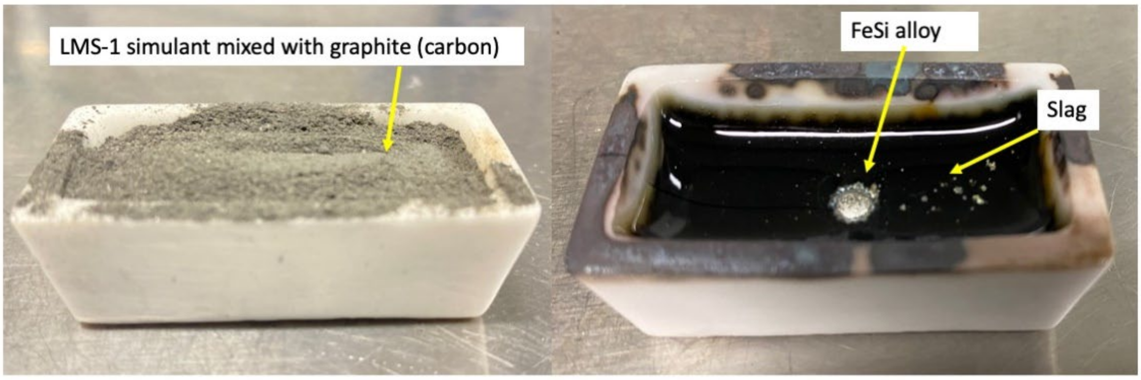

FIG 11 – (a) Mixture of LMS-1 simulant and graphite before experiment, (b) product of carbothermal reduction showing FeSi alloy at centre.

The product, as shown in Figure 11b, contains a glassy slag phase and a metal phase. The appearance of the metal phase suggests that the metal was in liquid form at the temperature of 1600 ºC. The crucible was broken using a hammer, and the metal phase was collected and analysed using a scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) for microstructure and chemistry. The results are shown in Figure 12.

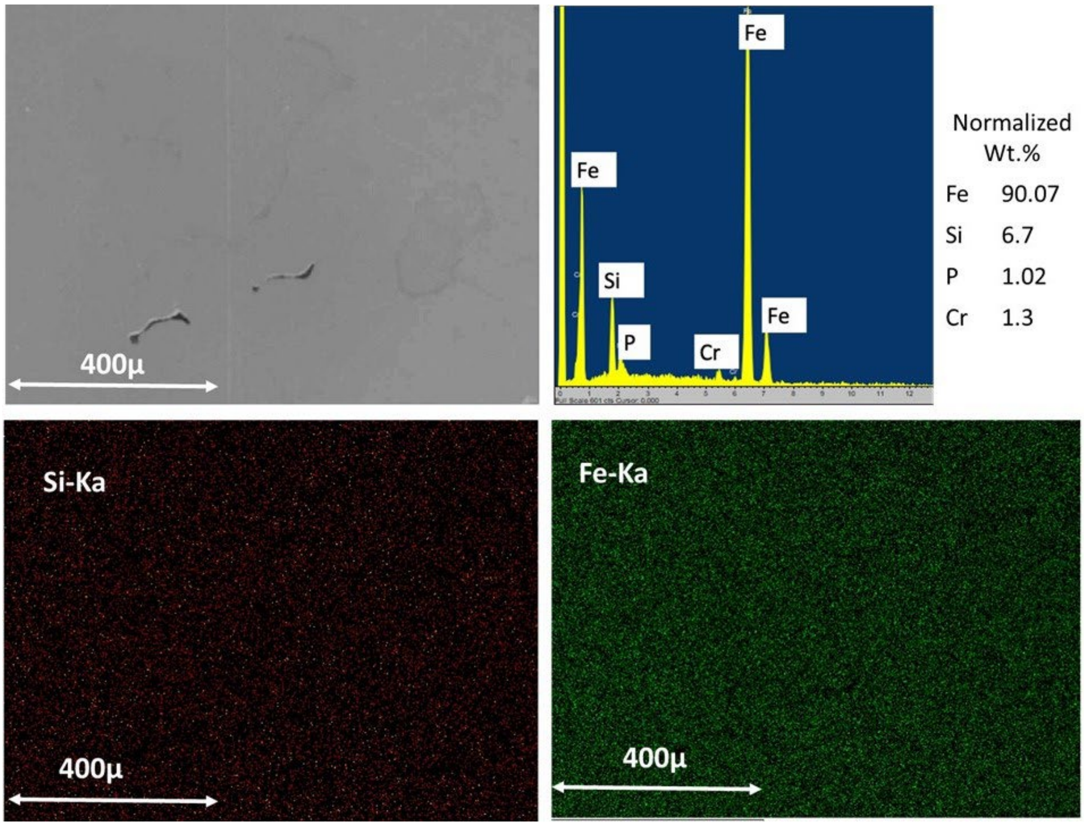

FIG 12 – (a) Mounted Fe-Si alloy in SEM, (b) EDS Spectrum and semi-quantitative analysis, (c) Si distribution map and (d) Fe distribution map.

The EDS spectrum and the semiquantitative analysis suggest the metal phase is a Fe-Si alloy with 90 wt.% Fe and about 7 wt.% Si, with minor P and Cr presence. Although Chromium is seen in the EDS spectrum, it is likely to be an impurity added during the sample cutting process as the cutting blade contains chromium and the initial regolith did not contain chromium. The presence of phosphorous could be critical in the further refining process of the Fe-Si alloy. As indicated by the thermodynamically predicted results, titanium is expected to be present in the liquid alloy; however, it was not detected in the EDS analysis conducted in this study. This discrepancy might be attributed to the fact that only the bottom part of the solidified liquid alloy was analysed under EDS. Moreover, the inclusion of impurities such as water and oxygen in bottled argon gas could have impacted the findings, as titanium has the capacity to scavenge oxygen in argon, potentially resulting in its retention within the slag phase. A comprehensive investigation is necessary to ascertain the presence of titanium in the liquid alloy. The hardness of this metal phase was also measured using a Vicker Hardness tester, and the average hardness value was found to be 515 ± 1% VHN.

CONCLUSIONS

In this paper, the thermodynamic study of the carbothermal reduction behaviour of lunar mare simulant LMS-1 was carried out under three different pressure conditions: 1 bar pressure to simulate Earth-like conditions, lunar vacuum condition of 3×10^{-15} bar pressure and an intermediate 3×10^{-8} bar pressure to account for the pressure rise due to gas evolution. The optimum thermodynamic conditions for carbothermal reduction of regolith and FeSi alloy extraction on the Earth are a minimum of 4 g carbon per 100 g regolith and a reduction temperature of 1400 ºC. Under both lunar conditions and an intermediate vacuum of $3\text{x}10^{-8}$ bar, iron can be extracted either in a vapour phase or as a solid phase in combination with other elements, as a liquid metal/alloy phase is not stable at these pressure conditions. In order to recover Fe-Si alloys in liquid form at this temperature, the vacuum pressure needs to be less than 10^{-5} bar. This paper also discusses the successful synthesis of Fe-Si alloy with 90 wt.% Fe from the lunar mare simulant LMS-1.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support of Swinburne University of Technology to undertake this research.

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