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Fundamentals of High Temperature Processes

An experimental study on the kinetics of fluidized bed iron ore reduction

A.HABERMANN et al.

To optimize existing iron ore reduction processes or to develop new ones, it is necessary to know the reduction kinetics of the iron ore of interest under the relevant operating conditions. In this work the reduction kinetics of hematite fine iron ore was studied for industrial-scale processes using the fluidized bed technology. Especially designed batch tests were performed in a laboratory-scale fluidized bed reactor fluidized with H_2, H_2O, CO, CO_2, N_2 at atmospheric and elevated pressures to simulate the relevant process conditions. To obtain the reduction rates and the degree of reduction, the concentrations of H_2O, CO, and CO_2 in the outlet gas were analyzed by FT-IR spectroscopy.

Preliminary reduction tests showed a strong effect of the sample weight on the reduction rates, especially in the early stages of reduction. The optimum sample weight was determined by partly replacing the hematite with silica sand. Additionally, the silica sand provided a constant and stable flow pattern throughout the reduction tests. The effects of temperature, gas composition, particle size and pressure on the rates of reduction were tested and discussed.

Rate analysis showed the existence of two phases with different rates during the reduction tests. Additional investigations (microscope analysis, SEM) demonstrated that in the first phase the rates were controlled by mass transport in the gas phase and in the second phase by the reduction process within the small grains of the iron ore particles.

(cf. *ISIJ Int.*, **40** (2000), 935)

Solubility of chlorine in aluminosilicate slag systems

T.HIROSUMI et al.

Thermodynamic properties of chlorine in the $CaO–SiO_2–Al_2O_3$, $Na_2O–SiO_2–Al_2O_3$ and $CaO–SiO_2–Al_2O_3–Na_2O$ slags have been investigated in the present study. The experiments were carried out using gas-slag equilibrium, controlling both P_{O_2} and P_{Cl_2} simultaneously ($P_{O_2}=10^{-16}–10^{-19}$ atm, $P_{Cl_2}=10^{-10}–10^{-13}$ atm at 1 673–1 748 K), and the solubility of chlorine in the 40mass%CaO–40mass%SiO_2–20mass%Al_2O_3 slag was found to vary in proportion to $P_{O_2}^{-1/4}$ and $P_{Cl_2}^{1/2}$. Accordingly, the chloride capacity (C_{Cl^-}), which represents the ability of slags to absorb chlorine, has been defined as;

$$C_{Cl^-}=\frac{(\text{mass\%}Cl^-)P_{O_2}^{1/4}}{P_{Cl_2}^{1/2}}$$

The C_{Cl^-} values were observed to increase with increasing slag basicity and temperature, and showed a reasonable relationship with that of $C_{S^{2-}}$.

(cf. *ISIJ Int.*, **40** (2000), 943)

Rupture phenomena of molten alkali silicate thin films

Y.SASAKI et al.

The rupture length of the iron bearing alkali silicate thin films (the $K_2O·2SiO_2$, $K_2O·2SiO_2$ and $Na_2O·2SiO_2–Fe_2O_3$ systems) are measured as a function of the silica content, the Fe_2O_3 content, temperature, and the drawn out rate. In these melts, it is found that the rupture length, L, depends on the drawn-out rate, v, at the high drawn-out rate conditions, but is independent from the drawn-out rate at the low drawn-out conditions. For all the systems of $K_2O–SiO_2$ and $Na_2O–SiO_2–Fe_2O_3$, the rupture length (L) at the high drawn out rate conditions is found to be proportional to the $-2/3$ of the power of the drawn-out rate (v),

$$L \propto (v)^{-2/3}$$

The rupture length at the low drawn-out rate strongly depends on the SiO_2 and Fe_2O_3 contents, but not temperature and drawn-out rate. From these results, it is confirmed that the rupture length at the low drawn-out rate is determined by the Si–O bond strength of the silicate tetrahedral units.

(cf. *ISIJ Int.*, **40** (2000), 949)

Behavior of slag foaming caused by blowing gas in molten slags

K.Wu et al.

The relationship between the height of foaming slag and blowing gas flow rate has been investigated at different temperature and with additives such as coal, coke, graphite and CaO, in order to understand the foaming phenomenon in most metallurgical processes comprehensively. On the basis of experimental results, the regressed foam behavior equations ($\Delta h=b\cdot V^m$) were obtained. Those correlation coefficients were in range from 0.995 to 0.999. It means that the foam behavior equation can be used to describe foaming ability of the slag foaming caused by blowing gas quantitatively. The foaming index Σ is only a limited case for of the foam behavior equation and can be used only at high temperature and without additives for the foaming phenomenon caused by blowing gas. It was found also that the large carbonaceous particles could decrease the height of foaming slag, however the fine carbonaceous and CaO powder could increase it. The basicity of the slag affects the height of foaming slag.

(cf. *ISIJ Int.*, **40** (2000), 954)

Observation of molten slag surface under gas impingement by X-ray computed tomography

T.SHIMADA et al.

Observation of molten slag at 1 673 K was carried out by X-ray Computed Tomography (X-ray CT) to make clear the shape of slag surface where gas was downward blown. X-ray CT scanning of molten slag heated in an electric furnace was conducted to take an image of its cross section. In order to examine the effects of gas momentum supplied on the surface shape, three kinds of inert gases, Ar, He and N_2, were employed as the blowing gas under the several conditions of gas flow rate and gas pipe diameter.

The obtained X-ray CT images successfully visualized the cross section of molten slag. The boundary line between gas and slag in the image was concave under the gas blowing, indicating a formation of depression on the slag surface by gas impingement. The concave boundary is completely different from the parabolic shape conventionally evaluated. The depression became larger, not only in depth but also in width, with increasing gas flow rate, whereas drastically smaller with increasing gas pipe diameter. In addition, larger depth was brought by impingement of larger molecular (atomic)-weight gas. These results were well explained by the conservation law of momentum between gas and slag. It was also revealed that the increment in the boundary line caused by formation of depression, which corresponds to an increase in the interfacial area, is in proportion to the depression depth in the range of more than 2 mm, and was independent of the pipe diameter and gas species.

(cf. *ISIJ Int.*, **40** (2000), 958)

Assessment of P_2O_5 activity coefficients in molten slags

E.T.TURKDOGAN

From a critical review of the existing experimental data, the free energy equations are presented. A new estimate is made of the standard free energy of formation of the hypothetical pure liquid P_2O_5, which is used in calculating the P_2O_5 activity coefficients from the experimental data of independent studies of slag–metal reactions. It is found that in low P_2O_5 slags, $\log(\gamma_{P_2O_5})$ is a linear function of CaO from 0 to 60%, independent of temperature. Whereas, in slags containing $P_2O_5>10\%$, $\log(\gamma_{P_2O_5})$ is a linear function of CaO content>40% only and increases with an increasing temperature. A novel concept of a slag–P_2O_5 emf sensor is presented for experimental evaluation.

(cf. *ISIJ Int.*, **40** (2000), 964)

Casting and Solidification

Experimental study of the transition from constrained to unconstrained growth during directional solidification

Ch.-A.GANDIN

Temperature measurements are carried out in 99.99 wt% aluminium and aluminium–silicon alloys. The experimental apparatus was initially built for the study of microporosity formation in aluminium alloys.[1] The construction is designed to obtain upward directional solidification by limiting lateral heat flow during cooling and suppressing fluid flow induced by the pouring sequence. Cooling occurs from the top part of the ingot, leading to the formation of a surface dendrite layer. In the 99.99 wt% aluminium, very few equiaxed grains sink down from the surface dendrite layer into the liquid. The density of the equiaxed dendritic grains is too low to block the columnar cellular front. Cooling curves show that, once superheat disappears (*i.e.*, when no substantial thermal gradient remains in the liquid), the liquid is kept at an almost constant temperature during the growth of the columnar front and a small negative thermal gradient forms in the liquid ahead of the growing columnar front. It is concluded that the liquid is reheated by the growing columnar front. In the case of the aluminium–silicon alloys, a columnar-to-equiaxed transition (CET) is observed

at almost two-thirds of the ingot length. The columnar length is found to increase slightly with decreasing the solute content. Recalescence is measured in a fully equiaxed region, while cooling rate, recorded by the thermocouple located just above the CET, remains negative.

(cf. *ISIJ Int.*, **40** (2000), 971)

The electroslag remelting of high-speed steel using a magnetic field

M.MURGAŠ et al.

The electroslag remelting process was studied when the consumable electrode made from the powder of M2 type high-speed steel was used and the effect of outside magnetic field was applied. The electromagnetic forces that arise from the interaction between the outside direct magnetic field and the one-phase electric current of the electroslag remelting process by a monofilar scheme alter the mechanism of the electrode remelting and thus, affect the solidification of a high-speed steel and its structure. The cast cutting tips made from ingots produced by this technology had tool life to be comparable to that of standard ones made from the wrought steel of the identical chemical composition and heat treatment.

It has been shown that a magnetic field also affects both the temperature ranges and the kinetics of phase transformation in a high-speed steel. This suggestion is proved by DTA measurements.

(cf. *ISIJ Int.*, **40** (2000), 980)

Prediction of density of stainless steel

H.MIZUKAMI et al.

The change of the density of stainless steel with phase at temperature range from 773 K to 1973 K has been studied by a sessile drop profile method. Measurement of the density by a sessile drop profile method has to be carried out under heating conditions to avoid the influence both of undercooling and the shrinkage within the sample during solidification. Accuracy of experimental results was evaluated in comparison with previous studies for pure iron. The density of stainless steel was dependent on the phase but not chromium and nickel contents. Equations for prediction of the density in L, δ and γ phase regions were determined using the experimental results of

$\rho_L=-7.20\times10^{-4}\Delta T_L+7.04$
$\rho_\delta=2.87\times10^{-4}\Delta T_\delta+7.27$
$\rho_\gamma=4.40\times10^{-4}\Delta T_\gamma+7.43$
18mass%≦Cr≦25mass%, 4mass%≦Ni≦25mass%,
773 K≦T≦1 973 K

The density of δ and γ coexisting phase ($\delta+\gamma$) is then predicted from the following equation.

$\rho_{(\delta+\gamma)}=\rho_\delta\cdot f_\delta+\rho_\gamma\cdot f_\gamma$
18mass%≦Cr≦25mass%, 4 mass%≦Ni≦25mass%,
773K≦T≦1 973 K

These estimated values are in good agreement with experimental results.

(cf. *ISIJ Int.*, **40** (2000), 987)

Instrumentation, Control and System Engineering

Robust multivariable control for hot strip mills

G.HEARNS et al.

A multivariable regulator for a hot strip mill is developed which is robustly stable for changes to the mill modulus while still providing high strip thickness accuracy. This is in contrast to the conventional automatic gauge control which has to trade of nominal performance to provide robust stability. The performance and stability off the multivariable $\mathcal{H}_\infty$ controller and the conventional controller are analysed and the benefits of the multivariable controller is demonstrated using a nonlinear simulation.

(cf. *ISIJ Int.*, **40** (2000), 995)

Modelling for control of a steckel hot rolling mill

E.SCHOLTZ et al.

In this work the derivation of a nonlinear plant simulator of a Steckel Hot Rolling Mill process is shown. The simulator reflects the thickness crown and tension behaviour of the strip, while the temperature, shape and flatness behaviour of the strip fall outside the scope of this work. Roll gap, stand, tension and hydraulic actuator models are incorporated in order to yield the simulator. The nonlinear simulator is used to identify a linear model for control system design. The linear model was identified at a certain operating point associated with a particular pass of a multiple pass rolling schedule. Step tests were applied to the manipulated variables of the simulator, and using system identification (SID) techniques a linear time invariant (LTI) multivariable input output (MIMO) transfer function model was identified. Lastly an initial control problem is formulated.

(cf. *ISIJ Int.*, **40** (2000), 1003)

Forming Processing and Thermomechanical Treatment

FE-based on-line model for the prediction of roll force and roll power in hot strip rolling

W.J.KWAK et al.

Investigated *via* a series of finite element process simulation is the effect of diverse process variables on some selected non-dimensional parameters characterizing the thermo-mechanical behavior of the strip in hot strip rolling. Then, on the basis of these parameters an on-line model is derived for the precise prediction of roll force and roll power. The prediction accuracy of the proposed model is examined through comparison with predictions from a finite element process model.

(cf. *ISIJ Int.*, **40** (2000), 1013)

Basic examination on strip wandering in processing plants

T.MASUI et al.

In strip processing plants, strip passing through long distance at high speed is particularly important to produce high quality products. If the strip wanders in the line because of strip shape, thermal effect or other factors, stable plant operation is disturbed.

This report is concerned with strip lateral wandering.

The cause of strip wandering is clarified by using a model processing plant. Strip camber and welded joint angle, that is a non-straight form of the strip, mainly gives rise to wandering. The effects of misalignment on the equipment are small.

Tension leveler that corrects not only shape defects but also camber and welded joint angle is very effective to depress wandering.

Pinch roll to prevent strip wandering, the hearth roll and the quenching roll to prevent a thermal crown have been newly developed to hold good strip tracking.

(cf. *ISIJ Int.*, **40** (2000), 1019)

Surface Treatment and Corrosion

Structure of electrodeposited Zn–Mn alloy coatings

Y.TSUCHIYA et al.

Structure of electrodeposited Zn–Mn alloys which are one of the corrosion-resistant zinc alloy coatings suitable for automotive body panels was investigated using X-ray diffraction, SEM and SIMS.

The coatings are composed of fine grains or granular grains which contain fine particles, depending on the Mn content. The coatings have an uniform depth distribution of Mn and Zn.

X-ray diffraction revealed that the coatings have either single or dual alloy phases which are the following phases, namely η phase, Γ phase, ε phase, γ-Mn phase and α-Mn phase, depending on the Mn content. The ε phase and γ-Mn phase as high temperature phases exist when the range of the Mn content is above 20 mass%. Both phases are stable when heated at 170°C. The γ-Mn phase is transformed to the β-Mn phase and the α-Mn phase by heating beyond 250°C, although the ε phase is unchanged.

(cf. *ISIJ Int.*, **40** (2000), 1024)

Functionally graded coating for steels by reaction diffusion using $FeAl_3$ powder

Y.TSUCHIDA et al.

The powder liquid coating method has successfully been applied to a commercially available pure iron and a carbon steel (JIS-S45C) using $FeAl_3$ powder. The coated layer is found well graded in terms of microstructure, chemical compositions and hardness, so that the coated layer is much stronger for thermal shock than that made by a conventional aluminizing method. Changes in the graded microstructure during coating procedures are studied; it is noted that an $FeAl/FeAl_2$ eutectoid structure formed in the coated layer shows 927HV and relatively high toughness. Although the present coating technique needs to heat a sample up to an elevated temperature as high as 1 473 K, the prior austenite grain size of the carbon steel was refined by so-called Grange method, without accompanying any serious damages in the coated layer.

(cf. *ISIJ Int.*, **40** (2000), 1029)

Transformations and Microstructures

The modeling of the grain growth in a continuous reheating process of a low carbon Si–Mn bearing TRIP steel

S.JIAO et al.

Making use of Anelli's idea and an own model for isothermal grain growth, a new model for the grain growth during a continuous reheating process was deduced. This model makes use of the real three dimensional grain diameter instead of the mean linear intercept distance as a measure for the grain size. Two series of experiments have been undertaken: in the first series, the validity of the model for several intermediate temperatures has been investigated, while in the second series, the influence of the heating rate on the validity of the model has been verified. In both cases a quite reasonable matching between the prediction of the model and the experimentally determined three dimensional grain size has been found.

(cf. *ISIJ Int.*, **40** (2000), 1035)

New Materials and Processes

Microstructural control for superplasticity simply by heat treatment without thermomechanical processing in a Ti–46Al–3.5Cr alloy

K.NIINOBE et al.

Microstructural evolution only by heat treatment has been studied for a Ti–46at%Al–3.5at%Cr alloy, in order to obtain a microstructure which causes superplasticity at high temperatures. By changing the cooling rate from 1 613 K in the α-Ti single-phase region, three kinds of microstructures were identified. Namely, lamellar microstructure appeared by furnace cooling, feathery microstructure took place by air-cooling and massive microstructure prevailed by oil quenching. During subsequent annealing at 1 473 K in the $\beta+\gamma$ two-phase region, the feathery microstructure turns to fine microdual structure with the equiaxed γ grains with 13 μm in grain size and the β precipitates formed along the γ grain boundaries. In a tensile test at 1 473 K with a strain rate of $3.2\times10^{-4}\,s^{-1}$, this β/γ microdual structure exhibits remarkable superplastic deformation with an elongation of 450%, which is the same with that obtained by the β/γ microdual structure prepared by a thermomechanical processing. On the other hand, the lamellar microstructure and the massive microstructure are not transformed to the β/γ microdual structure with the equiaxed γ grains, resulting in low elongations of 30% and 110% at 1 473 K, respectively.

(cf. *ISIJ Int.*, **40** (2000), 1041)