Interfacial Phenomena Occurring during Iron/Char Interactions in a Blast Furnace

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Using the sessile drop approach, interfacial reactions taking place in the iron/carbon interfacial region were investigated at 1550°C in a horizontal tube resistance furnace with an argon atmosphere. Two coal chars, labelled as 1 and 2 with respective ash concentrations of 10.88 wt% and 9.04 wt%, and electrolytically pure iron were used in this study. Liquid iron droplets were exposed to chars at high temperatures for times ranging between 1 to 180 min and the assembly was then withdrawn into the colder section quenching the droplet. To examine the time dependent growth of new phases formed in the interfacial region, FESEM and EDS investigations were carried out on the underside of the droplet, which effectively represents the iron/char interface. The transfer of carbon and sulphur into the iron droplet was also determined using a LECO Analyser. Interfacial regions for both chars showed a high occurrence of ash deposits, which were found to increase with time. Al, Ca, S, O, Fe were also detected in EDS analysis of the interface. However, very low levels of Si were found in the interfacial region despite high concentrations of silica in the chars initially suggesting chemical reactions involving silica. After three hours of contact, carbon pick-up by liquid iron reached only 0.12 wt% and 0.28 wt% for Char 1 and Char 2 respectively, both of which were much below the saturation level of 5.6 wt%. These results are discussed in terms of the formation of interfacial products, the consumption of solute carbon by reducible oxides and low intrinsic rates of carbon dissolution from non-graphitic Chars.

KEY WORDS: coal char; blast furnace; liquid Iron; carbonaceous materials; carbon dissolution.

1. Introduction

An understanding of iron/carbon interactions is of significance due to their extensive application in a number of metallurgical processes, especially in the blast furnace ironmaking process. One of the major advancements in the blast furnace technology has been the injection of pulverised coal through tuyeres to partially replace metallurgical coke as a source of heat and reductant. High injection rates for the pulverised coal (PCI), desirable for increasing the efficiency of the furnace, can seriously affect furnace stability with an increasing amount of unburnt char entering the blast furnace burden.1–4) After the unburnt char leaves the raceway, it could either be partially consumed within the furnace or escape along with the off-gases from the top of the furnace. The unconsumed char tends to collect at the interface between the softening zone and the dripping zone blocking gas flows, and its accumulation within a blast furnace is a major concern as it can adversely affect the permeability in the burden and therefore the stable operation of the furnace. The consumption of unburnt residual char can take place through reactions with the metal, slag and gaseous phases. It would be ideal for the unburnt char to actively participate in ore reduction reactions, consequently replacing some more of coke and also reducing high levels of unburnt solids in top off-gases. The main focus of this study is on interactions of coal chars with liquid iron, specifically carbon dissolution and interfacial phenomena. The dissolution of carbon from the char can make significant contributions to the carbonisation of liquid iron and can assist in reducing the consumption of coke at high rates of PCI, and the accumulation of the unburnt char within the blast furnace.

Coals are complex carbonaceous materials, which are only partially crystalline and contain high levels of ash and volatile matter. The transfer of carbon from coals is quite complex involving volatile release, carbon dissolution and impurity reactions. While there have been a large number of investigations on carbon dissolution from graphite,5–8) a number of recent studies9–11) have reported on cokes and coals as well. Carbon dissolution studies on graphite, both theoretical and experimental, have provided a great deal of information about the reaction kinetics and have generally concluded that mass transfer in the melt is the rate controlling mechanism for the overall carbon dissolution process. Such a generalisation may imply that the reactions at the interface do not control dissolution kinetics which, however, may not be the case for less ordered materials like coals and interfacial reactions could play an increasingly important role in their dissolution. Interfacial reactions occurring be-
tween ash-containing coals and liquid iron could also be quite different from those occurring between pure graphite and iron. A clear understanding of the processes occurring at the carbon/liquid iron interface is therefore of crucial importance.

Stationary rod studies of carbon dissolution from coke into liquid iron by Mourao et al.\textsuperscript{12} detected a viscous slag-like layer at the interface. This layer was composed of Al, Ca, Si, K, Na, Ti and P, and these elements were assumed to be in the form of oxides. These authors however did not provide the ash composition for coke, and therefore the changes in the ash layer during carbon dissolution could not be determined. It was suggested that a slag-like layer on the surface of the coke cylinders reduced the interfacial area available for carbon dissolution, and hindered the dissolution of carbon. In addition, coke was rather porous and could trap liquid iron in its pores. The trapped iron is likely to become saturated with carbon further limiting the area available for carbon dissolution.

Orsten and Oeters\textsuperscript{13} added CaO and CaF\textsubscript{2} to the coke to reduce the fusion temperature of coke ash and have reported high rates of carbon dissolution into liquid iron from cokes containing liquid ash. They have concluded that liquid ash was more easily removed from the interface by the action of stirring as compared to solid ash, and therefore would not hinder the transfer of carbon from coke into liquid iron. However two coke samples, containing different amounts of CaO and CaF\textsubscript{2} additives but with similar ash fusibility temperatures, had different dissolution rates. While it is not easy to explain the observed differences in dissolution rates, the results are nevertheless useful and suggest that the influence of ash fusion temperature, though important, may not be the only rate-limiting factor affecting carbon dissolution. It has been reported that ash can form an interfacial layer between liquid iron and carbonaceous material if its liquidus temperature is higher than the bath temperature. This ash-enriched layer is expected to contribute to surface blockage and will possibly retard the transfer of carbon into liquid iron. Using a sessile drop arrangement, Wu et al.\textsuperscript{14} investigated the interactions between natural graphite (8.8\% ash) and liquid iron at 1600°C. The rate of carbon dissolution from natural graphite was found to be quite slow (~50\% of synthetic graphite dissolution rate) and an ash enriched interfacial layer was observed during FESEM investigations on the interface. This work investigates the role of interfacial reactions and the formation of an ash-enriched layer between two especially prepared coal-chars and liquid iron at 1550°C. The transfer of carbon and sulphur from chars into the metal droplet is also reported.

2. Experimental Details

2.1. Preparation of Coal-chars

Raw coals were crushed in a jaw crusher and vibrating grinder, and wet sieved at 38 \( \mu \text{m} \) and 125 \( \mu \text{m} \) and the resulting size graded samples were dried in an oven prior to char making. The chars were produced from raw coals in a drop-tube furnace in an atmosphere of 23\% O\textsubscript{2} in N\textsubscript{2} at a temperature of 1200°C.\textsuperscript{15} A schematic of the drop tube furnace (DTF) is shown in Fig. 1. The furnace consisted of a water-cooled injector at the top of the furnace, and a collector at the bottom of the furnace. The coal particles were injected into the DTF through a screw feeder at the rate of 0.0167 g/s. In an attempt to simulate rapid heating and temperatures experienced by PCI coal during combustion in the tuyeres, and in the raceway region of the blast furnace, the experimental parameters for the drop tube furnace were carefully chosen to ensure complete devolatilisation and partial combustion. With a typical furnace residence time of 1 s, the heating rate for the coal particles was 10\textsuperscript{5} K/s.\textsuperscript{15} The gas flow rate was 0.02 l/s. Ash contents of coal-chars and their chemical composition is given in Table 1.

![Fig. 1. Schematic diagram of drop tube reactor.](image)

| Char  | Fixed C% | Ash % | Ash composition |
|-------|----------|-------|-----------------|
|       | SiO\textsubscript{2} | AI\textsubscript{2}O\textsubscript{3} | Fe\textsubscript{2}O\textsubscript{3} | TiO\textsubscript{2} | CaO | P\textsubscript{2}O\textsubscript{5} | K\textsubscript{2}O | MgO | SO\textsubscript{2} |
| Char 1 | 79.39 | 10.88 | 53.8 | 25 | 7.31 | 1.32 | 2.47 | 0.80 | 2.98 | 0.88 | 0.90 |
| Char 2 | 84.78 | 9.04 | 41.2 | 37.3 | 6.31 | 2.16 | 4.18 | 1.28 | 1.11 | 1.08 | 2.80 |

2.2. Experimental

Metal/char interactions and associated interfacial phenomena were investigated in a laboratory scale, horizontal tube resistance furnace using the sessile drop approach. The static nature of the droplet helps to preserve the interfacial layer that forms between the iron and the char, allowing a detailed investigation of the interface. Apart from

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Marangoni flow, there is minimal flow within the liquid iron droplet in the sessile drop method. A schematic diagram of the experimental arrangement is shown in Fig. 2. The furnace tube had an inside diameter of 50 mm. The weight of the metal used was \( \sim 0.70 \) g of electrolytic-grade iron (99.98% Fe). The char substrates were prepared by grinding the powdered chars finely (<38 \( \mu \)m). These were then compacted using a hydraulic press without a binder in a steel die and pressing to a pressure of 7.75 MPa. Initially, the metal/char assembly was held on a specimen holder, which could be pushed to the centre of the hot zone in the furnace with help of a graphite rod. The metal/char assembly was held in the cold zone of the furnace until the desired temperature (1550°C) was attained. The assembly was then inserted into the hot zone; this eliminated any reaction that could occur at lower temperatures and possibly influence the phenomena to be studied at the temperature of interest. The melting point of electrolytic iron is approximately 1540°C. A number of specimens of liquid iron were produced with varying contact times with char at 1550°C. Melting of iron marked the beginning of contact time. Once the liquid metal droplet had been exposed to the char for the desired time (1, 3, 5, 7, 10, 15, 30, 60 and 180 min), the tray assembly was pulled back into the colder section of the furnace effectively quenching the droplet of iron, and halting further reaction. The furnace tube was purged with argon throughout the duration of the experiment with a flow rate of 0.5 L/min.

To examine the new phases formed at the interface, the underside of the droplet (which effectively represents the iron/char interface) was inspected using a Field Emission Scanning Electron Microscope (FESEM), and analysed with Energy Dispersive Spectroscopy (EDS). This analysis allowed the time dependent growth of the interfacial material to be determined. After examining the droplets by EDS, the carbon and sulphur contents were determined using a LECO CS444* carbon sulphur analyser.

3. Results and Discussion

3.1. Reaction Products in the Interfacial Region

The interfacial region between liquid iron and two chars was investigated using FESEM and EDS analysis. The results for Char 1 after 5 min of contact with liquid iron are shown in Fig. 3. Aluminium, calcium, sulphur, oxygen and iron were detected in the interfacial layer. The white phase was predominantly alumina. A higher magnification image of interfacial region is shown in Fig. 4, wherein ash deposits and iron can be clearly observed. Small concentrations of magnesium and silicon were also detected indicating the presence of parent ash. While the ash in Char 1 contained 53.8 wt% silica, the silicon peak detected at the interface was very small and could be observed only in some areas at the interface. Figures 5 and 6 show the interfacial region after contacting metal droplets with Char 1 for 3 h at low and high magnifications respectively. The interfacial coverage had increased considerably over time and significantly less iron was measured indicating a further decrease in iron/Char contact area. The EDS spectra (Figs. 5 and 6) showed a large increase in the concentrations of both calcium and sulphur at the interface. Levels of Ca detected in this region were quite high considering the concentration of CaO was only 2.5 wt% of the ash in Char 1. This enrichment appears to be linked with increased sulphur levels at the interface. Char was the only significant source of sulphur in this system, which was picked up by the metal during contact with char. Enrichment of calcium along with the association of calcium and sulphur in the interfacial region suggests that the calcium oxide in the char ash was desulphurising the liquid iron:

\[
\text{CaO} + \text{S} + \text{C} \rightarrow \text{CaS} + \text{CO} \]...............(1)
The desulphurisation reaction\(^{(16)}\) shown in (1) produces calcium sulphide, which was detected on the interfacial region between iron and Char 1.

In Fig. 5 and Fig. 6, the aluminium peaks were much smaller when compared with those observed in Figs. 3 and Figs. 4 after 5 min of contact. The thickness of the interfacial layer showed an increase with time.\(^{(17)}\) With carbon dissolving in liquid iron, alumina present in ash deposits on the interface. However due to non-wetting behaviour between alumina with liquid iron, it has a tendency to stay closer to the char substrate away from liquid iron wherever possible. In non-wetting situations the contact area between iron and alumina is minimised. When the iron droplet was physically removed from the char substrate after quenching, it is quite possible that the alumina deposits were left behind on the char substrate and were therefore not detected.

**Fig. 4.** Left: High magnification image of interface of metal droplet after 5 min contact with Char 1, showing fused ash material and iron surface, ×1 500 magnification. Right: EDS spectra of region indicated.

**Fig. 5.** Left: Interface of metal droplet after 3 h contact with Char 1, showing high interfacial coverage, ×350 magnification. Right: EDS spectra of region indicated.

**Fig. 6.** Left: Interface of metal droplet after 3 h contact with Char 4, showing calcium sulphide complex, and iron surface, ×1 500 magnification. Right: EDS spectra of region indicated.
in the interfacial layer (underside of the iron droplet). It was very difficult to analyse the char side of the interfacial layer as the char was too friable and generally could not be handled after the experiment.

An important result emerging from Fig. 3 to Fig. 6 relates to the detection of very small levels of silicon in the interfacial region. Despite high levels of silica in the char initially (53.8 wt%), very low levels of Si were detected in EDS spectra even after 3 h of contact. This result suggests that silica undergoes chemical reactions and is therefore not present in the interfacial region. During our investigations on the coke/iron system at 1550°C, a large proportion of the silica in the coke was also found to have undergone reactions. Based on thermodynamic considerations, SiO₂ would be reduced by carbon at high temperatures, leading to the evolution of CO and CO₂ gases. This reaction can be seen as a combination of the following basic reactions:

\[ \text{SiO}_2 + \text{C} \rightarrow \text{SiO}(g) + \text{CO}(g) \]  

Silicon monoxide gas can react with solute carbon in the iron droplet:

\[ \text{SiO}(g) + \text{C} \rightarrow \text{Si} + \text{CO}(g) \]  

Silica present in char could either escape the system as SiO₂(g) or could dissolve in liquid iron as silicon. In a recent study on the influence of ash on the interfacial reactions between coke and liquid iron, a significant transfer of silicon into liquid metal was recorded. As the penetration of a 30 kV electron beam is limited to ~2 μm, it was probably unable to probe the deeper regions of the interfacial layer/bulk of iron droplet, and therefore could not detect the presence of silicon in the metal droplet in this study.

Experimental results for Char 2 also showed characteristics similar to those observed for Char 1 (Fig. 7), however some differences in its composition are worth noting. While the total amount of ash in Char 2 was slightly lower, it contained higher levels of alumina and calcia and sulphur. As in the case of Char 1, calcium was found associated with sulphur. Regions rich in Fe and CaS were clearly identified. Further, while the morphology of the CaS phase appeared the same across the two chars, the kinetics of its formation at the interface could be affected by other factors. One such factor may be the initial level of CaO present. A comparison between the two chars after 5 min of contact (Figs. 3, 4 and Fig. 7) showed that Char 2, which has a higher initial CaO level in the ash, was associated with a larger quantity of CaS at the interface (Fig. 8).

### 3.2. Carbon and Sulphur Transfer

Figures 9 and 10 show carbon and sulphur transfers into iron droplets from Char 1 and Char 2 respectively, at 1550°C as a function of contact time. For Char 1, the carbon levels showed a sharp increase reaching a maximum of 0.48 wt% after 5 min of contact followed by a dip and approximately stable levels (~0.10 wt%) for contacts up to 3 h. The corresponding results for Char 2 were slightly different. The carbon levels showed a small peak after ~5 min of contact and then tended to decrease slightly for some time followed by a small increase later on, reaching a maximum of 0.28 wt%. Carbon levels in liquid iron droplet for
both chars were much below the saturation level of ~5.6 wt% at these temperatures. Comparing these results with a sessile drop study between iron and natural graphite,\textsuperscript{14)} the carbon transfer into the iron droplet reached 4 wt% carbon after 1 min of contact. The transfer of sulphur from both chars into the liquid iron also occurred quite rapidly in the initial stages. While the sulphur content in the liquid iron in contact with Char 1 reached a steady level by 30 min (0.16 wt%), the liquid in contact with Char 2 continued to accumulate sulphur for up to 3 h, eventually reaching 0.2 wt%.

Several mechanisms could be responsible for the observed carbon and sulphur transfers from chars into liquid iron. It appears that transfer of carbon from chars into the liquid iron took place in the initial stages of contact and accumulated until some reduction reactions started consuming solute carbon. Reduction reactions of ash oxides such as SiO\textsubscript{2} and Fe\textsubscript{2}O\textsubscript{3} are quite likely at temperatures around 1 550°C.\textsuperscript{19,20)} Iron oxide will reduce rapidly\textsuperscript{21)} under these conditions and is expected to be totally reduced within 1 min of contact. Both chars, however, contain significantly less iron oxide than silica, so the relative impact of iron oxide reduction on liquid metal carbon levels is expected to be less than that of silica reduction. Reduction of silica is likely to consume some of the solute carbon from the iron droplet lowering its carbon content, and could therefore explain the decrease in the solute carbon concentration. While the Si content of metallic droplet was not specifically measured, video images of iron droplet were recorded over a period of time. These images clearly show the evidence for reactions occurring between Char 1 and liquid iron, with bubbles of gas forming within the droplet and visible gas halo surrounding the droplet. With silica in ash consuming some of the carbon in metallic solution, these results are expected to have important implications for the performance of coke and PCI coals on blast furnaces.

Both samples show consistent evidence that the operating transfer mechanisms are complex. Maxima and minima observed in the data over time also provide evidence of changing transfer mechanisms. While the specific mechanisms controlling dissolution are under more rigorous investigation, several important insights are evident. In the initial stage (<5 min) transfer from the carbon substrate is quite rapid. The initial carbon transfer rates for both chars are however much slower than the corresponding rate for natural graphite. These differences in the initial rate could be attributed to the atomic level structural differences between natural graphite and coal-chars. Graphite structure can be described by a regular, vertical stacking of hexagonal aromatic layers with the degree of ordering characterised by the vertical dimension of the crystallite ($L_c$). Coal-chars are only partially crystalline with no long-range order and usually have some carbon atoms arranged in small clusters resembling the graphite structure.\textsuperscript{22,23)} The main difference is in their crystallite size and the degree of ordering. $L_c$ values for natural graphite and two chars were
than the corresponding rate for carbon atoms in natural graphite. Inherent rate of dissociation of carbon atoms from their complex network in coals than that required in graphite. Inherent rate of dissociation of carbon atoms from the host char lattice is therefore expected to be much slower than the corresponding rate for carbon atoms in natural graphite. This transfer of carbon however does not continue unhindered. In an environment of rapidly changing physical and chemical interfacial conditions, the results confirm a growing resistance to carbon transfer to the metal, coincident with the carbothermic reduction of silica. The peak in carbon concentration observed for chars between 3 to 5 min suggests that the two reactions, transfer of carbon and the reduction of silica, do not initiate at the same time. There are a number of possible reasons. It may take some time for silica to form silica monoxide due to small delays in its heating up to the reaction temperature.

Factors affecting the initiation and delay between the dissolution and the onset of reduction reactions require further consideration. When the results shown in Figs. 9 and 10, are combined with evidence derived from analysis of the interfacial products discussed earlier, the effects of physical changes at the interface become more apparent. Results from examining the interface show that the delayed onset of reduction reactions may be a function of the presence and extent of reducible oxides available. Char 2 has a higher concentration of alumina as compared to Char 1. Along with the detection of high levels of alumina in the interfacial region after 5 min of contact, effect of alumina is also reflected in much smaller levels of carbon transferred in case of Char 2 during initial stages. Due to its non-wetting nature, the presence of alumina strongly inhibits dissolution of carbon into liquid iron.

The reduction reactions of silica, due to its high levels in ash, could have significant implications for the dissolution of carbon. The reduction of silica can take place in-situ by the solid carbon in the chars or with solute carbon in liquid iron. In detailed sessile drop investigations on coke/liquid iron system at 1 550°C,18) reduction of ash silica was monitored through an analysis of CO/CO₂ off gases emitted from the furnace. While the measurements of off gases from a blank coke substrate, were used to quantify in-situ reduction of silica by solid carbon in coke, much higher off-gas emissions from the coke/liquid iron substrate pointed towards additional silica reduction by solute carbon. These results were further corroborated with silicon pick-up by liquid iron. Silicon levels in liquid iron droplet increased from 0.02 wt% to 0.3 wt% after 30 min of contact. Although these measurements have not specifically been made for Chars, similar trends are expected. A part of the carbon dissolved from Chars into liquid iron can therefore be removed through silica reduction.

After nearly 3 h of contact, deposition of reaction products such as CaS and reduction of reducible oxide such as silica begin to play an important role. As Char 2 has slightly lower concentration of silica, and if the reduction of these oxides occurred at a similar rate for both chars, Char 2 will have carbon consuming reactions occurring for a shorter time than Char 1. This will result in a shorter period of no net carbon transfer to the liquid iron from the char and could be partly responsible for the slight increase in carbon pick-up observed for Char 2 with time. However, the formation of reaction products/ash at the interface was much more evident in the case of Char 2 when compared with Char 1. After much longer durations (3 h) of contact, examination shows evidence of continuous deposit on the iron/char interface. This would lead to a much-reduced contact between carbon and liquid iron, which could inhibit carbon transfer. The consumption of solute carbon by reducible oxides and the formation of a dense interfacial layer are seen to significantly affect carbon dissolution from these chars.

In the case of natural graphite,14) the system did exhibit the formation of physical interfacial barriers, but the transfer of carbon had already occurred to a great extent. While the natural graphite had a similar weight percentage of silica in the as these chars, along with comparable levels of fixed carbon, the observed carbon transfer rates were much higher during the initial stages of contact. This once-again highlights the differences in carbon dissolution rate between these two types of materials. The non-graphitic carbonaceous material has an intrinsic carbon transfer rate that is much slower than that for natural graphite. If the rate at which carbon dissolves from char into liquid iron is comparable to the rate at which solute carbon is consumed by reducible oxides, the carbon levels in liquid iron would remain far below the saturation limit. In the case of natural graphite, the intrinsic rate of carbon dissolution is quite high15,24) and the consumption of solute carbon by reducible oxides does not therefore significantly affect the carbon concentration in the melt.

In contrast to carbon transfer, the transfer of the sulphur reached a reasonably steady level fairly quickly; at least half of the final sulphur level was transferred in the first minute of contact. It was noted that the sulphur levels transferred in the natural graphite study14) (0.01 wt%) were significantly lower than in the case of chars (~0.2 wt%), despite sulphur concentrations being similar for all three materials. These levels however showed a drop in all cases after an initial peak, which was attributed to the formation of calcium sulphide. An earlier study by Moriss et al.25) had shown that the simultaneous presence of carbon and sulphur in liquid iron resulted in the formation of two immiscible regions one region rich in carbon but low in sulphur, and the second region low in carbon but high in sulphur. Monte Carlo simulations26) on Fe–C–S system also confirmed this result and observed that carbon and sulphur atoms tended to displace one other in liquid iron. Higher levels of carbon in the liquid iron would therefore provide a higher resistance to sulphur transfer into the iron. This could possibly explain the differences in sulphur levels in the iron droplets when reacted with chars and natural graphite. The transfer of sulphur in case of natural graphite takes place into carbon saturated iron, which offers very high resistance to sulphur transfer. On the other hand, the carbon levels in liquid iron are much below the saturation level in case of chars and therefore offer rather low levels of resistance towards sulphur transfer, which could probably explain much higher levels of sulphur transferred in case of chars as compared to natural graphite.
4. Conclusions

The dissolution of carbon from coal chars into liquid iron was seen to be influenced by more than the fusion point of the ash. Reactions between the ash oxides and the liquid iron influenced the level of carbon in the metal. Under sessile conditions, evidence is presented for the influence of both physical and chemical changes occurring at the interface. As the carbothermic reduction of silica with solute carbon resulted in a significantly lowering of the liquid iron carbon content, the silica content of ash was seen as an important factor affecting carbon dissolution from chars. The formation of interfacial products containing calcium and sulphur was also observed during iron/char contact. Knowledge of the ash reactions affecting carbon dissolution will allow better coal selection for PCI applications, with a view to maximising char consumption by blast furnace hot metal.

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