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Modelling and prediction of reactions involving metals, slags and fluxes

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A brief review of Computational Thermodynamics software concerning the modeling and prediction of reactions involving metals, slags and fluxes is presented. Their application for various steelmaking treatments is discussed. Examples concern phosphorus removal in BOF and EAF, ladle steel desulfurization, and specific treatments for inclusions control in semi-killed or Al-killed carbon and stainless steels.

Introduction

Several commercially available computational thermodynamics packages have found extensive applications in the metallurgical, materials and chemical engineering industries. The most widely used, Thermo-Calc[1], FACT[2], ChemSage[3], MTDATA[4] and GEMINI2[5] combine computation codes for multiphase, multi-component equilibrium calculation and databases. Common work, in particular on data assessments and solution models has, for a long time, been performed within SGTE (Scientific Group Thermodata Europe). Some software products are now developed in common by some of the teams. Thus, the FACT and ChemSage groups are proposing common packages called FACTSage and ChemApp with enlarged database platforms and improved users friendly access[6]. Another trend is the increased coupling of purely thermodynamic packages with kinetic models, for instance, the coupling of Thermo-Calc and DICTRA[7].

Nevertheless, because of their generality, these thermodynamic packages are not easily applied to actual industrial situations and require specific adaptation by users proficient in both thermodynamics and computer science[8]. The non-commercially available package CEQCSI, based in a large part on the IRSID slag model, has been developed for very specific applications in the field of Iron and Steelmaking[9]. This software is user friendly enough to permit its routine use in many plants of the ARCELOR group to optimize slag-steel treatments and inclusion control. The database used in the CEQCSI software includes:

- Liquid and solid Fe- based alloys covering the composition range of iron and steels (carbon, alloyed, stainless steels). Several formalisms are available to describe these solutions, in particular, the unified interaction parameter model, and the sublattice models used in Thermo-Calc. For some systems, developments on the basis of the 'Central Atoms' model[10] are also under way[11]
- Liquid oxides (with partial substitution of oxygen by sulphur and fluor). At present, the IRSID 'cell model' has been validated for the system SiO_2–TiO_2–Ti_2O_3–Cr_2O_3–Al_2O_3–Fe_2O_3–CrO–FeO–MgO–MnO–CaO–CaF_2–S. The model has been extended to describe the full range of oxisulfides in the system SiO_2–Al_2O_3–Fe_2O_3–FeO–MgO–MnO–CaO–S[12]. For systems containing P_2O_5, a good description is obtained with an adapted Flood-Grjotheim model, as discussed later
- Solid phases of oxides, sulphides, nitrides, carbides, either stoichiometric, or as solid solutions described by various models (spinels, carbonitrides,...).

Steel dephosphorization

Steel dephosphorization has always been a major concern in Europe and in Japan, whereas BOF steel plants in North America benefitted from easier refining conditions (hot metal with large Si content and very low P content). Only recently did a strong interest in dephosphorization develop in North America for EAF steelmaking, in particular linked to the use of direct-reduced iron and hot briquetted iron[13].

Both in Japan and in Europe, the requirement of improved dephosphorization led to the major innovations in BOF steelmaking during the 1980s. Based on a critical evaluation of the thermodynamics of dephosphorization, as discussed by Fuwa[14], Japanese steelmakers took a very radical approach and developed the new concept of hot metal pretreatment, thus taking full advantage of the beneficial effect of low treatment temperature. Two approaches with Na_2O or CaO based slags were concurrently developed, with the lime route finally taking over due to its technological simplicity. Sano has recently shown that the refining capacity of CaO based slags can be improved by small additions of Na_2O, and this will be applied industrially in NSC plants[15]. At IRSID, a much more incremental approach to improve the BOF process by adding some bottom stirring was taken, in what was to become the new line of combined blowing techniques (LBE process). At that time, we did not have a clear understanding of the large potential for improving P transfer between slag and metal; Turkdogan's evaluation of the phosphorus equilibrium[16] indicated inaccurately that traditionally achieved steel P contents were well below equilibrium with the top slag in the Q-BOP, and that there was no margin for improvement in the BOP. Neither did we have full conscience of the drastic improvement bottom stirring could bring to the fluid flow and mixing time[17]. We were thus faced with the painstaking task of understanding 6-ton pilot plant trials with ,often, very marginal composition changes between the practices tested, and in conditions difficult to make quite reproducible. One of the first steps was to improve our knowledge of phosphorus equilibrium and develop a model able to treat the quite large domain of industrially relevant compositions (both high P and low P hot metal)[18].

The model used to represent the equilibrium partition of phosphorus between metal and liquid basic slags is based essentially on the Flood-Grjotheim's treatment of unpolymerized ionic melts[19]. This treatment assumes that the ionic species present in the slag are the cations (Ca^{2+}, Mg^{2+}, Mn^{2+}, Fe^{2+}, Na^{+},...) and the complex (SiO_4^{4-}, PO_4^{3-}, AlO_3^{3-}, FeO_3^{3-},...) and simple (O^{2-}, S^{2-}, F^{-},...) anions. In its early applications, the hypothesis of an ideal mixing of the various binary systems involved was made, leading to a somewhat deceptive representation of industrial slag properties over wide composition ranges. Elliott *et al.*[20] then proposed to take into account the non-ideality of the ionic melt by means of empirical correlations, in a development that led to a spirited controversy during the period 1975–78, and has been recently revisited by Gaskell[21]. Their approach was applied at IRSID and improved correlations were derived from the very large number of published and original experimental data on both phosphate and silicate slags. Since then, it has been shown that the derived correlations apply also to slags in which iron oxide can be largely replaced by Al_2O_3 or MnO, making it suitable for typical EAF slags as well. In our experience, it is still the best tool we have to estimate the phosphorus partition between steel and basic BOF slags, as our attempts to incorporate phosphorus oxide in the IRSID cell model did not provide such precise results, by far. It is also applied to evaluate the equilibrium partition of sulphur and manganese.

According to this formalism, the phosphorus equilibrium partition between slag and metal is written:

$$\log \left(N_{PO_4^{3-}}\right)^2 \Big/ \left(N_{O^{2-}}\right)^3 \cdot a_P^2 \cdot a_O^5 = N'_{Ca^{2+}} \cdot \log K_{Ca^{2+}} + ... + g_P\left(T, N_{O^{2-}}\right) \quad [1]$$

where: a_P and a_O are the phosphorus and oxygen activities in the metal

N_i represent the ionic fractions and N'_i the equivalent electric fractions

K_i are the equilibrium constants for the elementary exchange reactions

g_P $(T, N_{O^{2-}})$ is the correlation used to take into account non-ideal mixing in the ionic melt.

For the calculation of the equilibrium oxygen activity, the iron oxide activity of the slag is obtained from:

$$\log a_{FeO} = \log\left(N_{O^{2-}} \cdot N_{Fe^{2+}}\right) + \log \gamma_{FeO} \quad [2]$$

equation in which $\log\gamma_{FeO}$ is expressed with an empirical correlation in terms of temperature and slag composition, derived from the aforementioned experimental data.

The values of the equilibrium constants for the elementary exchange reactions and the various correlations are listed in Table I.

Typical performances of various types of converters at the time the LBE process went into industrial operation and completely replaced the BOP in France are summarized in Table II, showing that, indeed, the difference between steel P content and equilibrium P is substantially reduced, in particular thanks to the post-blow stir. We have also included data from the treatment of high P hot metal, now completely obsolete, to illustrate that it was in that case possible to obtain very little disequilibria, owing to the fact that the early formation of a liquid and thus very reactive slag was easily achieved. Today steelmaker's know-how consists in controlling the oxygen blow in order to promote the early formation of the reactive slag, and combined blowing is an asset in that direction. In addition to more reliable analytical results, these processes have resulted in much reduced lime additions and slag weights, opening the way to a better management of converter slag, a by-product still difficult to valorize outside of the steel plant[22].

Ladle steel desulphurization

The basic principles of steel desulphurization are now well understood and the thermodynamic and kinetic requirements are well established[23–25]. The diagrams of Figure 1 indicate, for two slag systems, the calculated values of sulphur partition ratio L_S and $\underline{Al}$ activity at equilibrium with liquid metal containing 0.2%Si at 1600°C. These diagrams have been calculated with the CEQCSI software. The transposition to other steel grades is straightforward: for a given slag composition, L_S is proportional to $a_{Si}^{1/2}$, and $a_{\underline{Al}}$ to $a_{\underline{Si}}^{3/4}$. These diagrams can be applied for both carbon and stainless steels. Note, however, that the actual Al content is about equal to its activity in a low-carbon steel, but that it is about 3.6 smaller (value of the activity coefficient) in a 304 steel grade.

For Al-killed steels, very large equilibrium sulphur partition ratios can be obtained for CaO-Al_2O_3-SiO_2 slags near lime saturation (up to 1000 for lime saturated slags containing about 5% SiO_2, compositions which are

Table I

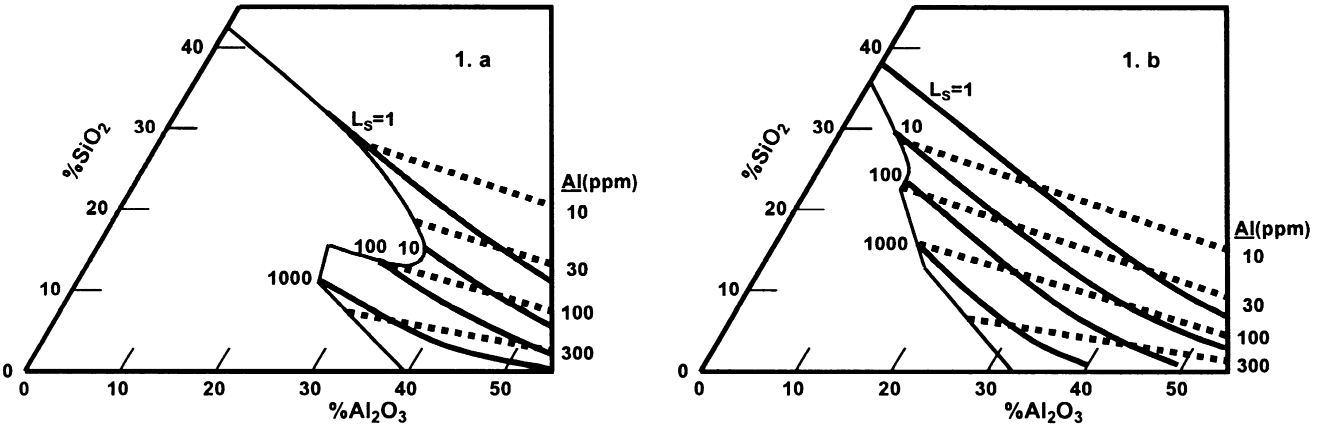
Values of various parameters use in the modified flood model for describing the equilibrium P distribution between steel and basic BOF slags

$\log K_{Ca^{2+}} = 82200/T–34.2$	$\log K_{Fe^{2+}} = 59300/T–33.9$
$\log K_{Mg^{2+}} = 75200/T–34.2$	$\log K_{Na^{+}} = 100600/T–31.7$
$\log K_{Mn^{2+}} = 64370/T–31.4$	
$g_P = -8.35+1.41\cdot\alpha + 13200/T + (40.55-6.95\cdot\alpha-64500/T)\cdot N_{O^{2-}} + (-13.1 + 5.91\cdot\alpha + 22100/T)\cdot(N_{O^{2-}})^2$	
$\log \gamma_{FeO} = 5.5-1.12\cdot\alpha - 5600/T + (-16.07+2.85\cdot\alpha + 20800/T)\cdot N_{O^{2-}} + (10.897 - 1.62\cdot\alpha - 16100/T)\cdot(N_{O^{2-}})^2$	
where $\alpha = N_{SiO_4^{4-}}/(N_{SiO_4^{4-}} + N_{PO_4^{3-}})$	

Table II

Typical P contents in various converters

Blowing conditions	%P steel	%P steel–%P equilibrium
BOF-low P hot metal	0.0127 (σ = 0.0037)	0.0075 (σ = 0.0032)
OLP-LDAC-high P hot metal	0.020 (σ = 0.005)	0.003 (σ = 0.002)
LBE after post-blow stir	0.0124 (σ = 0.0024)	0.0035 (σ = 0.002)
LWS/OBM after stir + Na_2CO_3	0.009 (σ = 0.003)	0.004 (σ = 0.002)



a) Slag: $CaO-Al_2O_3-SiO_2$

b) Slag: $CaO-Al_2O_3-SiO_2$-5% MgO-10% CaF_2

Figure 1. Equilibrium sulphur partition ratio (LS) and aluminium activity in steel with a_{Si}=0.2 at 1600°C for two slag systems:

compatible with steels containing 300 ppmAl). In fact, degrees of desulphurization as high as 80 to 90 per cent for treatments at atmospheric pressure, and 95 per cent for vacuum tank treatments can be consistently achieved provided the slag composition is well controlled and efficient stirring conditions are maintained[23]. Controlling the slag composition in the optimal composition domain requires a very good practice, as this slag contains not only deoxidation products and lime and fluxes additions, but also residual ladle slag and slag carry over from the melting reactor. Figure 2 shows the degradation in desulphurization results, arising from deviation of the slag from lime saturation[24]. In this figure, the lime saturation index represents the ratio of actual lime content to lime content at saturation along the line of constant Al content, as shown in Figure 1. Values smaller than 1 correspond to liquid slags (degree of desulphurization decreases due to the decrease in S equilibrium partition ratio) and values larger than 1 to heterogeneous slags (degree of desulphurization decreases mostly for kinetic reasons). Note also that MgO pick-up from the refractory increases sharply in the domain of liquid unsaturated slags.

In contrast, in the case of semi-killed steels in which very low Al contents are required, the potential for desulphurization is quite limited. The treatment efficiency mainly depends on the steel grade through its acceptable Al content. For a treatment with acid liquid slag of the $CaO-Al_2O_3-SiO_2$ system and Al content not in excess of 10 ppm, the values of equilibrium sulphur partition ratio are very low (around 1), due to the very large domain of dicalcium silicate stability. The domain of liquid slags can be appreciably enlarged by adding fluxes (MgO at contents up to around 8 per cent and CaF_2). Even with these slags, the sulphur removal possibilities remain quite modest, as maximum equilibrium sulphur partition ratios of about 30 can only be achieved. This corresponds, with 10 kgslag/t steel, to a degree of desulphurization of 25 per cent at best. If a strict control of sulphur input at the level of the melting shop cannot be guaranteed (taking into account, in the case of electric furnace, the coal added in the charge for slag foaming purpose, which may represent a very large sulphur input), a cumbersome and costly two-slag practice may be used in the ladle. The first desulphurization treatment with a reasonably basic slag leads to an excessive Al content, which will have to be removed by the subsequent acid slag treatment. Quite obviously, a careful elimination of the first slag is necessary to avoid sulphur reversion.

In the case of stainless steels, the desulphurization slag is no longer mostly a synthetic slag, but it results from the reduction and composition adjustment of the slag in the AOD or VOD. Large slag quantities are then obtained, with a composition difficult to control precisely, which depends on the reduction agent (Fe-Si or Al or a mix of the two). When the reduction is made with silicon only, or mostly silicon, the aimed slag composition is about 30% SiO_2–5% Al_2O_3–15% CaF_2 with typically an equilibrium sulphur partition ratio of 10 and an equilibrium Al content of 3 ppm. Large quantities of fluorspar have to be used to dissolve the lime adjustment necessary to form the desulphurization slag. In fact, a very heterogeneous slag with large quantities of undissolved lime is often obtained, and the final adjustment of steel Al content is done in a non-equilibrium steel/slag situation with a continuous drift difficult to manage. For grades in which the accepted residual Al content is not very low (for instance 30 ppm), it is possible to increase the amount of aluminium in the slag reduction mix and displace the desulphurization slag composition towards the more basic domain (15% SiO_2–25% Al_2O_3–5% CaF_2, corresponding to a value of LS ~ 100) and ensure at the same time the steel/slag Al equilibrium. In those conditions, the lime and fluorspar additions can be drastically reduced, and a calcium aluminate flux can, in some cases, be used to adjust further

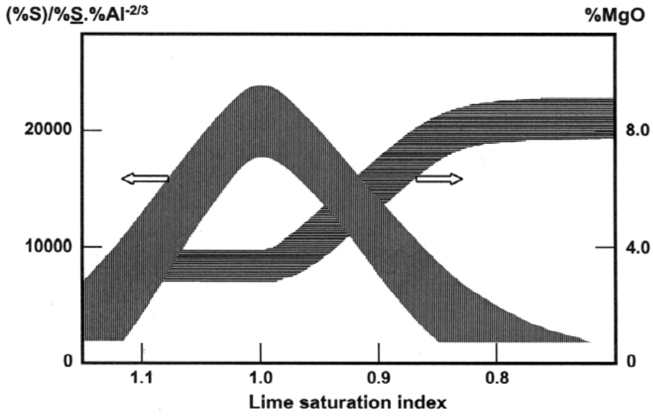


Figure 2. Effect of slag deviation from lime saturation on the desulphurization performance and on slag MgO pick-up from the ladle lining for treatments made in the vacuum tank degasser at the AG der Dillinger Hüttenwerke

Table III
Comparison of two practices for the desulphurization of a stainless steel grade

Additions (kg/ton steel)	Former practice	New practice
Al	1.6	2.9
Si	8.0	6.0
CaO	40	18
CaF2	10	3.8
Slag weight	68	32

the slag composition. An example of such a practice based on the precise thermodynamic evaluation, which has been recently implemented with great success in one of the ARCELOR steel plants is summarized in Table III.

Treatments for inclusions control in steels

Various applications of the CEQCSI software to optimize treatments for inclusions control in a wide variety of steel grades have been discussed in detail at the 6th International Conference on Molten Slags, Fluxes and Salts[26]. They concern the definition of calcium treatments in Al-killed steels, and specific treatments for inclusions control in semi-killed carbon and stainless steels.

A strong trend in modelling and prediction of reactions involving metal and slags is the coupling of computational thermodynamics codes and computational fluid dynamics (CFD) codes to describe reactors. A recent review on the use of fundamental process models in studying ladle refining operations[27] states that no fundamental model that takes all the individual parts of ladle refining into account has been found in the open literature. However, separate fundamental models for prediction regarding alloying, temperature control, sulphur removal, reoxidation, degassing do exist, and attempts at describing inclusion growth, composition change and removal have been made. In particular, a few studies link CFD codes and thermodynamic packages for reoxidation and inclusion coalescence and removal[28–29], taking into account the effect of top slag, refractory lining and ladle glazes (slag coating from previous heats on the lining), but their verification is still lacking. It is thus clear that there is ample room for innovation in this field.

Another domain of progress concerns the control, in addition to their composition, of the size distribution of oxides and other precipitates formed during the solidification of steel. Research in this field has been triggered by the development of the so-called ‘Metallurgy of oxides in steels’, a concept introduced by researchers from Nippon Steel Corporation[30–31]. In these steel grades for tubes or plates, it has been shown that a well-dispersed arrangement of micronic TiOx-containing inclusions formed during solidification, on which manganese sulphide can precipitate, can serve as nucleation sites for acicular ferrite after welding or heat treatment. A kinetic model of precipitation, coupled with CEQCSI and combining nucleation and growth of particles in the interdendritic liquid during steel solidification, has been developed at IRSID[32–33]. It has first been validated for the precipitation of TiN and is in the process of being extended to the precipitation of liquid oxides. Two main features of this kinetic model are that nucleation and growth are treated simultaneously, and they compete in consuming the supersaturation at each moment, and that a mixed controlled growth model combining solutes transport and interfacial kinetics at the precipitate/liquid steel interface is considered. As an example, Figure 3 compares model predictions of the amount of precipitated TiN during steel solidification to laboratory data obtained in directional solidification experiments for a steel grade with two different S contents. The x-axis on this figure indicates the decreasing temperature during the treatment that is treatment time increases from left to right. Actual precipitation occurs much later than would occur in equilibrium conditions, due to kinetic limitations arising from nucleation and growth phenomena. The retarding effect of an increase in sulphur content is explained by a postulated effect on the interfacial energy between TiN and steel, which affects both nucleation and interfacial kinetics. The observed size distribution of TiN precipitates is also in satisfactory agreement with model calculations.

Conclusions

Intensive efforts have been made in the last few years to use established metal and slag thermodynamic models for the appropriate description of more and more complex systems, and incorporate them in Computational Thermodynamics software/database packages. It is expected that the use of these models and confrontation with industrial results will help in defining the specific domains in which further experimental investigations of thermodynamic properties are needed and avoid duplication of studies in domains in which the data is fairly well established.

An ever-increasing number of industrial applications in the field of iron and steelmaking, that take full advantage of the models and of the accessibility of powerful calculation codes have been proposed and they generally show very encouraging results. This is, in particular, the case for ladle metallurgy and inclusions control in steels. More and more, the opportunity of coupling thermodynamic codes and CFD or kinetic codes is put into effect for a better understanding of processes and materials design.

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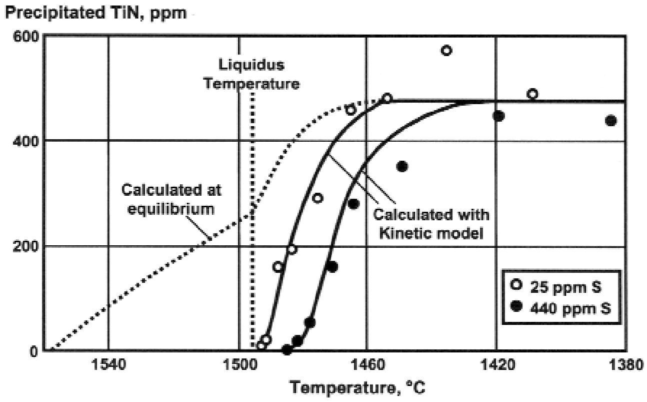

Figure 3. Effect of S content on the calculated (lines) and experimental (circles) sequence of precipitation of TiN during steel solidification, as simulated by experiments of directional solidification. Comparison of the predictions of the kinetic nucleation and growth model with equilibrium precipitation

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