Slag Chemistry on the Moon

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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 the permanent lunar base. Utilisation of regolith to generate oxygen and metals is expected to be important in the development of 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 indicates that the primary by-products of these processes will be Fe-Si alloys, along with oxygen as the main product. The 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°C to 1600°C under terrestrial conditions (1 bar pressure). However, under ultra-high vacuum conditions (3x10⁻¹⁵ 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°C. 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 10wt.% Si. Additionally, EDS analysis of the resulting Fe-Si alloy indicates the presence of phosphorus up to 1wt.%. Hardness of the Fe-Si alloy is measured to be around 510 to 520 VHN.

INTRODUCTION

Metallurgical engineering technologies have played a crucial role in the development of societies on earth. Now settling on the moon seems like the next big goal of humanity. For this kind of settlement, new metallurgical processes need to be developed which can work with extreme lunar conditions such as ultra-high vacuum of the order of $3x10^{-15}$ bar,1/6 gravity compared to earth's gravity and uneven temperature during lunar day and night. 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 beneficial to lunar steel production. Steel is the most used material on earth due to its wide range of applications. Recovery of iron from the lunar regolith and making steel out of it can significantly contribute to the establishment of a permanent lunar base.

A permanent lunar base demands 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 production using ISRU, (b) recycling the end-of-life products on lunar surface such as recovery of metals from the landers and rovers (c) outsourced metals from the earth. Lunar regolith and rocks are excellent sources of oxygen and metals. Lunar regolith is primarily composed of metal oxides close in chemistry to mineral deposits found on the 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 44.6% oxygen and several other metals like Si, Fe, Ca and Al [1]. Another source of metals on the moon could be the end-of-life products on lunar surface. In 2012, NASA prepared a catalogue of man-made objects left on the lunar surface [2]. It was estimated that about 200,000 kg of man-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 they are reused and recycled.

Extraction of the metals from lunar regolith using ISRU technologies can minimize the dependency on Earth for material supplies. Existing ISRU techniques such as Molten regolith electrolysis (MRE), solar pyrometallurgical reduction, vacuum pyrolysis, and other technologies, have shown their potential for oxygen as well as metal production [Ref]. These technologies are still in the development stage, with some prototypes having been tested under simulated lunar conditions [3], [4]. Alongside these metal extraction processes, the technology for mining and beneficiation needs to be proven to operate effectively in the lunar environment. The literatures available on ISRU technologies are primarily focused on extracting oxygen from the lunar soil as it is vital for human survival on the moon [Ref]. Ferrosilicon (Fe-Si) alloys are reported as the major by-product of these ISRU technologies [Ref]. In the MRE process, Fe and Si are the first elements to be reduced due to their lower dissociation potential compared to other elements like FeO and SiO₂ [5]. Carbothermal reduction is another potential ISRU technology to reduce lunar regolith, forming metal and CO₂ according to the following reaction.

$$MeO_{(S)} + CO_{(g)} \rightarrow Me_{(S/l)} + CO_{2(g)}$$

Gustafson et al. [6] 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. [7] 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. Michail et al. synthesized Fe-Si (Ti-Al) alloy from magnetite-enriched BP-1 lunar regolith simulant at 10⁻² bar pressure and a two-stage reduction and melting process [8].

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.

THERMODYNAMIC PREDICTIONS

Thermodynamic modelling is a critical tool to predict the equilibrium compositions based on Gibb's free energy minimization. In this study FactSage 8.1 thermodynamic software was used to predict the compositions of the products from the carbothermic reduction of lunar regolith under different pressure. FactPS, FSsteel and FToxide 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 on the earth's conditions i.e. at 1bar 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 $(3x10^{-15} \text{ bar})$. 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 $3x10^{-8} \text{ bar}$. The reason behind these calculations was that during a lunar carbothermal reduction process, the off gas generated from the chemical reactions will potentially increase the pressure inside the reaction chamber.

The composition of the lunar regolith varies differently across the lunar surface [Ref]. 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 5wt.% to over 20wt.% 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	(Shaw et al. [8])
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Constituents	Apollo 16 (Sample	Luna 24 (Sample ID	Apollo 17 (Sample	This study uses
(Wt.%)	ID 64501)	24999)	ID 76501)	LMS-1 Simulant
FeO	4.4	20.3	11.1	8.6
AI_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 ₂ O	0.4	0.3	0.4	1.7
K_2O	0.1	0.0	0.1	0.7
$P_{2}O_{5}$	-	-	-	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 atmospheric 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 shows 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 both liquid alloy and liquid slag were observed, although the quantity remained unchanged with increasing carbon quantity. As the temperature further increased to 1400°C, the amount of liquid alloy was predicted to slightly increase, while the quantity of slag more than doubled. A slight increase in the liquid alloy quantity of 0.1g can also be observed in Figure 1a when the carbon increased from 2g to 4g. Figure 1b shows the slag quantity jumped from 39g to almost 88g across all the carbon content. As the temperature increased further to 1600°C, the liquid alloy quantity is predicted to increase by about 28 wt.%, 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 10g carbon.

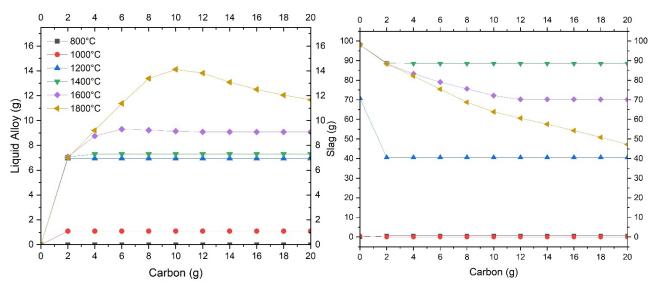
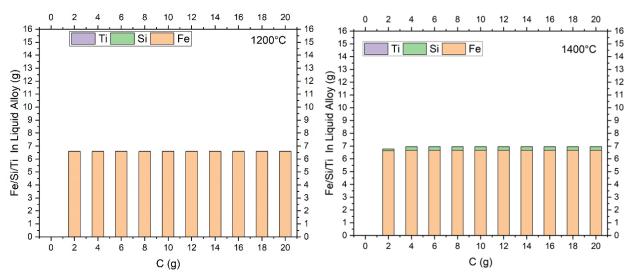


FIG 1 – (a) Predicted amount of liquid alloy and (b) liquid slag from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperature from 800° C to 1800° C at 1 bar.

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 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 are negligible. 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 10g 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 consists 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°C, it contains higher percentages of silicon and titanium compared to lower temperatures. As the lower Sicontaining Fe-Si alloys have a better balance between strength and ductility, lower temperatures of 1200 to 1400°C 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.



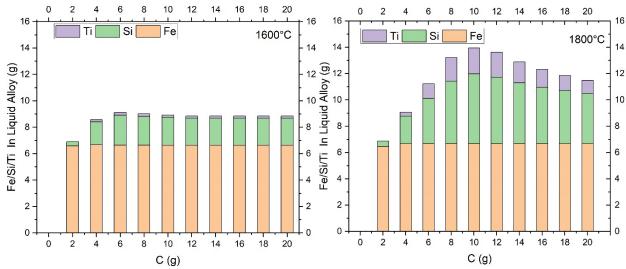


FIG 2 – Composition of liquid alloy predicted to be produced from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1200°C, 1400°C, 1600°C and 1800°C at 1 bar pressure.

Figure 3 shows the predicted composition of liquid slag at 1400° C and 1800° C. As can be seen in Figure 3, at 0g 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° C, the notable difference in slag composition lies in the reduction of SiO₂ and TiO₂ levels with carbon, compared to 1400° C. This is because at higher temperature, both SiO₂ and TiO₂ is reduced by carbon, forming Si and Ti that goes to the liquid alloy.

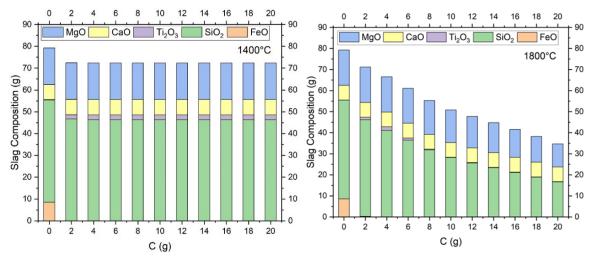


FIG 3 – Composition of liquid slag predicted to be produced from the carbothermic reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1400°C and 1800°C at 1 bar pressure.

From figure 2 and 3, 1400°C and 4g carbon appears to be sufficient for producing Fe-rich ferrosilicon alloys. However, to separate and extract 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. Figure 4 shows the stability of different phases that form during the carbothermal reduction of lunar regolith at 1200°C and 1400°C at 1bar. At 1200°C different silicates phases such as olivine, pyroxenes and feldspar are predicted to be present in the products in solid state. These silicates phases coexist with liquid alloy, liquid slag and gas phases at all carbon percentages as shown in Figure 4.a. At 1400°C, these silicate phases are predicted to be 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 as well as the gas phase. Therefore, 1400°C 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 4g carbon at 1400°C.

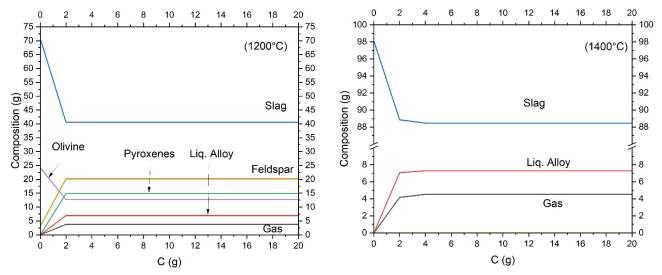


FIG 4 – Predicted equilibrium compositions from the carbothermal reduction of 100 g of LMS-1 simulant with variation in carbon content from 0 to 20 g and reduction temperatures at 1200°C and 1400°C at 1 bar.

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 4g and 6g. The graph indicates that the gas amount increases with temperature and carbon amount. The Figures 5 show that after about 1350°C, the primary constituents of the products are gas, liquid alloy, and liquid slag. A small amount of carbide phases is also predicted to form after 1450°C.

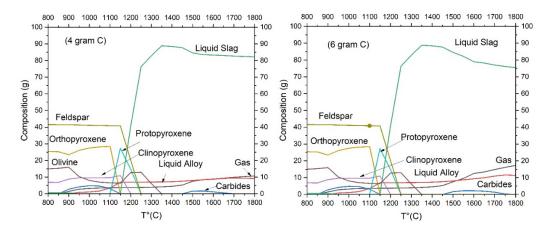


FIG 5 – Predicted equilibrium compositions of the products from carbothermal reduction of 100g lunar regolith at various reduction temperatures at 4g and 6g carbon.

Processing on the Moon

The equilibrium calculations for carbothermal processing of 100g lunar regolith simulant (composition shown in Table 1) were conducted at $3x10^{-15}$ bar pressure and 6g carbon and the equilibrium product composition under lunar vacuum conditions is shown in Figure 6. 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° C. Corundum forms between 1000° C to 1150° C. The quantities of Fecontaining 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 Fe₅Si₃ alloys. At lower temperatures of below 350° C, the majority of Fe is present in the BCC phase. After 350° C, the FCC phase starts to form. Both FeSi and Fe₅Si₃ alloy start to form at

 400° C. Initially, the quantity of the FeSi phase increases with an increase in temperature, reaching its peak at 500° C, and then decreases, finally disappearing at 600° C. Similarly, the Fe₅Si₃ alloy initially increases until 450° C and disappears after 500° C. Beyond 600° C, no solid phases containing Fe are observed in the phase diagram, indicating all the Fe has evaporated.

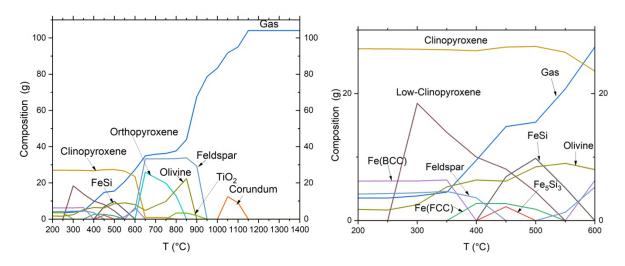
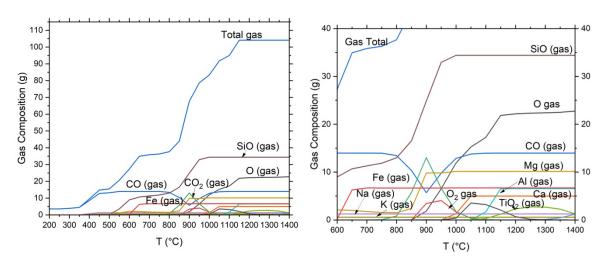


FIG 6 – (a) Predicted equilibrium compositions of the products from the carbothermal reduction of 100g lunar mare simulant (LMS-1) at 6g carbon and 3x10⁻¹⁵ bar pressure, (b) Magnified diagram

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°C reduction temperature with CO gas as the major constituent. With the rise in temperature to over 350°C, Na and K are predicted to appear in the gas phase. The formation of SiO gas starts at about 500°C and increases as the temperature rises. Upon comparing Figure 6 and 7, it is evident that initially, the SiO gas forms from the Si present in the FeSi alloy. Subsequently, between 600°C to 950°C, the SiO gas is produced from all the silicate phases containing SiO₂. Figure 7b shows the magnified image of Figure 7a at higher temperatures. The figure suggests that Fe vaporises at about 600°C and the quantity of Fe gas reaches a plateau after about 700°C. With further rise in temperature, Mg evaporates, followed by Ca and Al. The quantity of gas phase reaches its maximum at 1150°C and plateaus afterwards. Vacuum distillation process can be used to recover these metallic vapour phases. In order to recover Fe metal and especially the FeSi alloys, a reduction temperature of 400°C to 600°C could be used and the resulting product may undergo grinding and magnetic separation process to potentially eliminate silicate phases.



Processing at Intermediate Vacuum Condition (3x10⁻⁸ bar pressure)

As mentioned earlier, the gases produced during the carbothermal reduction will increase the vacuum pressure inside the reaction chamber. Kaur et al. [9] 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. Similar to the previous equilibrium calculations, 100g regolith was reduced using 6g 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° C to 1500° C, metals stay in the solid or gaseous form. Like the previous diagrams, different types of silicate phases form at different temperatures. The FeSi alloy is stable in the temperature range of 650° C to 900° C. The magnified image shown in Figure 8b suggests the presence of Fe_5Si_3 alloy until 950° C. 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 magnetic separated to extract the Fe/Fe-Si alloys from the silicate phases.

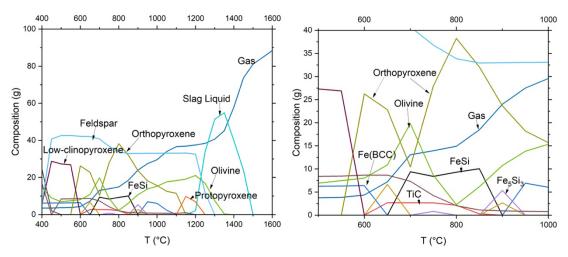


FIG 8 – (a) Equilibrium compositions of the products from the carbothermal reduction of 100g lunar regolith at 6g carbon and $3x10^{-8}$ bar pressure, (b) Magnified diagram.

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° C and 1400° C, 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 are predicted to be higher at 1400° C. 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.

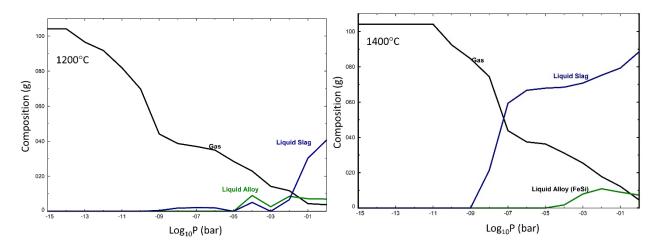


FIG 9 – (a) Predicted products of carbothermic reduction of LMS-1 simulant with 6g carbon at different pressures at (a) 1200°C and (b) 1400°C.

Figure 10 presents a schematic summary of the predicted product phases resulting from carbothermal regolith reduction under three distinct pressure conditions at 1400°C. As explained earlier, under earth's conditions of 1 bar pressure, both liquid Fe-Si alloy and liquid slag phases forms, while at lunar vacuum conditions and intermediate pressure of 3x10⁻⁸, Fe and Si (in the form of SiO) joins the gas phase.

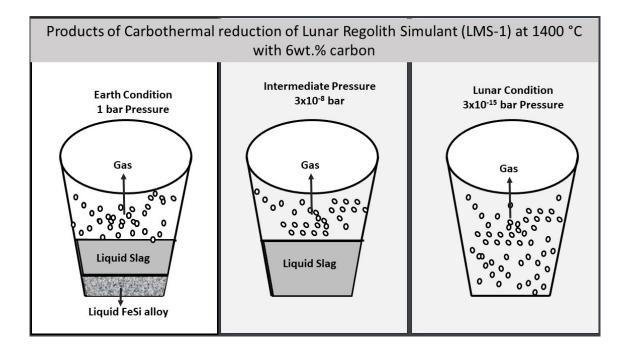


FIG 10 – Schematic diagram showing predicted results of carbothermal reduction of LMS-1 at 1400°C and 6wt.% C at different pressures.

EXPERIMENTAL FINDINGS

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

The experiments were conducted using a resistance-heated horizontal tube furnace. 30g regolith sample was mixed with 10% (i.e. 3g) 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°C/hr until the desired 1600°C was achieved at the hot zone. The experiments were conducted under the inert atmosphere of argon gas and the sample was cooled inside the furnace. The next day sample was taken out in cold condition. Figure 11 shows the sample before and after the experiment.

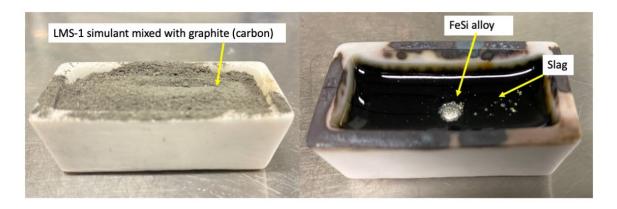


FIG 11 – (a) Mixture of LMS-1 simulant and graphite before experiment, (b) product of carbothermal reduction showing FeSi alloy at centre.

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

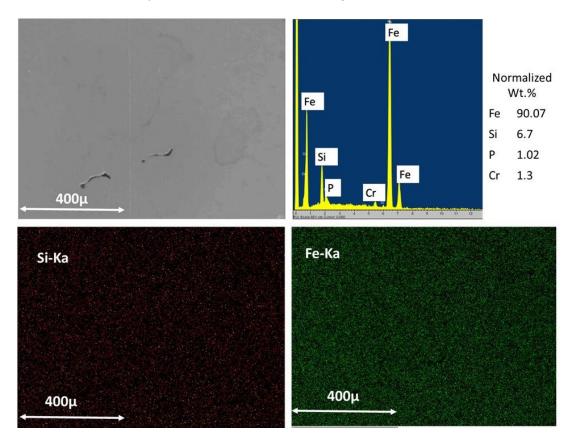


FIG 12 – (a) Mounted Fe-Si alloy in SEM, (b) EDS Spectrum and semi-quantitative analysis, (c) Si distribution map and (d) Fe distribution map.

The EDS spectrum and the semiquantitative analysis suggest the metal phase is a Fe-Si alloy with 90% Fe and about 7% Si, with minor P and Cr presence. Although Chromium is seen in the EDS spectrum, it might 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. The hardness of this metal phase was also measured using Vicker Hardness tester and average hardness value was found to be 520 HVN, indicating its potential suitability for mechanical applications.

CONCLUSIONS

In this paper, thermodynamic study of the carbothermal reduction behaviour of lunar mare simulant was carried out under three different pressure conditions; 1 bar pressure to simulate earth condition, lunar vacuum condition of $3x10^{-15}$ bar pressure and an intermediate $3x10^{-8}$ 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 4g carbon per 100g regolith and a reduction temperature of 1400° C. Under both lunar conditions and an intermediate vacuum of $3x10^{-8}$ 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 the vacuum pressure needs to be less than 10^{-5} bar. This paper also discusses the successful synthesis of Fe-Si alloy with 90% Fe from the lunar mare simulant LMS-1.

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