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Původní práce

VISCOSITY OF MELTS IN THE SYSTEM $CaO-FeO-Fe_2O_3-SiO_2$

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The viscosity of melts in the system $CaO—FeO—Fe_2O_3—SiO_2$ *decreases with increasing content of calcium oxide and iron oxides. The viscosity of the melts in question was found to be a linear function of the mean mole mass of the polyanions calculated on the basis of a statistical model, and an exponential function of the reciprocal value of thermodynamic temperature. The coordination of iron atoms in the melt is genetrally low. The* Fe^{3+} *cations were not proved to participate in the formation of the polyanionic network. Complex anions of the type* FeO_4^{5-} *or* $Fe_2O_5^{4-}$ *can be assumed to be present. Their content decreases with increasing temperature while a larger portion of larger silicate polyanions is formed. In this way, the* Fe^{3+} *cation takes over the function of the tetrahedral network modifier. The phenomenon results in an increase of the mole mass of polyanions with increasing temperature.*

INTRODUCTION

The system $CaO-FeO-Fe_2O_3-SiO_2$ can be regarded as a model system of steelmaking slags. The viscosity of melts in this system is one of the more important properties determining the technological parameters of the refining process. In view of the considerable reactivity of these melts, the experimental determination of their viscosity at high temperatures is exceptionally demanding. This is why the data on the viscosity of these melts in the literature are comparatively rare and frequently controversial.

The viscosity of melts in the system $CaO-FeO-SiO_2$ was measured for the first time by Kozakevitch [1] at 1400 °C. The author worked with a crucible made of sintered MgO and used argon atmosphere and a rotary viscometer. Further studies were concerned with measuring the viscosity of melts in the system $MgO-FeO-SiO_2$ [2] in Mo crucibles using argon atmosphere, and in the system $CaO-FeO-SiO_2-Al_2O_3$ [3] by means of a torsion viscometer in nitrogen atmosphere. Inert argon atmosphere, iron crucibles and a rotary measuring method were likewise used in measuring the viscosity of melts in the system $FeO-SiO_2$ in [4]. The papers [5, 6] were concerned with measuring the viscosity of melts in the system $Fe_xO_y-SiO_2$ in air atmosphere; the ratio of the bivalent and trivalent iron in the melt was given by temperature and the total content of iron oxides. The viscosity of melts in the system $CaO-FeO-Fe_2O_3-SiO_2$ in air atmosphere has not so far been measured.

In studying the properties of melts in the system $CaO-FeO_x-SiO_2$ in air atmosphere, it is necessary to know the equilibrium composition as a function of temperature and of total composition. The phase equilibria in this system and thus also the equilibrium compositions, based on the determination of the relative contents of bivalent and trivalent iron in rapidly quenched specimens of melts, were studied in [7, 8].

Viscous flow involves transport of momentum between the individual particles of the moving liquid. The coefficient of dynamic viscosity is therefore obviously related to the mass of particles constituting the given liquid. Unlike the electrical conductance of silicate melts, where the charge is transferred above all by cations, the transport of momentum in viscous flow is provided mostly by anions. In the case of silicate metls, the mean mole mass of the anions is a suitable correlation parameter with respect to dynamic viscosity. According to Masson et al. [9] the distribution of silicate anions in a melt is established by polycondensation reactions of the type

$$SiO_4^{4-} + Si_nO_{3n+1}^{2(n+1)-} = Si_{n+1}O_{3n+4}^{2(n+2)-} + O^{2-} \tag{1}$$

where the anions with the general formula $Si_nO_{3n+1}^{2(n+1)-}$ form solely linear and branched chains. Particularly at higher SiO_2 concentrations the melt will obviously contain also cyclic and globular particles. The existence of such particles was pointed out by Pretnar [10] who assumed that the melts of binary silicates include short chains ($n \leqq 5$) as well as globular particles with the general formula $Si_nO_{4n-f_n}^{2(2n-f_n)-}$, where $n \geqq 6$. The number of bonds between the SiO_4 tetrahedra in the polysilicate anions is designated f_n. It thus holds for chains that

$$f_n = n - 1. \tag{2}$$

And for condensed polyanions it then holds that [10]

$$f_n = 2n - 1.71 \times n^{2/3} - 0.5. \tag{3}$$

The equilibrium according to equation (1) can likewise be described generally by the schematic equation

$$2\,O^- = O^\circ + O^{2-} \tag{4}$$

having the equilibrium constant

$$K = \frac{x(O^\circ)\,x(O^{2-})}{x^2(O^-)}, \tag{5}$$

where O° and O^- represent the bridging and the non-bridging oxygen atoms respectively. The equilibrium, which determines the quantitative distribution of anions in binary silicate melts, can thus be described in this way using a single equilibrium constant.

The mean mole mass of silicate anions and the mean number of SiO_4 tetrahedra in the anion can be calculated according to the equations

$$\bar{M} = \frac{\Sigma M_n x_n}{\Sigma x_n}; \qquad n = \frac{\Sigma n x_n}{\Sigma x_n}, \tag{6}$$

where M_n is defined by

$$M_n = 28.086n + 16(4n - f_n) \tag{7}$$

and for x_n, Pretnar derived the equation

$$x_n = x(SiO_2)\,(1 - q)^2\,q^{n-1}, \tag{8}$$

where $q = x_{n+1}/x_n$ is the parameter of a geometrical series determining the concentration of polymerized anions in the form

$$\Sigma\, n^{2/3}\, q^{n-1} = 1 + \sqrt[3]{4}q + \sqrt[3]{9}q^2 + \dots \tag{9}$$

The polymerization degree P was defined by Pretnar as the ratio between the number of oxygen bridges (i.e. bonds between SiO_4 tetrahedra) present in the real structure, and the total theoretically possible number of bridges,

$$P = \frac{\Sigma f_n x_n}{2x(SiO_2)}, \tag{10}$$

where x_n is the mole fraction of the silicate anion formed by n SiO_4 tetrahedra. The following equation holds for the equilibrium constant (5) and the polymerization degree P:

$$P = \frac{1}{2 - 8K} \left\{1 - \frac{x(MeO)}{2x(SiO_2)} - 8K + \sqrt{1 + \frac{x(MeO)}{2x(SiO_2)} \left[\frac{x(MeO)}{2x(SiO_2)} + 16K - 2\right]}\right\}. \tag{11}$$

Pretnar derived the following relationship between parameter q and polymerization degree P:

$$\frac{2P}{1 - q^2} = \sum_{n=1}^{5} (n - 1)\, q^{n-1} + \sum_{n=6}^{\infty} (2n - 1.71 n^{2/3} - 0.5)\, q^{n-1}. \tag{12}$$

The present study was concerned with measuring the viscosity of melts in the system $CaO-FeO-Fe_2O_3-SiO_2$ in air atmosphere in the temperature range of 1560 K to 1900 K. Using the known equilibrium composition of the melts [7, 8] and Pretnar's statistical model [10] the mean mole mass values of the silicate polyanions were calculated, and the dependence of viscosity on these values was plotted.

EXPERIMENTAL

The viscosity measurements were carried out at 5—6 different temperatures using a high-temperature oscillation viscometer of our own construction. The apparatus is described in detail, together with the verification of its function, in [11]. The measuring body employed was a cylinder with conical ends, where the cylinder radius and height, as well as those of both cones were (3.718 ± 0.005) mm; the body was of PtRh40 alloy. The other parts of the suspension system with total mass of 497.5 g, were made of stainless steel and brass. The suspension system was suspended from a torsion wire made of PtW8 alloy, 0.3 mm in diameter and about 60 cm in length. The moment of inertia of the suspension system was $(5.464 \pm \pm 0.009) \times 10^{-5}$ kg m^2, the oscillation period in air was 4.6146 s. The damped harmonic oscillations of the suspension system were recorded by means of a halogen lamp beam, reflected from the suspension system mirror and received by two phototransistors placed in the beam trajectory. The set of time invertals was transmitted by means of an interface to the memory of the EMG 666 calculator. The mathematical processing of the set of time intervals, the calculation of the periods, the amplitudes and the logarithmic decrement were carried out in the way described in detail in [12].

The viscosity of the melts being studied was calculated according to the modified Verschaffelt's equation [13],

$$\eta = \frac{3\delta \,.\, I}{5\pi R^3 \tau_0} \left(\frac{1}{2 + b_1 R + P}\right). \tag{13}$$

where

$$P = \frac{b_1 R + 1}{(b_1 R + 1)^2 + b_1^2 R^2}\,; \quad b_1 = \sqrt{\frac{\varrho \pi}{\eta \tau}}\,,$$

η is the dynamic viscosity [Pa . s], ϱ is the density of the melt measured (kg m^{-3}), R is the radius of the measuring body (m), I is the moment of inertia of the torsion pendulum (kg m^2), τ is the period in the melt being measured (s), τ_0 is the period in air (s) and δ is the logarithmic decrement. The numerical constant 3/5 follows from the geometrical shape of the measuring body and was determined by calibration in aqueous glycerol solutions as described in [11]. The densities of the melts, required for viscosity calculations, were taken from [14].

About 100 g of the sample was placed in a platinum crucible at the centre of an electric resistance furnace. The temperature was measured by means of a PtRh6—PtRh 30 thermocouple immersed in the melt; the accuracy was ± 5 K.

The samples were prepared by fusing the corresponding amounts of calcined $CaCO_3$ A.R., SiO_2 A.R. and Fe_2O_3 A.R. in a platinum crucible. The composition of the samples was chosen, as far as possible, to follow the curve representing the change in composition of the model slag in the system $CaO-Fe_2O_3-SiO_2$ during the steel-making process, as published in [15]. Two sections were chosen, having the respective constant ratio $k_1 = x(CaO)/x(SiO_2) = 1$ and $k_2 = x(CaO)/x(Fe_2O_3) = 4$. The equilibrium composition of the melt at the experimental temperature was determined according to the equation

$$r = 1.7273 - 6.592 \times 10^{-4} T/K + 0.223 k_1 + 0.116\, x'(Fe_2O_3), \qquad (14)$$

where $r = x(Fe_2O_3)/[x(FeO) + x(Fe_2O_3)]$, T is the temperature (K) and $x'(Fe_2O_3)$ is the mole fraction of ferric oxide in the initial mixture. Equation (14) was derived in the present study on the basis of phase equilibrium investigations described in [7, 8]. The equilibrium melt composition was checked after the measurements by determining the content of bivalent and trivalent iron in rapidly quenched specimens. Volumetric analysis and atomic absorption spectroscopy were used, and the results were in satisfactory agreement with the equilibrium composition calculated according to equation [14]. Fig. 1 shows the composition plots for the samples investigated.

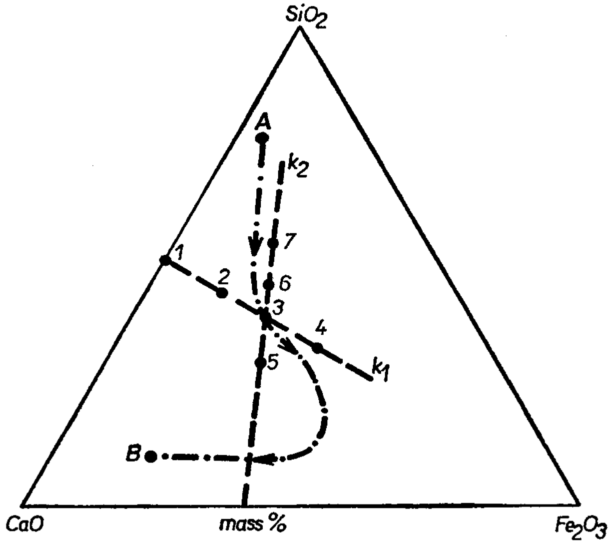

Fig. 1. Representation of the selected cross sections in the system $CaO—Fe_2O_3—SiO_2$. *1—7: figurative points of the initial composition of the samples; A—B: the course of change in slag composition in the course of the refining process [15].*

The temperature dependence of viscosity of the investigated melts was expressed by means of second degree polynomials in the form

$$\log \eta = a + b\frac{1}{T} + c\left(\frac{1}{T}\right)^2. \tag{15}$$

The a, b and c coefficients were calculated by the least squares method. The results of the viscosity measurements are listed in Table I.

Table I

The coefficients of the temperature dependence of viscosity for melts in the system $CaO — FeO — Fe_2O_3 — SiO_2 . \log \eta = a + b\frac{1}{T} + c\left(\frac{1}{T}\right)^2$; T (K); η (Pa . s)

Sample	x (CaO)	$x(Fe_xO_y)$	$x(SiO_2)$	a	$b \times 10^{-4}$K	$c \times 10^{-7}K^2$	$\sigma \times 10^3$	T/K
1	0.500	—	0.500	1.384	—1.485	2.068	3.4	1710—1900
2	0.472	0.056	0.472	1.164	—1.324	1.788	5.8	1720—1890
3	0.444	0.111	0.444	—1.184	—0.534	1.063	9.4	1610—1870
4	0.412	0.176	0.412	1.760	—1.578	1.893	6.5	1560—1830
5	0.517	0.129	0.353	—1.288	—0.581	1.073	14.2	1650—1890
6	0.383	0.096	0.521	2.660	—1.843	2.257	5.2	1620—1880
7	0.312	0.078	0.610	1.242	—1.322	1.892	4.1	1660—1890

RESULTS AND DISCUSSION

The viscosity of melts in the system $CaO—FeO—Fe_2O_3—SiO_2$ was found to increase with increasing SiO_2 content in the melt and with decreasing temperature. The melt viscosity was generally decreased by additions of iron oxides (Fig. 2).

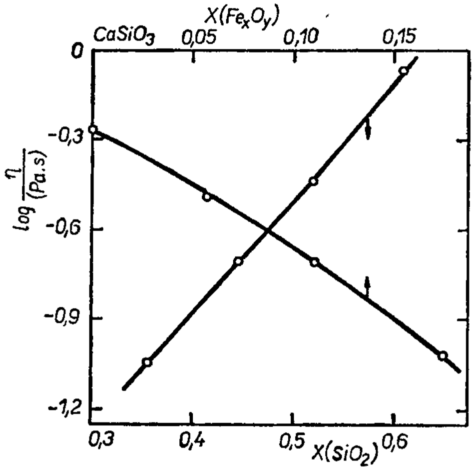

Fig. 2. Viscosity vs. composition for the melts in the system $CaO—Fe_xO_y—SiO_2$ *at 1723 K.*

This indicates that both calcium and iron cations reduce the polymerization degree of the silicate anions. To asses quantitatively this effect, the mean mole mass and the mean number of tetrahedra in the polyanions was calculated for the melts investigated. Such calculation was carried out for melts in the system $CaO-MgO-SiO_2$ in [16].

To calculate the mean mole mass of anions it is neccessary to know the equilibrium constants of the polymerization reactions. The literature [9] specifies the values of the dimerization constants in some binary systems $MeO-SiO_2$ (Me = Ca, Mn, Pb, Fe, Co, Ni). Balta and Balta [17] found a linear dependence between the logarithm of the equilibrium constant and the ionization potential of the metal cation. The values published in [9] were accepted for the calculation: for the system $CaO-SiO_2$, $K = 0.0016$, for the system $FeO-SiO_2$, $K = 0.7$ and for the system $Fe_2O_3-SiO_2$; the value $K = 20$ was accepted on the basis of the third ionisation potential of Fe. The temperature dependence of the equilibrium constant was not considered in the calculation.

In the four-component system $CaO-FeO-Fe_2O_3-SiO_2$, the equilibrium constant was calculated from the additive contributions of the logarithms of equilibrium constants in the boundary systems according to the equation

$$\log K = \frac{x(\mathrm{CaO})}{\Sigma\, x(\mathrm{MeO})} \log K(C - S) + \frac{x(\mathrm{FeO})}{\Sigma\, x(\mathrm{MeO})} \log K(F^2 - S) + \\ + \frac{x(\mathrm{Fe_2O_3})}{x(\mathrm{MeO})} \log K(F^3 - S), \tag{16}$$

where $\Sigma\, x\,\mathrm{MeO} = x(\mathrm{CaO}) + x(\mathrm{FeO}) + x(\mathrm{Fe_2O_3})$. The further procedure was as follows: The value of the mean mole mass of the silicate anions and the mean number of the SiO_4 tetrahedra in the particle were calculated according to equations (6, 7, 8 and 11). In view of the conditions under which the equations had been derived for the calculation, the required molar fraction values had to be converted into the system $CaO-FeO-Fe_{0'67}O-SiO_2$. The results obtained are listed in Table II for 1723 K; however, they were also calculated for 1823 K. The maximum number of tetrahedra in the anion employed in the calculation of distributions for the individual mixtures was limited by the relationship $\Sigma\, nx_n \doteq x(\mathrm{SiO_2})$. Table II indicates

Table II

Equilibrium composition of melt, equilibrium constant of the polymeration reaction, the mean number of tetrahedra and the mean mole mass of polyanions and melt viscosity at 1723 K

Sample	$x(\mathrm{CaO})$	$x(\mathrm{FeO})$	$x(\mathrm{Fe_{2}/_{3}O})$	$x(\mathrm{SiO_2})$	K	$\bar{n}$	$\frac{M}{\mathrm{g/mol}}$	$\frac{\eta}{\mathrm{Pa\,.\,s}}$	$n_{\max}$	$\Sigma\, nx_n$
1	0.500	—	—	0.500	0.0016	5.3	362	0.539	50	0.4998
2	0.432	0.009	0.126	0.432	0.0040	2.9	218	0.320	40	0.4320
3	0.375	0.016	0.233	0.375	0.0094	1.9	159	0.197	30	0.3750
4	0.319	0.023	0.339	0.319	0.0225	1.7	142	0.095	30	0.3190
5	0.417	0.007	0.290	0.285	0.0100	1.2	110	0.090	20	0.2850
6	0.334	0.019	0.193	0.454	0.0090	3.8	271	0.366	70	0.4540
7	0.283	0.021	0.148	0.547	0.0086	9.2	588	0.870	150	0.5470

that the mean chain length as well as the mean mole mass of the silicate anions increases with increasing SiO_2 concentration. The decrease of the mean mole mass is suppressed only at higher concentrations of ferric oxide (cf. samples 1—4). A linear dependence of dynamic viscosity on the calculated value of the mean mole mass of the silicate anions was established. This relationship is plotted in Fig. 3. The slope of this relationship is an exponential function of the reciprocal value of thermodynamic temperature. The dependence of viscosity on the mean mole mass of silicate anions and on temperature was described by the euqation

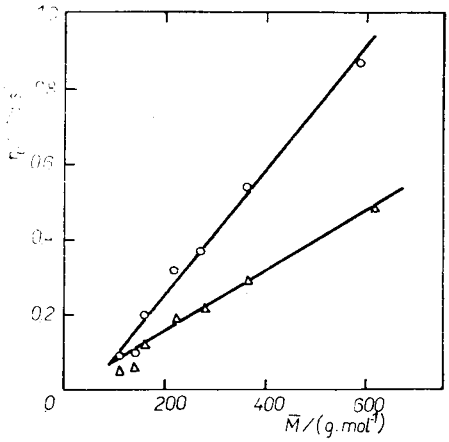

Fig. 3. Viscosity vs. mean mole mass of anions in melts of the system $CaO—FeO—Fe_2O_3—SiO_2$ *at 1723 K* (○) *and 1823 K* (△).

$$\frac{\eta}{\text{Pa . s}} = 2.881 \times 10^{-9}\left(\frac{\bar{M}}{g/\text{mol}} - 92\right)\exp\left(\frac{2.293 \times 10^4 K}{T}\right) + 0.05, \qquad (17)$$

where the standard deviation of the viscosity values measured $\sigma = 9 \times 10^{-3}$ Pa . s. On comparing this equation with a similar equation obtained for the system $CaO—MgO—SiO_2$ in [16] one sees a very satisfactory agreement in the value of the preexponential term (2.285×10^{-9} in [16]), as well as in that of the constant in the exponent (2.349×10^4 in [16]), which implies a general character of this relationship and its general validity for silicate melts. The conclusions that could be drawn from the calculation results are similar to those formulated in study [16]:

1. The structural units, i.e. the isolated silicate polyanions, are in fact units of flow.
2. Sterically, the silicate polyanions are obviously similar and to a considerable extent also isometric.
3. The melts in the system $CaO—FeO—Fe_2O_3—SiO_2$, at least to a content of up to 61 mole % SiO_2, are composed of isolated silicate anions, so that no gel is yet formed at this SiO_2 concentration.

Of considerable interest is the effect of the chemical nature of the cations present on viscosity, and thus also on the anionic structure of the melts. In view of the viscosity decreasing with increasing content of iron oxides, the assumption on the entry of the Fe^{3+} cations into the polyanionic network, and its participation in the formation of globular anions was not fully proved. However, in spite of this the tetrahedral coordination of at least some of the Fe^{3+} cations in the melt is on the whole logical and follows from the ratio of the ionic radii of ferric ions and oxygen ions. The ferric cations may then be present in the melt as isolated FeO_4^{5-} tetrahedra, twinned $Fe_2O_5^{4-}$ tetrahedra, or participate in the structure of anions with a low number of SiO_4^{4-} or FeO_4^{5-} tetrahedra; on the one hand, they thus acquire the character of complex anions, but on the other do not contribute to increasing the melt viscosity. Their content in the melt decreases with increasing temperature, producing a higher content of the larger purely silicate polyanions. This temperature-activated process has the result of increasing the mean mole mass of the anions with increasing temperature. This explanation of the behaviour of the Fe^{3+} cation in silicate melts is in agreement with the conception put forward by Waff [18] and with the results of Mössbauer's spectra measurements in [19—21], where complex compounds of the type $Ca_{0\cdot 5}Fe^{3+}O_2$ with a tetrahedral coordination of the ferric cation were considered.

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VISKOZITA TAVENÍN SÚSTAVY $CaO—FeO—Fe_2O_3—SiO_2$

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Pomocou vysokoteplotného oscilačného viskozimetra sa stanovila viskozita tavenín sústavy $CaO—FeO—Fe_2O_3—SiO_2$ vo vzdušnej atmosfére v teplotnej oblasti 1560—1900 K. Zistilo sa, že viskozita sledovaných tavenín klesá so stúpajúcim obsahom oxidu vápenatého a oxidov železa. Pomocou známeho rovnovážného zloženia tavenín sa pomocou štatistického modelu Pretnara vypočítali hodnoty strednej mólovej hmotnosti kremičitanových polyaniónov. Viskozita sledovaných tavenín je lineárnou funkciou strednej mólovej hmotnosti polyaniónov a exponenciálnou funkciou prevrátenej hodnoty termodynamickej teploty. Koordinácia atómov železa v tavenine je vo všeobecnosti nízka. Potvrdila sa účasť Fe^{3+} katiónov

na tvorbe polyaniónovej siete. Možno predpokladať prítomnosť komplexných aniónov typu FeO_4^{5-}, resp. $Fe_2O_5^{4-}$. Ich podiel so vzrastajúcou teplotou klesá, pričom vzniká väčší podiel objemnejších kremičitanových polyaniónov. Fe^{3+} katión tak preberá funkciu modifikátora tetraédrickej siete. Dôsledkom tohto deja je zvyšovanie strednej mólovej hmotnosti polyaniónov s rastúcou teplotou.

Obr. 1. Znázornenie vybraných rezov v sústave $CaO—Fe_2O_3—SiO_2$. *1—7: figuratívne body naváženého zloženia sledovaných vzoriek; A—B: priebeh zmeny zloženia trosky počas skujňovania (15).*

Obr. 2. Závislosť viskozity od zloženia sledovaných tavenín sústavy $CaO—Fe_xO_y—SiO_2$ *pri teplote 1723 K.*

Obr. 3. Závislosť viskozity od strednej mólovej hmotnosti aniónov v taveninách sústavy $CaO—FeO—Fe_2O_3—SiO_2$ *pri teplotách 1723 K* (o) *a 1823 K* (△).

ВЯЗКОСТЬ РАСПЛАВОВ СИСТЕМЫ $CaO—FeO—Fe_2O_3—SiO_2$

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С помощью высокотемпературного осцилляционного вискозиметра устанавливали вязкость расплавов системы $CaO—FeO—Fe_2O_3—SiO_2$ в атмосфере воздуха в температурной области 1 560—1 900 К. Было установлено, что вязкость исследуемых расплавов понижается с растущим содержанием оксида кальция и оксидов железа. На основании известного равновесного состава расплавов с помощью статистической модели Претнара расчитывали величины среднего молярного веса силикатных полианионов. Вязкость исследуемых расплавов является линейной функцией среднего молярного веса полианионов и показательной функцией обратной величины термодинамической температуры. Координация атомов железа в расплаве в общем низка. Прямое участие Fe^{3+} катионов в образовании полианионовой сетки непотвердилось. Можно предполагать присутствие комплексных анионов типа FeO_4^{5-} или $Fe_2O_5^{4-}$. Их доля с растущей температурой понижается, причем возникает большая доля более объемных силикатных полианионов. Таким образом Fe^{3+} катион берет на себя функцию модификатора тетраэдрической сетки. Результатом приводимого действия является повышение среднего молярного веса полианионов в зависимости от растущей температуры.

Рис. 1. Изображение подобранных разрезов в системе $CaO—Fe_2O_3—SiO_2$: *1—7 — фигуративные точки подаваемого состава проб, A—B — ход изменения состава шлака во время передела (15).*

Рис. 2. Зависимость вязкости от состава исследуемых расплавов системы $CaO—Fe_xO_y—SiO_2$ *при температуре 1 723 К.*

Рис. 3. Зависимость вязкости от среднего молярного веса анионов в расплавах системы $CaO—FeO—Fe_2O_3—SiO_2$ *при температурах 1 723 К* (o) *и 1 823 К* (△).

CARBON '86, Mezinárodní konference o uhlíku, organizovaná pracovní skupinou „Uhlík" Německé keramické společnosti, se bude konat 30. 6. až 4. 7. 1986 v Baden-Badenu, NSR. Původní příspěvky budou zaměřeny do tematických okruhů:

Uhlíkové prekursory a mezofáze
Výroba uhlíku
Uhlíková vlákna a kompozitní materiály
Mechanické a fysikální vlastnosti
Povrchové vlastnosti a aktivní uhlí
Reaktivita
Metody studia
Aplikace.

Zájemci o konferenci se mohou obrátit na prof. Dr. H. P. Boehma, Institut für anorganische Chemie der Universität München, Meiserstrasse 1, D-8000 München, od něhož obdrží další informace.

Bartuška