# THE FUNDAMENTAL ASPECTS OF IRON ORE REDUCTION

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RON ore reduction is the conversion of iron oxide minerals to metallic iron. The chemical reactions involved take place in the blast furnace during the production of hot metal or in the several proposed solid-state processes that produce sponge iron. Because there are three oxides of iron, hematite, magnetite and wustite, and be cause both carbon and hydrogen are used as reducing agents, the fundamental aspects of the reduction process are rather complex. It is therefore important that these fundamentals be thoroughly understood in the development of a new process if success is to be achieved.

For many years the blast furnace process has been successfully used for the production of iron. However, this process is dependent upon the use of high quality metallurgical coke. Those countries in which the iron and steel industry is most developed are those which have had highly adequate reserves of coking coals. Where these reserves are approaching depletion and in those countries which do not have coking coals, great interest is directed toward the development of iron ore reduction processes which are independent of metallurgical coke. Sponge iron or solid-state processes of which there are several possibilities, comprise the majority of those deserving of close investigation. It is essential to realize that, in any

of the proposed methods, the cost of iron production must be such that the process is, at least, reasonably competitive with the blast furnace.

One of the notable features of the blast furnace is its high throughput rate. Even a small furnace produces in excess of 1000 tons of iron per day while many produce over 4000 tons. Much of this achievement is due to the high temperature at which the furnace is operated resulting in an iron product in the liquid state. In contrast, solidstate processes must operate at much lower temperatures which is a significant disadvantage. No sponge iron furnace of comparable size to a blast furnace can produce metal at the same high rate. Thus the kinetics of iron ore reduction is an important fundamental aspect.

The sponge iron processes differ from each other in the type of furnace used which may be a rotary kiln, shaft furnace or fluidized bed reactor. They also differ in the type of fuels used. These can be solid, liquid or gaseous. In countries which have abundant reserves of non-coking coals, the use of solid fuels for heating and reduction is particularly attractive. However fuel oils and gases have several advantages over coal not the least of which is the presence of sulphur in many coals which is particularly troublesome. Thus it can be seen that the fundamental aspects of iron ore reduction include not only the chemical reactions involved, their equilibria and kinetics but also the consideration of the reaction temperatures, the heat requirements, heat transfer problems and the limitations imposed by the type of furnace and fuel used. In developing a new process, it is always worthwhile to critically compare its anticipated performance with that of the blast furnace with which it must compete. Finally the sulphur problem which confronts all iron ore reduction processes must not be overlooked and a satisfactory solution must be found.

#### **IRON OXIDES**

Iron has three oxides, ferric oxide or hematite ( $Fe_2O_3$ ), magnetite ( $Fe_3O_4$ ) and ferrous oxide or wustite FexO, where x has a value that can vary between about 0.85 and 0.95). Both hematite and magnetite exist in nature and are the chief minerals occurring in iron ores. Wustite does not exist in nature as it is unstable below 570°C (843°K). Its variable composition is due to 5 to 11 per cent vacancies in the iron lattice of the crystal. Electrical neutrality is maintained by the formation of two trivalent iron ions for every vacant lattice site. For convenience the formula for wustite is given as FeO although this does not correctly describe the compound.

# IRON OXIDE REDUCTION REACTIONS

The chemical reactions involved in iron ore reduction processes are shown in Table I. They are numbered for easy reference. Reaction 1 occurs at the tuyeres of the blast furnace where carbon is oxidized to carbon dioxide by reaction with the oxygen in the air blast. At the high temperatures involved, the carbon dioxide, so formed, is converted to carbon monoxide according to Reaction 2. This is the well-known Boudouard Reaction. Reaction 2 can also take place between carbon and carbon dioxide produced by Reactions 5, 6 and 7, in which case, it is generally referred to as the Solution Loss Reaction. Reaction 3 is the sum of Reactions 1 and 2:

> $C + O_2 = CO_2 \qquad \triangle H_{1400} = -94,510$  $C + CO_2 = 2CO \qquad \triangle H_{1400} = +39.750$

Adding:  $2C + O_2 = 2CO riangle H_{1403} = -54,760$ 

Reaction 4 takes place between carbon and water vapour from the combustion air or moisture from the furnace charge materials.

The reduction of the iron oxides by carbon monoxide takes place in three stages as follows:

I II III  
F
$$e_2O_3 \longrightarrow Fe_3O_4 \longrightarrow FeO \longrightarrow Fe$$

The reactions involved are numbers 5, 6 and 7 in Table I. It is to be noted that these three are of the gas-solid type. That is one reactant is a gas (carbon monoxide) and the other is a solid (the oxides). In the blast furnace, the iron oxides are usually completely reduced before the melting of the iron begins and, of course, in the sponge iron processes there should be no melting of the metal. By long usage it is usual to refer to Reactions 5, 6 and 7 as the Indirect Reduction by Carbon since solid carbon takes no part in the reactions.

Reaction 8 is merely a combination of Reactions 5, 6 and 7 to show the combined heat effect. Reaction 9 shows the Direct Reduction by Carbon. However, in general, this reaction does not take place, as shown, to any great extent. More correctly it is the combination of Reactions 2 and 7. The mechanism can be illustrated as follows:

$FeO + CO = Fe + CO_2$	$\triangle H_{1400} = -4,230$
$C + CO_2 = 2CO$	$\triangle H_{1400} = +39,750$

Adding: FeO + C = Fe + Co  $H_{1400} = +35,520$ 

This takes place at temperatures in excess of about 1000°C (1400°K) where the carbon dioxide product from the first reaction reacts vigourously with carbon to regenerate carbon monoxide in the second. It is to be noted that Reaction 9 is strongly endothermic, a point of great significance in iron oxide reduction.

Reactions 10, 11 and 12 show the stepwise reduction of the iron oxides by hydrogen, while Reaction 13 is the combination of these.

615	000
G	3
-	1
P	9
F	
c	- ·

IRON OXIDE REDUCTION REACTIONS

Reac- tion No.	Equation for Reaction	Hcat of Re Cal./molo 1000°K	action, △H° e C or H <sub>3</sub> 1400°K	Free Energy Cal/mo Cal/mo 1000°K	of Reaction, ∆G° le C or H₁ 1400°K	CO/CO, ol Ra 1000°K	r H,/H,O tio 1400°K
H	C + 0, = CO,	94,320		94,610		1	1
7	C + C0₂ ☴ 2CO	+40,780	+39,750	- 1,270		2.610	в
ŝ	$C + \frac{1}{2}O_{2} \parallel CO$		-27,380	47,940		1	ļ
4	$C + H_a O = CO + H_a$	+ 32,440	+32,360	— 1,910		1	.1
\$	$3Fe_{a}O_{3} + CO = 2Fe_{3}O_{4} + CO_{a}$	-11,250	-11,630	-22,770		1.05×10 <sup>-5</sup>	5.71x10 <sup>-6</sup>
9	$Fe_{3}O_{4} + CO = 3FeO + CO$ ,	+ 3,750	+ 4,270	- 420	- 2,280	0.809	0.440
7	$FeO + CO = Fe + CO_{3}$	— 4,750	- 4,230	+ 880	+ 3,020	1.557	2.961
8	$Fe_{s}O_{s} + 3CO = 2Fe + 3CO_{s}$	- 3,583	- 3,16	I	1	1	1
6	FeO + C = Fe + CO	+36,030	+ 35,520	390	-14,910	1	1.
10	$3Fe_{3}O_{3} + H_{2} = 2Fe_{3}O_{4} + H_{3}O_{3}$	— 2,910		-22,130	29,440	1.46x10 <sup>-5</sup>	2.53x10 <sup>-5</sup>
Ξ	$Fe_{3}O_{4} + H_{3} = 3FeO + H_{3}O_{3}O_{3}O_{3}O_{3}O_{3}O_{3}O_{3}O$	+12,090	+ 11,660	+ 220	+ 4,540	1.117	0.195
12	$FeO + H_a = Fe + H_aO$	+ 3,590	+ 3,160	+ 1,520	+ 760	2.149	1.314
13	$Fe_sO_s + 3H_s = Fe + 3H_sO$	+ 4,760	+ 4,227	1	1	ŀ	

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Table I also shows the Heats  $(\triangle H^\circ)$  and Free Energies  $(\triangle G^\circ)$  of Reaction for each reaction. These were calculated from the standard Enthalpies and Free Energies of Formation for all the oxides concerned. These standard data were obtained from the work of Elliott and Gleiser (1) and are shown in Table II.

The heats of reaction were calculated at 1000 and 1400°K (about 700 and 1100°C) because, as will be shown, these are critical 'temperatures in sponge iron processes. In keeping with the accepted nomenclature, negative heats of reaction indicate a decrease in the heat content of the system, hence, an exothermic reaction while positive enthalpies indicate endothermic reactions. It will be noted that seven of the thirteen reactions in Table I are endothermic of which Reactions 2, 4 and 9 are highly so.

The Free Energies of Reaction were calculated and recorded in Table I in order to calculate the CO/CO<sub>2</sub> and the  $H_2/H_2O$  equilibrium ratios for each reaction. By means of the following equation:

# $\triangle G^{\circ} = -4.575 \text{ T log K}$

the equilibrium constant, K, was calculated. In turn, K equals  $(CO_2)/(CO)$  or  $(H_2O)/(H_2)$  in all reactions except No. 2 where K equals  $(CO)^2/(CO_2)$ . The CO/CO<sub>2</sub> and the  $H_2/H_2O$  ratios, the reciprocals of K, were then computed. No free energies nor equilibrium ratios were calculated for Reactions 8 and 13 since these are merely the combination of other reactions and no equilibria in the true sense exist.

From the equilibrium gas ratios at various temperatures it is possible to construct equilibrium phase diagrams for the systems Fe-C-O and Fe-H-O. These are shown in Fig. 1 and 2 respectively. The curves divide the diagram into areas and each area shows the solid phase which is stable within its boundaries. Along each curve, the two solid phases from the adjacent areas are in equilibrium with each other and the gas phase at the temperature indicated on the abscissa. The composition of the gas phase is shown on the ordinate.

As previously noted, wustite is unstable at temperatures below 570°C. In this region, the equilibrium between iron and magnetite is described by the reaction:

 $Fe_3O_4 + 4CO = 3Fe + 4CO_2$  (14)

and the corresponding reaction for hydrogen and water vapour.

The equilibrium curve for the Boudouard reaction (reaction 2) is also included in Fig. 1. Carbon dioxide is the stable gas to left of this curve and carbon monoxide to the right. Thus at low temperatures, carbon monoxide tends to decompose into carbon and carbon dioxide. This process is referred to as Carbon Deposition. This reaction is somewhat inhibited because it is required that the carbon must first nucleate and grow on some suitable catalytic surface. However, partially reduced iron ore particles do provide such a surface so that the reducing gas can loose its reducing power. Thus a study of the equilibrium diagram of Figure 1 will show that magnetite cannot be reduced to wustite at temperatures below about 700°C (973°K) nor can wustite be reduced to iron at temperatures below about 750°C (1023°K) by carbon monoxide. This minimum reduction temperatures does not apply to iron oxide reduction by hydrogen.

#### TABLE II

Standard	Enthalpies	and	free	energies	of	Formation	for	Oxides

Oxide	Enthalpy	of Formation free Ener	rgy of Formation	
5.	1000°K	1400°K	1000°K	1400°K
Fe <sub>2</sub> O <sub>3</sub>	-191,900	-191,900	-133,900	-110,600
Fe <sub>3</sub> O <sup>4</sup>	-259,700	-240,100	-188,900	-160,300
FeO	- 62,800	- 62,900	- 47,550	- 41,400
CO2	- 94,320	- 94,510	- 94,610	- 94,690
СО	- 26,770	- 27,380	- 47,940	- 56,310
H₂O	- 59,210	- 59,740	- 46,030	- 40,640



FIG.1 Fe-C-O Equilibrium Diagram



# TEMPERATURE LIMITATIONS IN SOLID – STATE REDUCTION

When iron ores are to be reduced in the solid state to produce sponge iron there is also maximum limit below which the furnace temperature must be maintained in order to prevent melting. sintering and sticking of the charge materials. There is an iron-carbon eutectic at 4.3 per cent carbon which melts at 1130°C (1403°K). However there is very little likelihood that the reduced iron will absorb this large amount of carbon at

the temperatures and in the time concerned. More serious are the effects of the gangue constituents in the ore. Silica forms an eutectic with wustite (25% SiO<sub>2</sub>, 75% FeO) which melts at 1177°C (1450°K). Also in the presence of lime there is an eutectic (12% CaO, 43% SiO2, 45% FeO) with a liquidus temperature, at 1105°C (1378°K). If operated above this temperature, fusion and sinfering can occur which can seriously impede the movement of the ore through the furnace. This applies particularly to rotary kilns when using fine materials or ores which decrepitate to dust readily. A ring forms around the circumference of the kiln in the hot zone which is extremely troublesome. Difficulties can also develop in fluidized bed reactors for the same reasons causing the bed to become defluidized. Shaft furnaces are less likely to have problems in this respect because the weight of charge above tends to keep the whole mass moving. However, it can be seen that solid-state process using carbon or carbon monoxide as the reductant are limited to a useful temperature range of about 700 to 1100°C (1000 to 1400°K).

Controlling the furnace temperature within the narrow range of 400 degree may be readily achieved in laboratory furnaces or even small pilot-plant furnace but it becomes increasingly more difficult and expensive as the size of the plant unit is increased. In designing a large commercial furnace by scaling up laboratory or pilotplant data this point must not be overlooked. When handling large masses of material in large furnaces temperature gradients throughout the mass can be large particularly if the mass is heated from the outside and endothermic reactions take place on the inside.

#### HEAT REQUIREMENTS FOR REDUCTION

Heat is required in the production of iron to heat the charge materials to the reaction temperature and to supply the necessary energy for endothermic reactions. This heat is furnished from the combustion of the fuel and from exothermic reactions. An examination of the heats of reaction in Table I (Reactions 5 to 13) shows that there is very little heat to be expected from the latter. Most of the heat must come from the fuel combustion. It is in this respect that the blast furnace has a big advantage over other processes. Carbon is burned by preheated air at the furnace tuyeres producing carbon monoxide at a very high temperature. The sensible heat in the gaseous combustion products is transferred to the charge materials very efficiently by countercurrent flow through the furnace. The reductant, carbon monoxide, is produced in excess and reduction takes place largely exothermically through indirect reduction according to Reaction 8. Direct reduction or solution loss varies to some extent depending on the nature of the ore but is relatively unimportant. As far as the reduction part of the process is concerned, the blast furnace may be considered to be a process that uses a gaseous reductant. Thus the heat requirements in the blast furnace process are largely for preheating the charge and for melting the reduced iron and slag.

By contrast, the heat requirements of the sponge iron processes can be quite different. In the first place no heat is required to melt the iron and gangue materials but the charge must be heated to and maintained at the maximum tem-(about 1100°C or 1400°K). perature permissible (Because of the slow rate of reaction, any temperature below the maximum allowed is hardly tolerable) Secondly, on the other hand, the heat requirements for endothermic reactions can amount to a major item. This applies particularly to those processes where solid fuels are used as the reductant and the carbon monoxide required is obtain. ed mainly through the Solution Loss reaction (Reaction 2). This is a major problem for rotarykiln methods where, by its very nature, the solid phases and the gaseous phase are almost completely separate. In order to intermix the reductant with the ore, it too must be a solid fuel. This point has been emphasized elsewhere (2) This means that reduction is almost entirely direct as in Reaction 9 and all the heat required must be transferred from the zone of combustion in the gas phase to the zone of reaction by conduction through the solid phases. This is a problem of heat transfer.

#### HEAT TRANSFER

The importance of heat transfer has already been touched on in the preceeding section. Again the blast furnace shows that most successful transfer of heat takes place from the gaseous combustion products to the solid charge ingredients through a process of intimate mixing and countercurrent flow. This subject has received thorough investigation and is well understood.

heat across the interface between the two phases and the flux is proportional to the difference in temperature. In the blast furnace, the high combustion gas temperature promotes the exchange. Again the sponge iron processes are limited by the maximum temperature permissible and this point should be carefully considered in the process design.

Shaft-furnace processes involving the countercurrent principle between solids and reducing gases have the greatest promise of success provided that the gas flow is evenly distributed across the cross-section of the shaft and channelling prevented. This is best achieved by the use of a charge of uniformly sized particles free from fines.

Fluid-bed reactors provide the best method to intimately mix solids with gases but the counter-current principle is missing unless the reactor consists of a series of superimposed hearths in which the fluidized solids flow from one to the next below in the opposite direction to the gas flow. Of the three types of furnaces, the rotary kiln is by far the poorest from the heat transfer viewpoint.

When treating finely ground materials such as magnetic or flotation concentrates which must be pelletized before reduction, it is well to take advantage of the sintering (sometimes called indurating) process to help solve the heat transfer problem. Too often it is attempted to sinter and reduce pellets in the same furnace to save both capital and operating costs. This is a big mistake. For sintering, an operating temperature of 1200 to 1300°C (1500-1600°K) is needed to produce a hard, strong, non-spalling well indurated pellet whereas the reduction temperature must be kept below 1100°C as already explained. If the pellets are sintered in one furnace and then transferred without cooling to the reduction furnace the heat transfer problem is eliminated. In fact the overheated pellets will promote the endothermic reduction reactions and will be cooled thereby in the process.

### **REACTION RATES**

Success in any sponge iron process also depends upon a rapid rate of reaction in order that the maximum production for a given furnace unit Heat transfer takes place by the diffusion of will be obtained. The reaction rate is controlled

mainly by a property of the ore generally referred to as the reducibility. This depends a great deal upon such factors as particle size and distribution, hardness, density, porosity and chemical composition. In preparing ores for reduction, much can be done to improve the reducibility. This subject has been dealt with thoroughly elsewhere <sup>3, 4, 5</sup>. Only a few of the major points are discussed here.

Reaction rates increase with reaction temperature. Therefore it is essential to keep the operating temperature as close as possible to the maximum permitted in the process. Again the sponge iron processes are at a disadvantage in that they are limited to a maximum of 1100°C. The only solution here is to take particular care in the ore preparation in order to insure that it has the best possible reducibility.

The porosity of iron ore particles is one of the most important factors in their reducibility. This has been well demonstrated by the work of Joseph (6). When a particle of a hard dense partially-reduced ore is sectioned in half, a topochemical type of reduction is observed. This is illustrated in Figure 3 which shows a core of hematite surrounded by three concentric layers, an inner layer of magnetite, a layer of wustite and an outer layer of metallic iron. For very porous ores, a more diffuse type of reduction is observed with no distinct interfaces but a gradual transformation from iron on the outside to hematite at the center. This is illustrated in Figure 4. However, at very high magnifications, the microstructure of the grains making up the porous particle also show a topochemical type of reduction.

The reduction reactions must take place at the interfaces between the oxides and metallic phases. Several different mechanisms have been proposed. For hard dense ores, that suggested by Edstrom (7) is the most widely accepted. This mechanism usually produces a topochemical pattern of reduction and it is claimed that oxygen is removed from the iron-wustite interface only according to Reactions 7 or 12. The other oxides are reduced to the next lower oxide by the diffusion inwards of iron ions and electrons according to the following reactions:

$$Fe_3O_2 + Fe + 2e = 4FeO$$
(15)

 $4Fe_3O_3 + Fe + 2e = 3Fe_3O_4$  (1)









The mechanism requires that the reducing gas, carbon monoxide or hydrogen, diffuse inwards through the iron layer to the iron-wustite interface and the product gas, carbon dioxide or water vapour, diffuse outwards.

A second mechanism is applicable to very porous ores through which the reducing gases can penetrate faster than they can react at any one interface. In this case the reduction takes place according to Reaction 5, 6 and 7 as expected and a reduction pattern as shown in Figure 4 results.

Both of these mechanisms require that the reducing and product gases must be able to diffuse at least through the iron layer. In addition the concentration of the reducing gas must exceed that of the product gas to the extent that their ratio is greater than the equilibrium ratio as shown in Table I. For this to occur the flow rate of the gases through a bed of solid particles, whether fixed or moving, must be large enough such that a stagnant layer of gas cannot build up around each particle. The ideal conditions are most nearly approached in the fluidized-bed reactor, where each individual particle is supported by an upward current of gas. The shaft furnace fulfils this requirement moderately well provided that the gas (16) flow rate is sufficiently rapid and that it can

the rotary kiln, where the solid and gas phases are separated, the atmosphere is almost completely stagnant except for whatever reducing gas can be provided by the Boudouard reaction (Reaction 2).

#### SULPHUR PROBLEM

In the blast-furnace process, the sulphur problem has been largely overcome by the adsorption of sulphur in the slag. The desulphurizing power of basic slags has been the subject of much research and is now well understood. The work of Professor Chipman (8) and his colleagues is particularly notable in this field. The requirements for good desulphurization are as follows:

- (1) A highly basic slag, rich in lime and magnesia,
- (2) A strongly reducing atmosphere,
- (3) A high temperature,
- (4) Sufficient time allowance for the transfer of sulphur from metal to slag and
- (5) The complete separation of metal and slag.

These requirements are only partially fulfilled, if at all, in the sponge iron processes. In the first place there is no separation of slag and metal. Some processes overcome the problem to some degree by the addition of lump limestone to their furnace charge. The lime is then separated later from the metal by magnetic means. However the transfer of sulphur from one solid particle to an other in loose contact can never be as efficient as from one liquid to another.

The source of sulphur in ironmaking comes primarily from the solid fuels used. Sulphur in the ore, fuel oil or gaseous fuels can largely be eliminated by some preliminary treatment before reduction but the sulphur in coal presents a Problem for which there is as yet no satisfactory practical solution. Unlike the studies on slags, there have been no expensive investigations on the transfer of sulphur between solid phases. Much work in this field remains to be done.

# SUMMARY AND CONCLUSIONS

In the development of a process for the production of sponge iron, critical comparisons with the established blast furnace process can be extremely helpful since the blast furnace still remains the best process for iron production in many respects. The blast furnace is dependent upon the use of high quality metallurgical coke and other methods, particularly sponge iron methods, look particularly attractive in countries where coking coals are unavailable and where non-coking coals and other fuels are more readily available.

Sponge iron processes have been designed for shaft furnaces, fluidized bed reactors and rotary kilns using either solid, liquid or gaseous fuels.

The chemical reactions involved in iron oxide reduction are complex because there are three different iron oxides involved. The reduction takes place through reactions of the gassolid type. Solid and liquid fuels must first be converted to gas. Some of the reduction reactions are exothermic while others are endothermic. The process should be designed to avoid the latter if possible.

The concentration of reducing and product gases in the gas phase must be such that the reducing gas always exceeds the equilibrium the calculated concentration as shown by CO/CO2 or H2/H2O ratio. A study of the equilibrium Fe-C-O phase diagrams shows that iron oxides cannot be efficiently reduced for this reason below about 700°C while melting, sintering and sticking of charge materials limits the maximum temperature to about 1100°C. In large furnaces treating large masses of material it is extremely difficult to maintain the operating temperature between these narrow limits.

The heat requirements of the process are for heating the charge to the reaction temperature and for carrying out endothermic reactions. The latter should be avoided if at all possible. Heating the charge is best achieved by a counter current process in which the sensible heat in the flowing reducing gas is transferred to the solids moving in the opposite direction particularly, this is what happens in the blast furnace.

Sponge iron processes using shaft furnaces and preheated reducing gases most nearly approach ideal heat-transfer the conditions. Fluidized bed reactors make good gas-solid contact but the counter current flow principle is lacking except in reactors of the multi-hearth type. Rotary kilns are the poorest type of furnace from the viewpoint of efficient gas-solid heat exchange because the gas and solid phases are essentially separated.

Where fine ores and concentrates require to be agglomerated before reduction, it is a mistake to attempt to sinter and reduce the ore in the same furnace. A high temperature is needed to make a well indurated pellet and it is much better to sinter the ore in one furnace and to transfer it without cooling to a second furnace which operates at a lower temperature. In addition, the problem of heat transfer to heat the ore to the reduction temperatures is largely overcome.

Because sponge iron furnaces must operate at lower temperatures than the blast furnace, the reduction reaction rates become much more critical. It is important to prepare the ore before reduction to provide the optimum reducibility. In addition it is essential that no stagnant layer of gas builds up around each ore particle. This requires a rapid flow rate of reducing gas through the bed of particles. The fluidized-bed reactor approaches most nearly the ideal conditions in this respect followed by the shaft furnace. The rotary kiln is the poorest from this viewpoint.

Sulphur presents a serious problem in any iron reduction process. The blast-furnace process provides the most satisfactory answer through the use of basic slags of high desulphurizing power. In sponge-iron processes, in which no separation is made between metal and gangue constituents, the solution is much more difficult. The obvious solution, is to pretreat the raw materials to remove the sulphur before reduction. This is a practical solution for the ores and gaseous fuels but for solid fuels there is as yet no satisfactory answer.

#### ACKNOWLEDGEMENT

The author gratefully acknowledges the assistance of Dr. S. V. Muthuswami in checking the thermodynamic calculations.

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