

# Overview of thermodynamics concepts in production of some ferroalloys (ferrochrome, ferromanganese, ferrotitanium and ferrovanadium)

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Ferrochromium (FeCr) and ferromanganese (FeMn) alloys are produced commercially by reduction smelting of their oxide minerals with carbonaceous reductant in a submerged arc furnace. Whereas ferrotitanium can be produced from titania slag obtained from vanadium-titanium magnetite with over 40 wt% TiO<sub>2</sub> through aluminothermic reduction. To produce FeV, first vanadium is primarily recovered from titanium-bearing magnetite ore processed to produce pig iron. The process produces a slag containing 20–24% vanadium pentoxide, which is further processed to ferrovanadium containing 40–50% vanadium. Most ferrovanadium is produced by aluminothermic reduction of such slags. The overview of thermodynamic concepts underlying reduction smelting of various oxide minerals and other processes are discussed with the help of free energy–temperature (Ellingham) diagram with a focus on the role of direct reduction, the smelting temperatures required for a given oxide mineral, the thermal energy requirements, the suitability of carbon as a reductant, the slag–metal equilibria, and the formation of carbides. The purpose of this work is to underline the importance of principle of thermodynamics and thermodynamic data in educating the concepts of pyrometallurgy of production of ferroalloys.

## INTRODUCTION

Ferrochrome and ferromanganese alloys are produced by reduction smelting of their oxide minerals with carbonaceous reductants like coke or charcoal in submerged arc furnaces and sometimes by metallic reductants like aluminium. Generally overall reduction requires large thermal energy as reactions are highly endothermic. With the help of an Ellingham diagram (Figure 1) the thermodynamic principles underlying reduction smelting are discussed. The Ellingham diagram shows standard Gibbs energy of formation of various oxides as a function of temperature, with respect to 1 mol of oxygen gas.

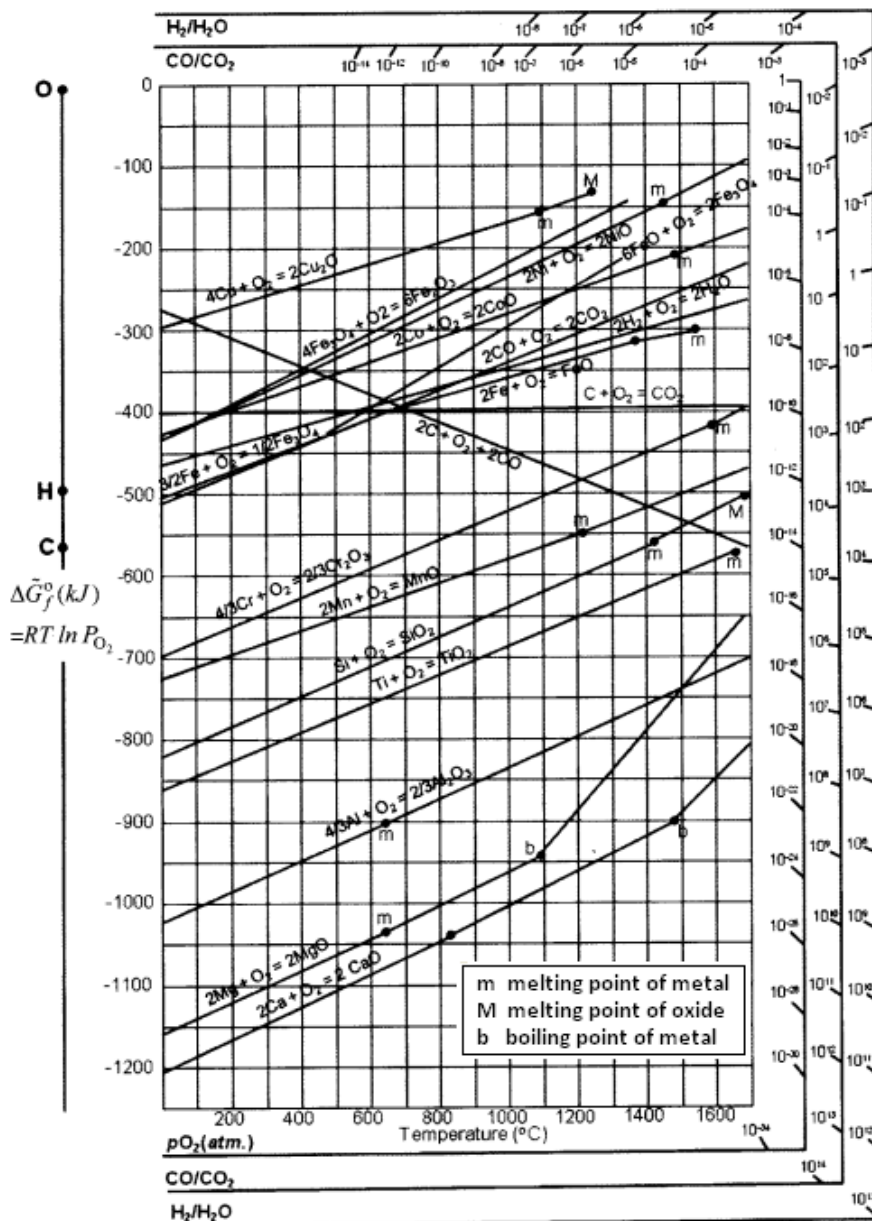


Figure 1. Standard Gibbs energies of formation of metal oxides as a function of temperature [3].

From Figure 1 of the Ellingham diagram it can be noted that oxides appear in the following order: FeO, Cr<sub>2</sub>O<sub>3</sub>, MnO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO, and CaO where more stable oxide appears in the lower part of the diagram. Thus, if conditions are made favourable for the reduction of MnO, the oxides of iron, phosphorus, and chromium will also be reduced. It is also noted that for any given oxide mineral in the Ellingham diagram those oxides which are below are more stable and hence elements like Al, Si act as reducing agent. This principle is utilised in the metallothermic reduction processes.

#### Direct and indirect reduction using carbon-based reductants

MO+C=M+CO direct reduction

MO+CO=M+CO<sub>2</sub> indirect reduction

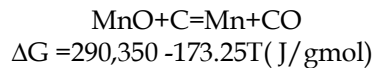
Of these reactions, the indirect reduction is more exothermic[3] compared to direct reduction. So if CO formed in direct reduction is used for subsequent indirect reduction then energy efficiency of a process

could be significantly enhanced. Here solid-state reduction of the furnace is pursued, especially in ferromanganese smelting.

### Smelting temperature requirements

Temperature requirements are such that the final products are molten alloy and slag. The alloy composition should be within the specified/desired limits and their temperatures should be high enough (~100°C above the liquidus) to impart adequate fluidity for good separation.

In the case of ferromanganese, the equilibrium pressure of CO produced from the reaction below reaches 1 atm at about 1400°C (corresponding to the intersection of MnO and CO lines on the Ellingham diagram). This generally happens in the furnace coke bed when rich MnO slag contacts coke at high temperatures.



### Thermal energy requirement

Ferroalloy production is an energy intensive operation due to endothermic reduction reactions as shown below:

Enthalpy change $\Delta H_{298}$		
	kJ/g mol	kWh/kg-metal
$\text{MnO}_2 + 2\text{C} = \text{Mn} + 2\text{CO}$	300.0	1.52
$\text{Cr}_2\text{O}_3 + 3\text{C} = 2\text{Cr} + 3\text{CO}$	803.1	2.15

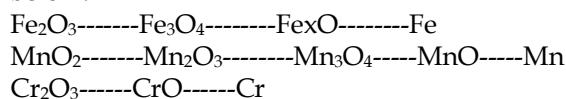
Also in order for clean separation, additional heat must be supplied for melting metal and slag to raise their temperature. Heat losses in surrounding and cooling water must also be taken into account. Taking all of them together, heat requirement increases significantly; for manganese, it was estimated to be 2.45 kWh/kg [3]. Due to such a large thermal requirement ferroalloys are produced in a submerged arc furnace, as arc provides the necessary heat and also maintains the reaction zone at a high temperature. The final state of the metal-slag bath is governed by the hearth temperature, which should be reasonably high to effect metal-slag separation. The slag produced in ferroalloy process usually contains appreciable amounts of the valuable metal as shown below.

Alloy	Mn	Cr	C	Si
HCFeCr	-	65.5	7.0	4.5
MCFeMn	72	-	2.0	0.8

Alloy	Slag Composition						Slag-Metal Ratio
	MnO	Cr <sub>2</sub> O <sub>3</sub>	CaO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	
HC FeCr	-	6.2	-	37.2	30.6	26.0	0.83
MCFeMn	14	-	38	5.0	28.0	3.0	

### Slag metal equilibria in carbothermic smelting

The reduction of oxide minerals of iron, manganese, and chromium takes place in stages as shown below:



The reduction of Mn ores up to the stage of Mn<sub>3</sub>O<sub>4</sub> occurs by thermal dissociation and indirect reduction (like that of iron ores to Fe<sub>3</sub>O<sub>4</sub>) and thereafter, by direct reduction. Direct reduction occurs for production of manganese and chrome metals. Indirect reduction with CO can produce Fe. However, a

lot of research has been done to develop solid-state reduction of Mn and Cr whose separation with slag could be done through melting.

Thermodynamic equilibrium is approached at the last stage of direct reduction of oxides for slag-metal phases as follows:

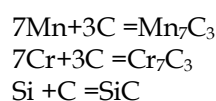


where parentheses for MO indicate the slag phase and brackets for C and M indicate the liquid alloy phase. If M is Mn, the main mechanism will revolve around the activity of the MnO in the slag and C of the coke bed. In order to promote reaction in a desired direction the knowledge of equilibrium constant K, through standard Gibbs energy data, is required for which extensive thermodynamics studies on oxide slags and liquid iron solutions are quite useful. Also the activities of various components can be estimated and used to access the extent of equilibrium achieved in a given process.

Several investigators with the help of phase rule and Gibbs energy minimising technique have analysed multiphase equilibria (Factstage and HSC) such as those pertaining to ferroalloy processes in order to predict distribution of a metal between slag and alloy phase for different charge composition and operating conditions of temperature and pressure.

### Carbide formation

Coke-carbon are economically preferred reductants (in bulk ferroalloys, the preferred reductants are coke and anthracite coal). Dissolution of carbon from coke results in the formation of carbides, ferroalloy thus produced contains high levels of carbon-promoting formation (precipitation) of metal carbides such as:



These carbides are refractory materials and the low temperature at the hearth bottom is likely to help precipitate  $\text{Cr}_7\text{C}_3$  and  $\text{SiC}$ , which may cause furnace bottom build up.

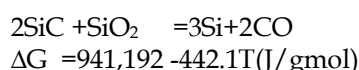
At smelting temperature for manganese and chromium, the first carbides to be precipitated are  $\text{Mn}_7\text{C}_3$  or  $\text{Cr}_7\text{C}_3$ . However equilibrium carbide phase after solidification is mostly  $\text{M}_{23}\text{C}_6$ .

From a thermodynamic viewpoint, it should be possible to reduce these carbides and recover the metal into the alloy by reacting the former with metal oxide at relatively high temperatures, as shown below:



For a molten bath saturated with  $\text{Mn}_7\text{C}_3$  ( $a_{\text{Mn}_7\text{C}_3} = 1$ ) and in contact with solid MnO, with  $P_{\text{CO}} = 1$  atm. and taking  $a_{\text{Mn}} = 0.3$  for a 75% Mn in ferromanganese alloys, one finds that the above reaction becomes feasible (negative Gibbs energy) at about 1400°C. Although, practically this has been tried for production of low C FeMn but it resulted in poor efficiency.

Similarly, for example, for



using  $a_{\text{SiC}} = 1$ ,  $a_{\text{SiO}_2} = 1$ ,  $P_{\text{CO}} = 1$  atm, and  $a_{\text{Si}} = 0.82$  for a 75% Si in ferrosilicon, it is found that  $\Delta G$  equals zero at 1830°C. Thus, at temperatures above 1830°C, the above reaction will become feasible, promoting dissolution of silicon carbide into the Fe-Si alloy.

## Thermodynamics involved in ferrochrome production

Four grades of ferrochromium are commercially produced, characterised in terms of their carbon and chromium contents

- (Cr: >60%, C: 6-9%) ▼ High-carbon ferrochromium(HCFeCr)
- (Cr: 50-60%, C: 6-9%) ▼ Charge chrome
- (Cr: 56-70%, C: 1-4%) ▶ Medium-carbon ferrochromium(MCFeCr)
- (Cr 56-70%, C: 0.015-1.0%) Low-carbon ferrochromium(LCFeCr)

Approximately 80% of world production of ferrochromium is used in stainless steel making. Demand for LC ferrochromium has decreased due to the commercial development of Argon-Oxygen-Decarburisation (AOD) and Vacuum-Oxygen-Decarburisation (VOD) processes, which allow removal of carbon from stainless steel with acceptable loss (oxidation) of chromium. Hence, high carbon ferrochromium is now the most widely produced and consumed grade of chromium containing ferroalloys [2]. Production of high carbon ferrochromium is based on reduction smelting of chromite ore with coke in the presence of flux in a submerged arc furnace and sometime DC furnaces.

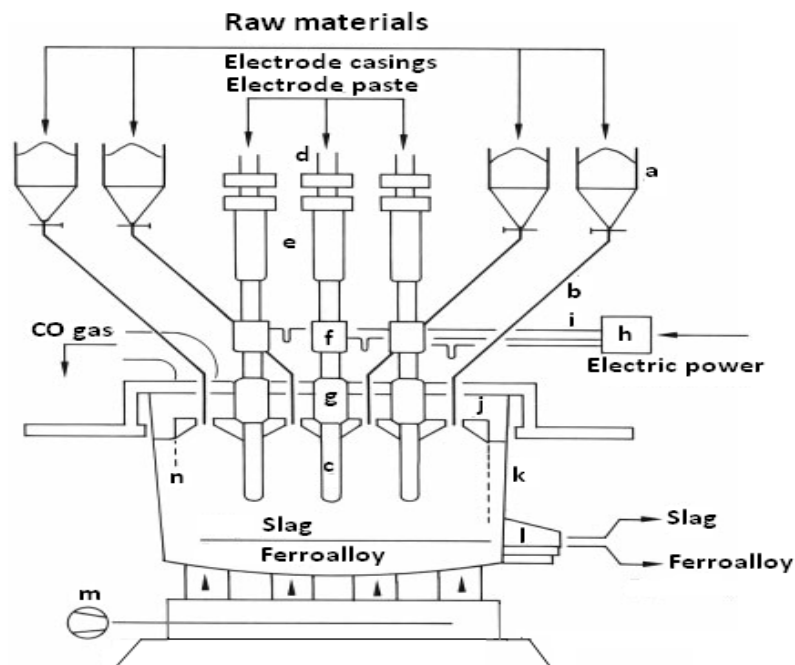


Figure 2. Layout of an electric arc furnace. (a) charging bins, (b) charging tubes, (c) electrodes, (d) electrode slipping device, (e) electrode-positioning devices, (f) current transmission to electrodes, (g) electrode scaling, (h) furnace transformer, (i) current bus bar system, (j) furnace cover, (k) furnace shell, (l) tap hole, (m) furnace bottom cooling, and (n) refractory material [3].

## Submerged arc furnace

Production of ferrochromium is done by conventional smelting process in electric reduction furnace (Figure 2) having Soderberg electrode submerged in the burden [1,3].

## Thermodynamic considerations

Most of the reduction reactions taking place in the submerged arc furnace are highly endothermic and are listed in Table 1 along with standard Gibbs free energies as a function of temperature. Table 1 also lists for various reactions the temperature at which the CO pressure is one atmosphere, under standard state conditions for the reactants and products. In actual smelting practice, the reduction temperature will be raised if the oxide is chemically combined in the gangue, and lowered if the reaction product can dissolve in the ferrochromium alloy. The gangue present in a chromium ore has a significant effect

on the temperature of the smelting zone, thereby influencing the carbon content of the ferroalloy. An ore having a relatively high MgO content will require a higher smelting temperature.

The data in Table 1 indicate that at these smelting temperatures, iron, chromium, and silicon form stable carbides.

In the case of Cr<sub>2</sub>O<sub>3</sub>, it is first reduced to Cr in the alloy. The higher order carbides then take part in the reaction with Cr<sub>2</sub>O<sub>3</sub> to form lower order and more stable carbides.

Table 1. Standard Gibbs energies and equilibrium temperatures of reactions during carbothermic reduction of chromite[3]

Reaction	$\Delta H_{298\text{ K}}$ (kJ/mol)	$\Delta G$ (J/mol)	T in K for pCO=1 atm.
Fe <sub>3</sub> O <sub>4</sub> +C=3FeO+CO	194.2	200,595 -212.86T	942
FeO+C=Fe+CO	161.5	153,251-152.08T	1001
Cr <sub>2</sub> O <sub>3</sub> +3C=2Cr+3CO	803.1	825,029-486.0T	1698
Fe <sub>3</sub> O <sub>4</sub> +5C=Fe <sub>3</sub> C+4CO	703.8	670,083-682.28T	983
3FeO+4C=Fe <sub>3</sub> C +3CO	509.6	470,114-469.42T	1001
7Cr <sub>2</sub> O <sub>3</sub> +27C=2Cr <sub>7</sub> C <sub>3</sub> +21CO	5300.4	5426,008-3453.5T	1571
SiO <sub>2</sub> +3C=SiC+2CO	616.5	624,559-346.06T	1805
3SiO <sub>2</sub> +2SiC=Si+4SiO+2CO	2163	2,167,307-1025.48T	2113
Cr <sub>2</sub> O <sub>3</sub> +3Cr <sub>7</sub> C <sub>3</sub> =Cr <sub>23</sub> C <sub>6</sub> +3CO	956.7	937,000-450.0T	2082

### Thermodynamics involved in ferromanganese production

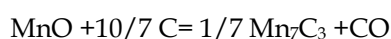
Many manganese-containing ferroalloys are produced and are used in stainless steel industries, foundry and mild steel industries [4].

Table 2. Types of ferromanganese and their general compositions [3]

Alloy	Composition %		
	Manganese	Carbon	Silicon
HCFeMn	72-80	7.5	<1.25
MCFeMn	75-85	<2.0	
LCFeMn	76-92	0.5-0.75	
Silicomanganese	65-75	<2.5	15-25
Ferromanganese silicon	58-72	0.08	23-35
Spiegeleisen	16-28	<6.5	11-45

High-carbon ferromanganese and silicomanganese are produced from a blend of manganese-containing ores, and in the case of silicomanganese, slags and silica are added. Electric submerged arc furnaces are mostly used to produce ferromanganese and silicomanganese. Silicomanganese can be refined to medium or LC ferromanganese as well as manganese metal. High-carbon ferromanganese can be converted to medium-carbon manganese by an oxygen-blowing process.

The reduction of manganese (II) oxide occurs by the contact of carbon with the molten oxide in the slag phase [5]. The overall reaction is:



for which

$$\Delta G = 265.7 - 0.18T \text{ (kJ/mol)}$$

The heat input into electrothermal processes is not only to compensate for heat losses but to drive the endothermic physicochemical reactions. Heating takes place by the flow of electricity from the tips of the electrode, which are submerged in the burden, through the burden and slag to the metal, as well as through the flow of electricity between the electrodes. The ratio of CO to CO<sub>2</sub> in the off-gas is important and can be used to monitor the condition of the furnace. The higher the CO<sub>2</sub> content of the off-gas, the higher the energy efficiency of the process because the reducing potential of the gas is being more fully utilised. Good operation of the furnace is indicated by a CO<sub>2</sub>/(CO<sub>2</sub>+CO) [3] ratio of 0.55. CO/(CO<sub>2</sub>+CO) defined by the Boudouard reaction. This ratio is a function of the temperature. The Boudouard reaction is predominant at high temperatures and produces mainly CO that reacts with the oxidic reducing ores of the burden into CO<sub>2</sub>.

This ratio also depends on the furnace design and operating parameters as well as on the burden parameters. This ratio, as well as the MnO content of the slag, can be used to control the coke rate of the furnace. Undercoking of the furnace is indicated by high MnO content of the slag and a low CO<sub>2</sub> content in the off-gas. A further influence on the MnO content of the slag is the basicity ratio (CaO+MgO)/SiO<sub>2</sub>.

The hotter slag improves the reaction between the slag and the coke and, consequently more MnO is reduced. Increasing the basicity of the slag thus decreases the residual MnO content. The MnO content of the slag is also reduced by increasing the penetration of the electrodes, which also increases the slag temperature. By increasing the basicity of the slag, the recovery of manganese as metal is increased; however, consumption of carbon and electricity also increases.

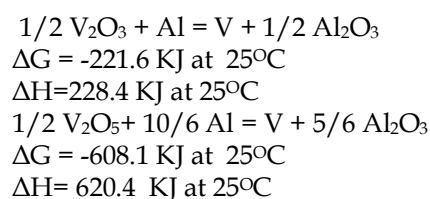
#### **Ferro vanadium production and thermodynamics considerations**

Ninety-eight percent of vanadium mine production occurs in South Africa, China, and Russia. Vanadium is recovered from titanium-bearing magnetite ore processed to produce pig iron in these three countries [6,9]. Slag obtained from this process contains 20–24% vanadium pentoxide; when this slag is further processed to produce ferrovanadium it contains 40–50% vanadium. In order to produce ferrovanadium, aluminothermic reduction of such slags is done, which requires little heat to preheat raw materials in order to conserve aluminium, otherwise the process is self-sustaining. Electric furnaces are used for this process.

The aluminothermic process is used to prepare ferrovanadium alloys as reaction is highly exothermic and ferrovanadium with low carbon content (0.02% to 0.06% C). The charge consists of vanadium pentoxide, aluminum powder, steel chips (iron scrap), and lime. The process might be improved with some additions of magnesia [7,8]. Maximum metal output is achieved when lime content is about 30% to 40% by weight of the vanadium pentoxide amount. The metal contains 82% to 84% V, 1% to 2% Si, <0.05% P, 0.1% Ti, 1.5% Mn, and slag ends with <4.5% V<sub>2</sub>O<sub>5</sub>. Extraction of vanadium (yield) in the alloy is 90% to 96%.

Slag consists of vanadium and loss of vanadium in slag can be reduced by using additional heating at the end of the process. If the vanadium content in the slag remains high, it is recycled to the next heat. The Ellingham diagram (Figure 3) shows that both V<sub>2</sub>O<sub>5</sub> and V<sub>2</sub>O<sub>3</sub> can be reduced by either silicon or aluminium. Silicothermic reduction is no longer economically viable because it involves multiple stages [7] with resulting high losses and vanadium recoveries rarely exceeding 75–80%. The recovery in aluminothermic smelting usually exceeds 96%.

Aluminothermic reduction of vanadium oxides is represented by the following reactions, where the thermodynamic data are taken from the HSC for Chemistry database.



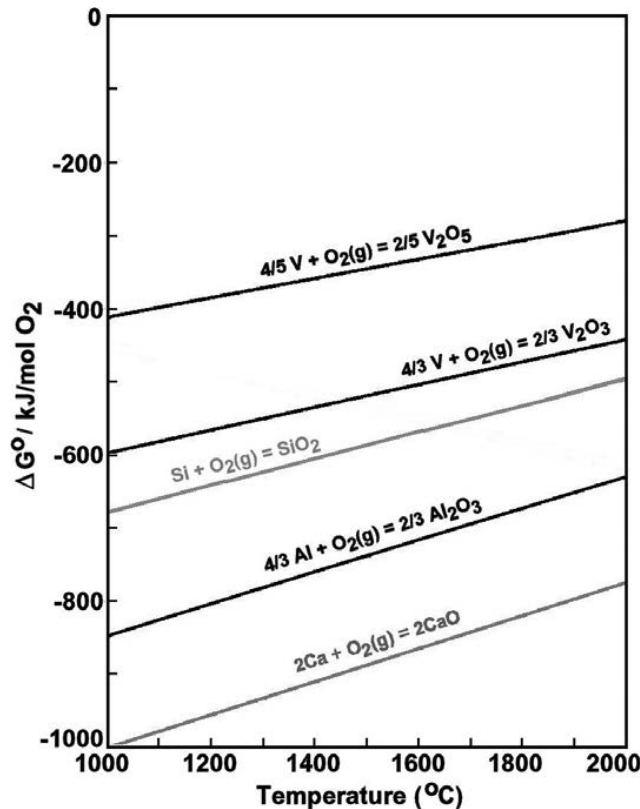


Figure 3. Ellingham diagram for some oxide relevant to ferrovanadium production [7].

Whether or not a reaction provides enough exothermic heat of reaction for successful smelting can be assessed by a rule of thumb, where the heat of reaction is expressed as kilojoules per kilogram of all products [7].

If this value is:

- (i)  $>4500 \text{ kJ} / \text{kg}^{-1}$ , the reaction is uncontrollably violent,
- (ii) between  $4500$  and  $2250 \text{ kJ} / \text{kg}^{-1}$ , the reaction is controllable and requires no external energy input,
- (iii) but if it is  $<2250 \text{ kJ} / \text{kg}^{-1}$ , then an external energy supply is necessary.

The reduction of  $\text{V}_2\text{O}_5$  evolves to  $4562 \text{ kJ} / \text{kg}^{-1}$  and so is predicted to be on the threshold of being violent, but the reduction of  $\text{V}_2\text{O}_3$  only evolves to  $2239 \text{ kJ} / \text{kg}^{-1}$  and is predicted to require an external heat supply. However, the production of  $\text{FeV}_8\text{O}$  requires addition of iron to form the alloy and flux to form the slag, which will add sensible heat demands. The HSC Chemistry for Windows 'heat balance' module was used to determine the adiabatic temperature of the reactions, including the required amounts of iron and CaO. Reduction of  $\text{V}_2\text{O}_3$  gave a temperature of  $1910^\circ\text{C}$  and that for  $\text{V}_2\text{O}_5$  was  $2760^\circ\text{C}$ .

Fe-V Binary phase diagram in Figure 4 is used to estimate liquidus temperature of ferrovanadium being produced; liquidus temperature influences temperature required during smelting. Binary phase diagram shows the liquidus temperature of the  $\text{FeV}_8\text{O}$  is  $1730^\circ\text{C}$ . Smelting takes place at  $1850\text{-}1900^\circ\text{C}$  as superheat is required to allow phase separation.

Aluminothermic smelting produces  $\text{Al}_2\text{O}_3$  as a waste product, and it has a melting point of  $2054^\circ\text{C}$ . A flux is required to lower the liquidus temperature and the viscosity of the slag. The cheapest suitable flux is lime CaO.



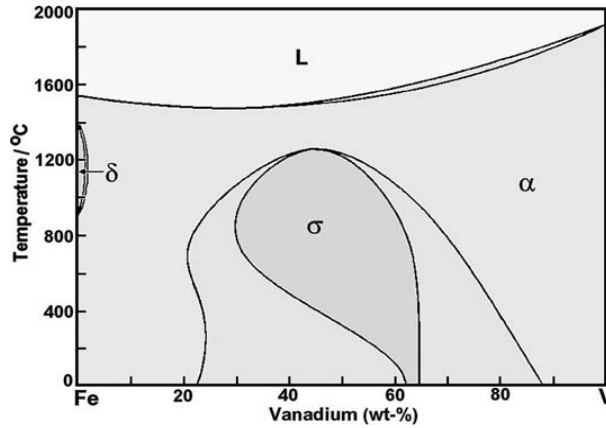
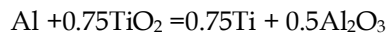


Figure 4. Iron-vanadium phase diagram [7].

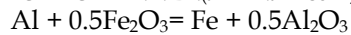
The Ellingham diagram (Figure 3) shows that CaO will not be significantly reduced by aluminium. Temperatures reached during smelting are very high so the slag aggressively attacks the MgO furnace refractories, becoming more severe the higher the temperature, with the result that the slag contains dissolved MgO. The mass of CaO used should be minimised to reduce costs, heat demand and slag volume. The smaller the slag volume, the smaller the mass of dissolved  $V_2O_3$  and unsettled ferrovandium prills, thereby increasing vanadium recovery. These factors indicate that the slag should have a high  $Al_2O_3/CaO$  ratio.

#### Ferrotitanium production and thermodynamic consideration

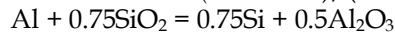
Ferro titanium can be produced from titania slag obtained from vanadium-titanium magnetite with over 40 wt%  $TiO_2$  through aluminothermic reduction. CaO and  $Fe_2O_3$  are added to ensure the slag fluidity, alloy quality, and sufficient physical heat. Addition ratios of Al are optimised by the thermodynamic calculation. Ferrotitanium alloy produced by this method has 30.5 wt% Ti, 8.4 wt% Al, 3.2 wt% Si [10]. However, the macro analysis indicates that some alloy particles with different contents are dispersedly distributed among the slag, and the slag consists of three different phases. It is mainly associated with the reaction intensity and thermodynamic stability. The thermodynamic priority of reduction by Al is  $Fe_2O_3 > TiO_2 > SiO_2$ .



$$\Delta G_1 = \Delta G_1^O + R.T. \cdot \ln \cdot (a_{Ti}^{0.75} \cdot a_{Al_2O_3}^{0.5}) / (a_{Al} \cdot a_{TiO_2}^{0.75})$$



$$\Delta G_2 = \Delta G_2^O + R.T. \cdot \ln \cdot (a_{Fe} \cdot a_{Al_2O_3}^{0.5}) / (a_{Al} \cdot a_{Fe_2O_3}^{0.5})$$



$$\Delta G_3 = \Delta G_3^O + R.T. \cdot \ln \cdot (a_{Si}^{0.75} \cdot a_{Al_2O_3}^{0.5}) / (a_{Al} \cdot a_{SiO_2}^{0.75})$$

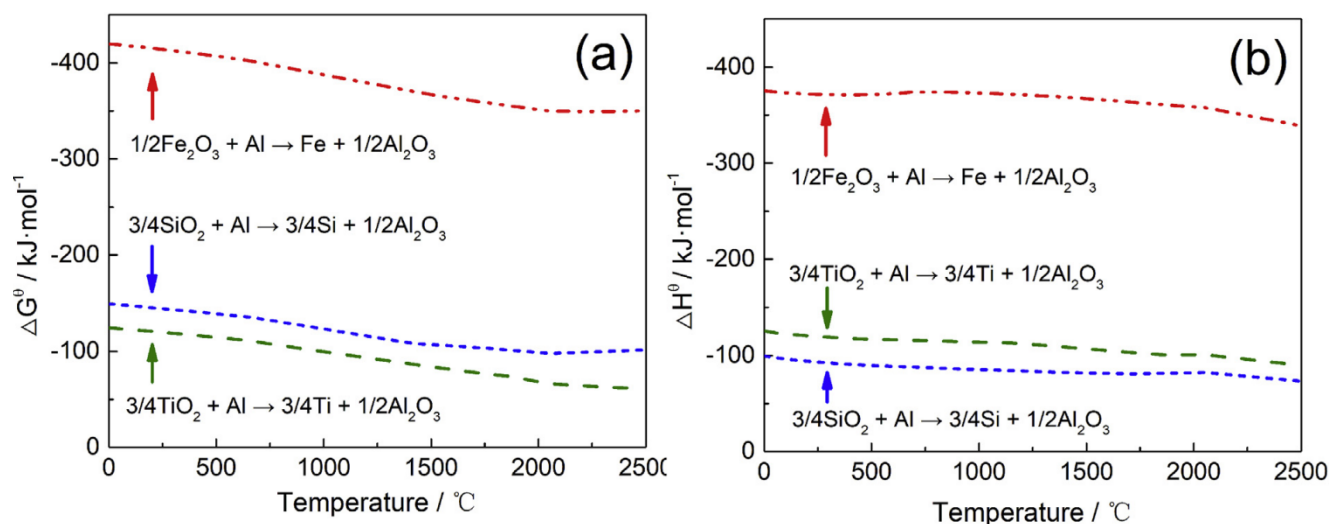


Figure 5. Gibbs free energy change (a) and enthalpy change (b) of the main reactions [10].

It has been reported in other studies [10,11] that the thermodynamic data from Factsage software is reliable for the aluminothermic reduction process. Therefore, according to the Gibbs free energy change of chemical reaction from Factsage calculation shown in above Figure 5(a), the constituents of  $\text{TiO}_2$  and  $\text{SiO}_2$  in titania slag and additive  $\text{Fe}_2\text{O}_3$  are able to spontaneously react with metallic Al in the experimental temperature. In terms of thermodynamic reaction priority, the aluminothermic reduction of  $\text{Fe}_2\text{O}_3$  occurs most easily and preferentially, while  $\text{SiO}_2$  is closed to  $\text{TiO}_2$  whereas Figure 5(b) depicts the changes of reaction enthalpy, illustrating that all three reactions are exothermic, especially the aluminothermic reaction of  $\text{Fe}_2\text{O}_3$ , with  $\sim 350\text{-}370 \text{ kJ mol}^{-1}$ .

Increasing the ratio of Al/titania-slag leads to an increase in Ti, Al, and Si contents in alloy, and a decrease in the slag/alloy ratio.

## SUMMARY AND CONCLUSIONS

The purpose of this paper is to review thermodynamic conditions under which reactions involved in the production of ferro-alloys are feasible and progressing. In order to promote reaction in the desired direction, the knowledge of equilibrium constant  $K$ , through standard Gibbs energy data, is required, for which extensive thermodynamics studies on oxide slags and liquid iron solutions are necessary. Also the activities of various components can be estimated and used to access the extent of equilibrium achieved in a given process. This underlines the importance of the principles of thermodynamics and thermodynamic data in studying the concepts of pyrometallurgy of production of these ferro-alloys.

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