EXPERIMENTAL STUDY AND MODELLING OF VISCOSITY OF CHROMIUM CONTAINING SLAGS

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ABSTRACT

Ferrochromium is the key alloy when stainless steels are produced. It is made by reducing from chromite ore with carbon in a submerged arc-furnace at high temperatures over 1650°C. The metal droplets formed via solid state and smelting reduction processes coalesce and disperse out of the slag as a heavier phase and settle onto the bottom of the furnace as ferrochromium melt. The chromium yield depends on the thermodynamic reaction equilibria between the slag and ferrochromium, but also on the reaction kinetics. Slag´s viscosity in the furnace is a very essential factor influencing both reduction kinetics and separation of metal droplets from the slag. Due to extremely severe conditions, extremely high temperatures and reactive slags, viscosity measurements for FeCr slags have been performed quite little.

First in this study modelling of viscosity is discussed. More emphasis is directed to modified Urbain model and Iida model and its optimization. They were then used to examine the results of viscosity experiments and assessed for certain parameters in order to describe the influence of chromium oxides on slags´ viscosity.

In experimental part an apparatus was constructed to measure the viscosities of molten slags at high temperatures up to 1750°C. Viscosities were measured for Al_2O_3*-CaO-MgO-*SiO_2 *slags, and for several slag systems containing chromium oxides, from a quasi-binary system* CrO_x*-*SiO_2 *to quasi-ternary and quaternary systems. At low oxygen partial pressures the chromium in the slag appears simultaneously as divalent (CrO) and trivalent (*$CrO_{1.5}$*) oxides. The influence of different factors on slag viscosity and the applicability of different models are discussed.*

INTRODUCTION

Ferrochrome is the key alloying component in stainless steel, which is the fastest growing metal in consumption with the steady growing rate of 5-7% per year (*ISSF Statistics Jan2008*). Stainless steels typically contain 18-20% Cr. That has given an impact for increasing production of FeCr which reached the level of 7.75 Mt in 2007 (*CRU Statistics Feb2008*). Ferrochrome is produced in submerged-arc furnaces where chromite ore (lumpy ore, pellets, sinter) are smelted with carbonaceous reductant (coke, anthracite, char) into ferrochrome (containing typically 52% Cr, 2-5 % Si, 7% C, Fe balance) as well as into associated slag. The slag is created in the process from gangue material associated with the ores as well as fluxes added to the burden. The fluxes are added to assist in the smelting by affecting slag formation and the activities of the oxides in the slag, its viscosity and electrical conductivity too.

Slags in FeCr process are based on Al_2O_3-MgO-SiO_2 system with minor contents of CaO and CrO_x. The liquid slag dissolves the already partially reduced solid chromite as the ionic species Cr^{2+}, Cr^{3+}, Fe^{2+} Fe^{3+} and O^{2-}, along with some additional impurity elements. Carbon reacts with oxygen anions in the oxides and forms carbon monoxide gas (CO). As a consequence, metal cations in the slag are reduced, iron first and then even chromium, and preferentially precipitated out of the slag forming metal droplets. The metal droplets coalesce and disperse out of the slag as a heavier phase and settle onto the bottom of the furnace as ferrochrome melt.

The chromium yield depends on the thermodynamic reaction equilibrium between the slag and ferrochromium, but also on the reaction kinetics. In particular, low viscosity slag speeds up the reactions and helps the metal droplets to segregate out of the slag, which consequently improves the yield of the ferrochromium process. Chromium content measured in FeCr slags is typically from 5 to 10% of which a major part is as metallic particles dispersed in the slag. A special feature is that Cr is present in two different valence states in the process conditions, namely Cr^{2+} and Cr^{3+}. The thermodynamics of Cr-containing slags were studied by several authors [1]. Oxidation state of chromium depends on the local oxygen partial pressure, temperature as well the composition of the slag.

There is little previously published data regarding the viscosity of chromium containing slags. Some research was conducted in the late Soviet Union on the viscosities and electrical conductivities of ferrochrome process slags [19]. However, the results were not fully comparable with the present study which had precise control of oxidation stages of chromium in slags and high sensitivity of the viscometer. In general, low P_{O2} increases the fraction of CrO instead of $CrO_{1.5}$ and lowers the liquidus temperature of the slag [2, 3, 4].

Another reason for the lack of reliable experimental viscosity data is the high melting temperature of chromium containing slags, which often exceeds the maximum long-term range of the experimental furnaces and limits of the available construction materials for crucibles and rotating cylinders. As a consequence, viscosity measurements are quite expensive to be performed because of the high temperature refractory materials which often can only be used once or for a limited time. The measurements are also very time-consuming due to the long heating and cooling times of the furnace, along with sample preparation and analysis. Furthermore, the experimental runs often fail, and the results are subject to fairly large errors [6].

VISCOSITY MODELS

Arrhenius was the first researcher who systematically studied the temperature dependence of different chemical phenomena and material properties such as viscosity, at the end of the 19th century. According to him, a logarithmical correlation against the temperature can be written for viscosity:

$$\eta = A\,exp\left(\frac{\Delta G}{RT}\right) \tag{1}$$

where A is a proportionality constant, ΔG is the activation energy for viscous flow, T represents temperature and R is the universal gas constant. Later, the viscosity equation was derived from basic fundamental principles of physics. The most famous solutions were conducted by Eyring *et al.* based on absolute reaction rate theory [7], Bockris and Reddy [8], and Weymann [9] both based on the hole theory. Of these the Weymann equation is shown below.

$$h = \left(\frac{RT}{E_W}\right)^{\frac{1}{2}} \cdot \frac{(2mkT)^{\frac{1}{2}}}{v^{\frac{2}{3}} P_V} \cdot exp\left(\frac{E_W}{kT}\right) \tag{2}$$

where m and v are the mass and volume of the structural unit, E_W the energy well, k and T Boltzmann constant and absolute temperature, and P_V is the *hole* probability connected with the structural model of liquid. The solutions may be expressed in a form of *general Arrhenius equation*:

$$N_V = exp\left(\frac{\Delta S_V}{R}\right) \cdot exp\left(\frac{-\Delta H}{RT}\right) \tag{3}$$

where n is either 0, ½, or 1 for the Eyring, Bockris & Reddy or Weymann equations, respectively. Thus the Eyring equation is consistent with the classical Arrhenius equation. These theoretical equations are mainly applicable to liquid metals with a simple monoatomic structure. Concerning complex liquids like silicate melts the concept of activation energy and logarithmic temperature relationship can still be represented using the formulas $\log\eta = A + B/T$ or $\log\eta = A + B/T + C/T^2$. The problem is how to incorporate the composition dependent structural effects into the viscosity equation. Only few approaches are shortly referred here.

G. Urbain [10] conducted a viscosity expression starting from the statistical viscosity model by Weymann. The two parameters A and B in *generalised Arrhenius equation* were related to the composition, which was first simplified by dividing components into glass forming, modifying and amphoteric cations, and calculating the equivalent composition of each element. Weymann constructed the viscosity Equation 2 on the basis of the hole theory of liquids, according the ‘hole’ probability P_v, which is proportional to the concentration N_v of ‘holes’ given at T by the equilibrium:

$$N_V = exp\left(\frac{\Delta S_V}{R}\right) \cdot exp\left(\frac{-\Delta H}{RT}\right) \tag{4}$$

where ΔH_V and ΔS_V are the partial molar enthalpy and entropy associated with the 'hole' formation. Parameters A and B were expressed in Equation 3 in molar quantities:

$$A = \left(\frac{k}{E_w}\right)^{\frac{1}{2}} (2mk)^{\frac{1}{2}} \left(\frac{1}{v}\right)^{\frac{2}{3}} exp\left(\frac{-\Delta S_V}{k}\right) \tag{5}$$

$$B = \frac{E_w + \Delta H_V}{R} \tag{6}$$

A simple relation between A and B was written: $-\ln A = m \cdot B + n$ (7)

where the pre-exponential term A is linked to the parameter B, with adjustable parameters m and n.

According to Urbain, ionic liquids like slags have very distinctive values of m and n. In order to conduct a more accurate description of A and B over the entire compositional range of the studied slag systems Kondratiev and Jak [11] proposed that the m value would be a composition dependent variable and came to *Modified Urbain formalism*:

$$m = \sum m_i X_i\,, \tag{8}$$

$$\eta = A\,exp\left(\frac{10^3 B}{T}\right) \tag{9}$$

where mi is a value of pure oxide (Al_2O_3, CaO, 'FeO', SiO_2, etc.), and Xi is the mole fraction of the corresponding oxide. Often, experimental viscosity data do not exist for pure oxides because of their high melting temperatures, but it is possible to extrapolate the activation energies (B-parameters) of pure oxides from the binary data (e.g. CaO-SiO_2). The n parameter still was considered constant, but a new value was proposed, which was more coherent with the new m values. As a result, Kondratiev and Jak constructed a viscosity model, which was able to predict the viscosities of the Al_2O_3-CaO-'FeO'-SiO_2 system with good accuracy. Viscosity η in the modified Urbain model is expressed through the Equation (9), where *T* is the absolute temperature [K], and the pre-exponential term *A* is linked to the parameter *B* through the compensation law (*m* and *n* are the adjustable parameters (Equation 7). Later in this work the Modified Urbain Model is applied.

The viscosity model developed by T. Iida is based on the Arrhenius type of equation, where the network structure of the slag is taken into account by using the basicity index (Bi) [12]. In the original Iida model all the needed parameters could be found from handbooks or calculated from physical properties. The temperature-dependent parameters were optimized based on viscosity data. To consider also amphoteric oxides, such as Al_2O_3 and Fe_2O_3, the model was modified by defining composition dependent α-parameters (α^*) for each slag system and temperature [13].

Several other viscosity models have been developed based on Weymann-type equation, application of different slag models to describe composition dependence (basicity) and different mathematical expressions e.g. polynomial approaches: Du Sichen, J.Bygdén and S. Seetharaman, 1992; L. Zhang and S. Jahanshahi, 1998; M. Nakamoto, J. Lee and T. Tanaka, 2005; A. Kondratiev and E. Jak, 2005; K. C. Mills, 1993 [4].

EXPERIMENTAL

The concentric cylinder method using molybdenum or platinum components has been shown to provide the most accurate results for measurements of slag viscosities at high temperatures in a 'round robin' test coordinated by BCR (Bureau Communautaire de Référence) [6]. Similar technique was applied in the present study.

The vertical furnace was an Entech ETF 50 –175 V, equipped with molybdenum silicide heating elements and a maximum continuous temperature of 1750°C. Temperature was controlled with a Eurotherm 903P-unit. A commercial Brookfield LVDV-II+ viscometer incorporated in the furnace. The principle of operation is to drive a spindle, which is immersed in the test fluid, through a calibrated spring with certified error limits within ±1% of the maximum viscosity range. The measurement range is determined by the rotational speed of the spindle, the size and shape of the spindle, the size of the crucible, and the full scale of the rotating spring. The viscous drag of the fluid against the spindle is measured by spring deflection and detected with a rotary transducer. The viscometer was installed in the lift, which enabled immersion and removal of the spindle from the liquid slag.

In most experiments both the crucible and spindle were made of molybdenum which is well resistant at high temperatures against FeCr type slags but without iron. In special experiments even chromium crucibles and spindles were used when an equilibrium p_{O2} between Cr metal and *CrO* in slag was pursued. The furnace was hermetically sealed and an inert or reducing atmosphere was kept in the furnace by purified argon, Ar + H_2 or CO + CO_2 flow.

The experimental set-up has been described in details elsewhere [2, 3, 4].

EXPERIMENTAL RESULTS

Almost one hundred viscosity measurements were performed, but only about half of them succeeded. Main problems were caused by too high melting temperature of the slag or material failure problems due to high temperatures. Results of successful experiments of about 50 different compositions were shown in reference [5]. Selected results representing different slag compositions are shown in Table 1. The slags were chemically analysed after the trial even for CrO and Cr_2O_3 except for CaO-CrO_x-SiO_2 slags equilibrated with metallic chromium (slags 23-28) which are given as target compositions, and the distribution of total chromium content CrO_x into CrO and Cr_2O_3 was estimated by regression analysis based on an earlier work [14].

In Figure 1 viscosity values of slags No. 2, 3 and 4 were compared with corresponding data from the literature in order to validate the measuring technique. The results are in very good agreement when taking into account somewhat differing compositions [15, 16].

In Table 1 viscosity measurements for chromium containing slags are summarized. Figure 2 is based on slags 23-28. It suggests that addition of CrO_x has a rather strong decreasing effect on slag viscosity but the effect gradually becomes weaker at high percentages. However, because the ratio of CaO/SiO_2 was not constant but decreased when adding CrO_x the phenomenon cannot be seen directly. When looking at the changes of composition from slag 23 to slag 28 it is seen that SiO_2 content decreases and the sum of the basic oxides, CaO and CrO_x increases. Two-valent oxide CrO is generally assumed to be basic whereas three-valent oxide Cr_2O_3 (or $CrO_{1.5}$) could be considered amphoteric like Fe_2O_3. However, viscosity measurements have shown that even Cr_2O_3 is a basic oxide although somewhat weaker base than the two-valent CrO [4]. Corresponding effect was observed in other systems like CaO-MgO-SiO_2 slags when adding CrO_x [5].

Table 1: Examples of results of the viscosity measurements for slags. Viscosity in Poise; 1P=0.1Pa s

	CrO	Cr_2O_3	Al_2O_3	CaO	MgO	SiO_2	Log η (P), T(K)	Temp. Range (°C)
1				51.91		48.09	$\frac{7443.9}{T} - 3.8724$	1460-1750
2				54.5		45.5	$\frac{7989.1}{T} - 4.1227$	1440-1750
3					37.4	62.6	$\frac{8890.3}{T} - 4.0138$	1550-1750
4				24.6	24.0	51.4	$\frac{7971.3}{T} - 4.0672$	1440-1750
6			27.05	10.21	19.57	43.16	$\frac{8987.4}{T} - 4.2274$	1590-1750
7			23.02	2.20	34.43	40.35	$\frac{8345.4}{T} - 4.0039$	1620-1750
12			22.22	2.08	39.25	36.45	$\frac{8554.1}{T} - 4.2775$	1690-1750
16	1.60	1.58		52.84		43.98	$\frac{7974.3}{T} - 3.9934$	1570-1750
18	3.00	1.60		50.26		45.14	$\frac{8063.7}{T} - 4.1689$	1470-1750
23	9.77	0.23		35		55	$\frac{8658.8}{T} - 4.2288$	1440-1750
24	17.88	2.12		30		50	$\frac{8067}{T} - 4.0721$	1450-1750
25	23.61	6.39		25		45	$\frac{6921.6}{T} - 3.7161$	1500-1750
26	27.27	12.73		20		40	$\frac{6237.2}{T} - 3.5109$	1550-1750
27	28.96	21.04		15		35	$\frac{7698}{T} - 3.3615$	1600-1750
28	28.84	31.16		10		30	$\frac{8521.5}{T} - 5.0355$	1550-1750
37	1.40	18.37			27.05	53.18	$\frac{9948.7}{T} - 5.056$	1500-1750
38	3.10	26.24			24.31	46.36	$\frac{10984}{T} - 5.9907$	1400-1750
39	3.24	37.31			13.93	45.53	$\frac{10717}{T} - 5.7385$	1430-1750
40	1.14	18.17	14.78		10.74	55.18	$\frac{9361.8}{T} - 4.0937$	1600-1750
41	2.52	25.51	9.73		9.32	52.93	$\frac{9.996.2}{T} - 4.5761$	1600-1750
42	4.08	33.63	7.71		7.30	47.28	$\frac{11437}{T} - 5.6094$	1630-1750
43	2.17	6.63		23.23	20.90	47.07	$\frac{7754.3}{T} - 4.0557$	1445-1750
44	5.04	12.18		21.17	18.85	42.75	$\frac{9513}{T} - 5.1844$	1445-1750
45	8.12	12.46		21.48	16.61	41.33	$\frac{20501}{T} - 10.7736$	1550-1750
48	2.86	7.54	38.69	50.91			$\frac{19557}{T} - 10.2619$	1680-1750

The code numbers (1 to 48) are the same as those used in reference [5]

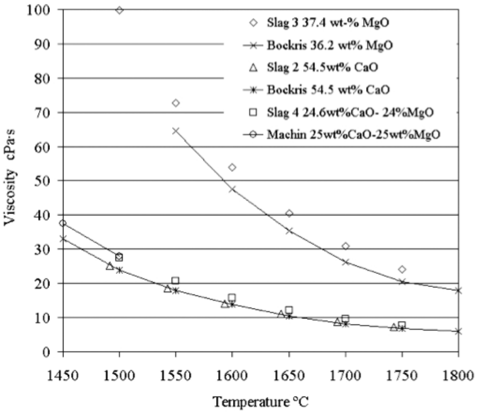

Figure 1: Comparison of viscosities of slags 2, 3 and 4 with literature data [15, 16]

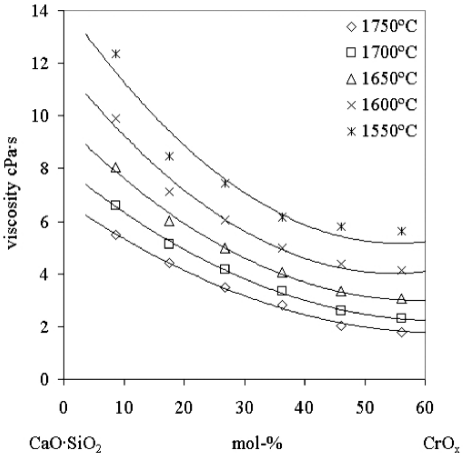

Figure 2: Effect of CrO_x addition on the viscosity of $CaO\text{-}SiO_2$ slags (numbers 23-28 in increasing order). The whole compositions were given in Table 1

DISCUSSION

As mentioned above there is a clear shortage of reliable data concerning the effect of chromium oxides on slags´ properties like viscosity. Respectively, the role and behaviour of CrO and Cr_2O_3 have not been tried to take into account in viscosity modelling. The research group of the authors has done plenty of experimental work and also made efforts to model viscosity of Cr-containing slags. In the earlier studies both the modified Urbain model and the modified Iida model were extended to slags containing Cr oxides and the parameters were assessed with experimental data [2, 3, 4, 13]. Figure 3 was constructed applying the modified Urbain model, for which the CrO and Cr_2O_3 ($CrO_{1.5}$) parameters were optimised using the measured viscosities of several slag systems containing chromium [4]. At zero concentration of CrO_x, the modified Urbain model deviates from the modified Iida model with 1.12 cPa s, which is a moderate deviation. For practical purposes, it is most interesting to know the viscosities of the slags which contain chromium less than 10 wt% and are in contact with liquid chromium-iron alloy. In this region, the addition of a total of 10 wt% of $CrO\text{-}CrO_{1.5}$ (in 50:50 ratio) decreases the viscosity by 2 cPa s in the Iida model with conventional parameters α, and by 2.87 cPa s with optimised α parameters. Similarly, the addition of 10 wt% CrOx mixture decreases the viscosity by 4.87 cPa s according to the modified Urbain model. For the $CaO\text{-}CrO_x\text{-}SiO_2$ slags measured at high oxygen partial pressure [2, 3] and containing less than 9 wt% of CrO_x, the Iida model and the modified Urbain model performed equally well with average deviations of 11.77% and 11.90%, respectively.

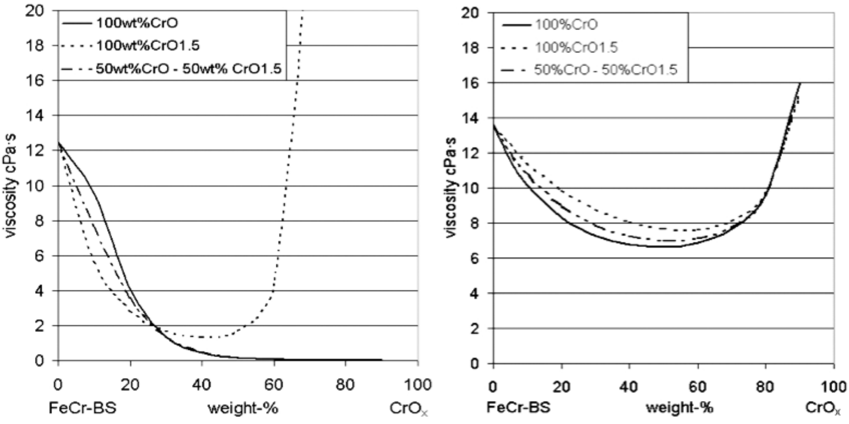

Figure 3: Effects of hypothetical pure CrO and $CrO_{1.5}$ additions into typical FeCr-process base slag (FeCr-BS) on viscosity according to the modified Urbain model (left) and optimized Iida model (right) [5]

The measure of characteristic basicity of an oxide in the Urbain model can be considered the value of the m parameter, i.e., the lower the m-value, the more acidic the oxide, and vice versa. Even though the m parameter for CrO was defined to be 0.75 and less than 0.5 for $CrO_{1.5}$, the modified Urbain model predicts that $CrO_{1.5}$ decreases viscosity more effectively than CrO up to 25 wt% CrO_x concentration. For viscosity predictions of real slags, this is not an issue because real slags contain a mixture of CrO and $CrO_{1.5}$ oxides, and the error is counterbalanced. This reveals a weakness in the Urbain formalism, which requires a large number of viscosity parameters (polynomial constants) to be identified, which is quite difficult, especially in the case of multivalent elements such as chromium. According to Figure 3 the modified Urbain model predicts that viscosity decreases to very low values when pure CrO is added over 40 wt%, whereas addition of pure $CrO_{1.5}$ leads to abrupt viscosity rise at 60 wt% $CrO_{1.5}$. The result is quite reasonable if this is an indication of $CrO_{1.5}$ saturation and solid phase formation. When a 50 wt% CrO-50 wt% $CrO_{1.5}$ mixture is added to the slag, the effect of CrO seems to override the effect of $CrO_{1.5}$ and the slag is still liquid and low viscous at high CrO_x contents.

Concerning the performance of chromium in slags and its influence on slag properties e.g. viscosity two aspects are especially interesting: the predominance of Cr^{3+} and Cr^{2+} as well as the total effect of CrO_x to slag behaviour and properties. The relation between the two oxides can be written:

$$CrO + \frac{1}{4} O_2 = CrO_{1.5} \tag{10}$$

By writing the equilibrium constant: $K = a_{CrO1.5}/a_{CrO} \cdot p_{O2}^{1/4}$ it can be seen that ($\%Cr^{3+}/\%Cr^{2+}$) should be related to $p_{O2}^{1/4}$. Assuming that the ratio of activity coefficients $\gamma_{CrO1.5}/\gamma_{CrO}$ is constant there is a linear logarithmic relationship. However, if the activity coefficients depend on the composition there would be some deviation from linear behaviour. As different researchers have worked under different oxygen partial pressures it is possible to collect together data from literature and put in a same figure. In Figure 4 selected results from Xiao [1], Pei *et al.* [17] and Morita *et al.* [18] were collected, recalculated to get ($\%Cr^{3+}/\%Cr^{2+}$) and drawn against the respective p_{O2} in logarithmic scale. A reasonable dependence can be stated although the slag compositions i.e. basicity and chromium contents greatly vary, and the experimental and analysis techniques can also differ.

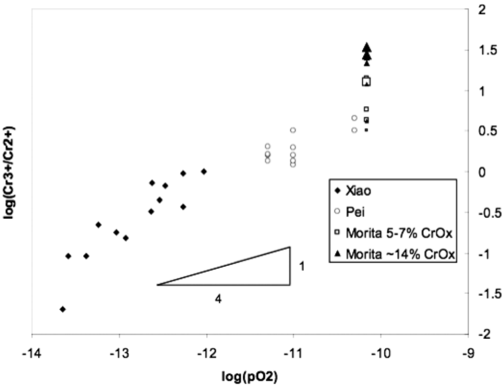

Figure 4: Compilation of some results from different researchers showing the ratio of 3-valent and 2-valent chromium in slags as a function of p_{O2}. T= 1600°C. The data consist of different slag compositions with varying basicities and CrO_x contents: [1]:B= 0.9-1.1; [17]:B=2.2-2.4; [18]:B= 0.54 -1.40; symbol size grows with increasing basicity.

CONCLUSIONS

Viscosity measurements were carried out to find the effect of chromium oxides on slags viscosity. Results were utilized to assess specific parameters for Cr-oxides in viscosity models for multi-component slags in large composition range. The modified Urbain model and modified Iida model were assessed and tested. The models gave reasonable results and confirmed the basic network modifying character of both CrO and Cr_2O_3. CrO had slightly stronger basic effect.

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