（＊掲載記事題目及び掲載頁は変更になる場合があります。）

Fundamentals of High Temperature Processes

Characteristics of inclusions generated during Al–Mg complex deoxidation of molten steel

R.TAKATA et al.

The complex deoxidation of molten steel with magnesium vapor produced *in-situ* by aluminothermic reduction of magnesium oxide and aluminum wires was carried out. The Mg vapor was directly injected into molten steel with Ar carrier gas from an immersion tube.

With injection of magnesium vapor into aluminum killed molten steel, the total oxygen concentration was further decreased due to magnesium deoxidation and removal of inclusions by rising bubbles. The final total oxygen concentration could decrease to 15 ppm. SEM observation and EDS analysis show that the periphery around an Al_2O_3 inclusion particle was changed into spinel. This is beneficial for prevention of aggregation of inclusions. The inclusion size in the melt was decreased with time, and the inclusions remaining in the ingot contained mainly MgO and spinel.

In the case of magnesium deoxidation followed by addition of aluminum wires, the inclusion size in the melt was also decreased with time. Therefore, in the present experiments, the larger inclusions were easily removed from the melt by floating up with the rising bubbles, while the smaller ones tended to remain in the melt.

(cf. *ISIJ Int.*, **47** (2007), 1379)

Mechanism of carbothermic reduction of chromium oxide

T.MORI et al.

The isothermal experiment of carbothermic reduction of chromium oxide was carried out. The reduction rate increased by increasing temperature, using a moderate carrier gas flow rate and the fine graphite particles.

XRD analysis revealed that Cr_3C_2, Cr_7C_3, $Cr_{23}C_6$ and Cr were formed in turn as the reaction products at 1 673 K. The relation among the products, reduction temperature and time was summarized from the XRD results at the temperature range from 1 373 to 1 673 K. It is confirmed that the reduction contains various kinds of reactions, such as direct reduction, indirect reduction of chromium oxide, the carbon solution loss reaction and the reaction of chromium carbide with carbon dioxide. At the initial stage of reduction, the direct reduction dominates the reduction rate. At the later stage of reduction, because of the chromium carbide layer formed on the surface of graphite particles and rather slow reduction rate, the rate-controlling step is deduced to be CO and CO_2 gas diffusion through the layer of reduction products.

(cf. *ISIJ Int.*, **47** (2007), 1387)

Mechanism of carbothermic reduction of hematite in hematite–carbon composite pellets

J.YANG et al.

Isothermal reduction experiments are carried out to study carbothermic reduction of hematite in hematite–graphite composite pellets. The carbothermic reduction is dominated by the direct reduction at the initial stage of reduction, and it is determined by the indirect reduction together with the carbon solution loss reaction when the CO partial pressure exceeds a certain value, which is dependent on the temperature. The reduction is greatly promoted due to the increment of the contact area between the hematite and graphite particles and enhancement of heat conduction in the hematite–graphite composite pellets.

The indirect reduction together with the carbon solution loss reaction is markedly activated with increasing temperature. At a moderate carrier gas flow rate, the carbothermic reduction rate reaches the highest value. The reduction can also be effectively improved with decreasing the graphite particle size.

The relationship of reduction products, heating temperature and holding time has been obtained from the XRD analysis results at the different stages for various temperatures under the present experimental conditions. The SEM observation indicates that the indirect reduction together with the carbon solution loss reaction should dominate the carbothermic reduction of hematite at the later stage of reduction.

(cf. *ISIJ Int.*, **47** (2007), 1394)

Effect of lime particle size on melting behavior of lime-containing flux

J.YANG et al.

By use of an infrared-ray vacuum furnace, effect of CaO particle size on melting rate of lime-containing flux is studied in the present work. Two kinds of CaO particles of the average diameters of 10 μm and 75 μm are used. Four kinds of lime-containing fluxes are employed, which are $CaO–SiO_2$, CaO–FeO, $CaO–SiO_2–Al_2O_3$ as ironmaking slag and $CaO–SiO_2$–FeO as steelmaking slag.

The relationships of reaction products, heating temperature and holding time are obtained for the fluxes of $CaO–SiO_2$ and CaO–FeO under the present experimental conditions. As for the flux melting, decreasing the lime particle size can shorten the holding time, but can not decrease the heating temperature. In the melting process of $CaO–SiO_2$, a layer of $2CaO \cdot SiO_2$ is observed on the periphery of CaO particle, which tends to inhibit the melting of CaO particles. Decreasing the CaO particle size can also effectively promote the dissolution of CaO particles into both kinds of slag of $CaO–SiO_2–Al_2O_3$ and $CaO–SiO_2$–FeO.

(cf. *ISIJ Int.*, **47** (2007), 1401)

A model for estimating viscosities of aluminosilicate melts containing alkali oxides

M.NAKAMOTO et al.

A model to estimate the viscosity of aluminosilicate melts, including alkali oxides, was constructed in the present study. The model is based on an earlier model, and considers the bonding state of oxygen in molten silicate and the flow mechanism of melts with a network structure. The straightforward method of Susa *et al.* to evaluate bonding states of oxygen in chemical compositions was applied to the present model since their method can easily be extended to any multi-component system. The calculation results of our model can express the dependence on viscosity in quaternary aluminosilicates containing alkali oxide and their subsystems.

(cf. *ISIJ Int.*, **47** (2007), 1409)

Application of 28 GHz microwave irradiation to oxidation of ilmenite ore for new rutile extraction process

S.ITOH et al.

Microwave heating has been applied to the oxidation in the new rutile extraction process developed by the authors in which rutile is extracted from a natural ilmenite ore by oxidation and magnetic separation followed by leaching with diluted acid. Since ilmenite $FeTiO_3$ and pseudobrookite Fe_2TiO_5 strongly absorb 28 GHz microwave, Australian ilmenite ore mainly composed of $FeTiO_3$ was rapidly heated up to about 1 273 K and oxidized in air with keeping almost constant temperature. As a result, two equilibrium phases of rutile TiO_2 and pseudobrookite were quickly formed at microwave power of 1.5 kW. The growth rate of the pseudobrookite phase in the microwave irradiation was found to be much faster than that of the rutile phase in the conventional resistance furnace, indicating a drastic enhancement of the growth rate by the microwave irradiation.

(cf. *ISIJ Int.*, **47** (2007), 1416)

Ironmaking

Coke oven carbon deposits growth and their burning off

V.ZYMLA et al.

Burning off is the main method of carbon deposit removal from coke oven chambers. Optimisation of this operation is of a great interest both from the point of view of current production loss reduction and of coke oven plant lifetime extension. In this study the deposits originating from different parts of chambers have been characterised by chemical analysis, optical microscopy, XRD, Raman spectroscopy and mercury porosimetry. This investigation has shown that deposits contain two carbonaceous phases: the char from pyrolysed carry over coal particles and the pyrolytic carbon from raw gas cracking.

Furthermore, the monitoring method for carbon deposits growth in coke oven chamber is presented. The statistical analysis of the visual inspection data has been a useful tool in the evaluation of the rate of deposit growth in individual coke oven chambers.

The combustion kinetics of carbon deposits were studied by thermogravimetry under non-isothermal conditions with natural air draft and under isothermal conditions at controlled air flow rate. It was found that the reactivity of carbon deposits is in-between metallurgical coke and electrographite reactivity. At the temperatures below 700°C, under laboratory conditions, the combustion rate (on the basis of external surface) is controlled by surface reaction at higher temperature and activation energy is around is 170 kJ/mol. At temperature higher than 700°C combustion is controlled by gas film diffusion.

Modelling of combustion in thermobalance experiments and in industrial coke oven chamber was

carried out on the basis of measurement of kinetics using commercial CFD software.

(cf. *ISIJ Int.*, **47** (2007), 1422)

Casting and Solidification

Free dendrite growth of Fe–0.5mass%C alloy–Three-dimensional phase-field simulation and LKT model–

K.OGUCHI et al.

The free dendrite growth of iron–0.5mass% carbon alloy has been investigated using a three-dimensional phase-field model with thin-interface-limit parameters. The dendrite growth velocity and the tip radius of curvature are determined at different degrees of undercooling, and then the stability parameter for the alloy is estimated. The dendrite growth velocities obtained by phase-field simulations are compared with those obtained using the LKT model with the stability parameter of 0.05. The two are in good agreement and the validity of the LKT model is discussed with regard to practical applications.

(cf. *ISIJ Int.*, **47** (2007), 1432)

Design of swirling flow submerged entry nozzles for optimal head consumption between tundish and mold

Y.TSUKAGUCHI et al.

A technology of swirling flow formation in a submerged entry nozzle is proposed as an effective measure for controlling the flow pattern in a continuous casting mold. As a result of a joint study between Nippon Institute of Technology, Osaka University, Kyushu Refractories, and Sumitomo Metal Industries, swirling flow submerged entry nozzles with a swirl blade for steel slab casting have been developed to improve the productivity of the process and the surface quality of slabs and coils.

The swirl blade in the submerged entry nozzle forms the swirling flow expending the potential energy between the tundish and the mold. Thus, the swirl blade and the internal shape of the submerged entry nozzle, which determine the swirling flow rate, should be designed considering the flow rate in the nozzle and the head between the tundish and the mold of the applied casting machine.

From full-scale water model experiments, we obtained an empirical equation to estimate the required head between the tundish and the mold from the flow rate, the internal shape of the nozzle, and the specification of the swirl blade. Then, we investigated energy efficiency to create the swirling flow, using an empirical equation from the results of a water model experiment and a numerical analysis of the swirling flow in the nozzle.

The obtained empirical equation is valuable to design swirling flow submerged entry nozzles adopted for various casting conditions.

(cf. *ISIJ Int.*, **47** (2007), 1436)

Instrumentation, Control and System Engineering

Adaptive feed-forward automatic gauge control in hot strip finishing mill

Y.K.LEE et al.

The conventional roll force automatic gauge control (RFAGC) adjusts the roll gap to reduce the delivery thickness variation in the hot strip finishing mills. In this process, the information about the roll force is used to control the roll gap indirectly because the strip gauge cannot be measured owing to insufficient space in the mill and the hostile environment. Moreover, there exists an uncertainty in thickness due to roll eccentricity. Therefore, a small gain is preferable in avoiding product defects when this uncertainty is taken into account in RFAGC. However, in such a case, partially uncontrolled variation still remains on delivery thickness because of insufficient control input. To overcome this drawback, a feed-forward controller (FFAGC) was developed.[12] This scheme adjusts the roll gap to reduce variations in the current stand by using an estimated uncontrolled delivery thickness variation of the previous stand. Although this FFAGC has an advantage over RFAGC, it still has a problem: The FFAGC cannot cope with the continuous variation of system parameters and circumstance because the existing feed-forward controller has been designed as a low pass filter with constant weights. In other words, desirable control performance in the automatic gauge control system cannot be obtained owing to a phase delay and filtering errors.

To overcome the problem, it is essential to estimate the long-term thickness variation exactly in the hard environment, to select a proper gain and to synchronize the feed-forward control input under a time varying speed of the gauge control process. In this paper, an enhanced adaptive filtering structure is proposed to improve the controller performance. And the relation between the gains of RFAGC and FFAGC are discussed to select a proper gain. The proposed controller can readily be implemented on the conventional control system, improving the performance of the system without additional cost. This improvement was demonstrated by using the real coil data which are collected under the proposed control scheme in POSCO.

(cf. *ISIJ Int.*, **47** (2007), 1444)

Chemical and Physical Analysis

Characterization of different solid particles transformed from green rust in aqueous solution—Using XRD, Mössbauer spectroscopy, and XANES

K.INOUE et al.

The X-ray diffraction method and Mössbauer spectroscopy were used for characterizing solid particles transformed from hydroxysulfate GR2(SO_4^{2-}) by oxidation in aqueous solution. The results revealed that different ferric oxyhydroxides (α-FeOOH and γ-FeOOH) and magnetite (Fe_3O_4) were formed through the precipitation of ferrous and ferric ions dissolved from GR2(SO_4^{2-}) in aqueous solution. The X-ray absorption spectroscopy along with Mössbauer spectroscopy was also used for analyzing the chemical state of iron in GR2(SO_4^{2-}). The molar ratio of ferrous to ferric ions [Fe(II)]/[Fe(III)] in GR2(SO_4^{2-}) was shown to be nearly two during the transformation from GR2(SO_4^{2-}). Moreover, the oxidation-reduction potential (ORP) and pH of the GR suspension were measured to examine formation conditions of the different ferric oxyhydroxides and magnetite. The results showed that formation conditions of magnetite in the aqueous solution were different from those of the ferric oxyhydroxides, indicating that reaction conditions in aqueous solution strongly influence the formation of different ferric oxyhydroxides and magnetite.

(cf. *ISIJ Int.*, **47** (2007), 1452)

Sensitivity of conventional and non-destructive characterization techniques to recovery and recrystallization

M.OYARZÁBAL et al.

The sensitivity and limitations of magnetic coercive field, hardness, metallographic measurements and Electron Back Scattered Diffraction analysis are studied and discussed in terms of monitoring recovery and the onset and evolution of recrystallization in a cold rolled low carbon steel. The magnetic coercive field shows much higher resolution than hardness measurements when measuring recovery. Hardness is almost insensitive to the microstructural changes taking place in the material during annealing treatments performed at low temperatures. The conditions under which the magnetic coercive field measurements can also be used to monitor recrystallized fraction are also discussed.

(cf. *ISIJ Int.*, **47** (2007), 1458)

Forming Processing and Thermomechanical Treatment

Restoration processes during hot deformation in the δ-ferrite and austenite dual phase region of AISI430 ferritic stainless steel

J.S.HINTON et al.

The restoration mechanisms of AISI430 ferritic stainless steel under industrial rolling conditions (strain 0.6, strain rate $16\,s^{-1}$) were studied experimentally. Dynamic recovery followed by static recrystallisation and concurrent static recovery were observed in the ferrite phase at the hot rolling temperature (960°C). Complete recrystallisation (>70%) was hindered by the impingement of the new grains on the widespread γ phase and the extensive recovery. At lower strain rates, typical of nominal laboratory experiments, restoration through a more continuous process was observed, where recovery mechanisms were promoted by the longer deformation time. An explanation of the microstructural evolution of this material under hot rolling conditions and the interaction of these mechanisms is presented.

(cf. *ISIJ Int.*, **47** (2007), 1465)

Combined macro–micro modeling for rolling force and microstructure evolution to produce fine grain hot strip in tandem hot strip rolling

T.MORIMOTO et al.

T.M.C.P. (Thermo Mechanical Control Process) has been widely used in the steel industry. We have produced fine grain hot strips industrially through high reduction and low temperature asymmetric rolling which decreases rolling force by generating a cross shear zone.

We needed combined macro–micro model to predict rolling force in the finishing train and microstructure evolution of fine grain hot strips. First, deformation analysis model, an asymmetric rolling theory based on numerical analysis using Orowan's theory was proposed. Second, to obtain the material data for the kinetics of microstructure change in the low temperature austenite region, the compression tests were performed and we measured recrystallization ratio by EBSD (Electron back scattering diffraction). Third, microstructure evolution model to formulate the ferrite transformation using the intragranular nucleation was introduced.

Accuracy of the new combined macro–micro model for rolling force and microstructure evolution was very excellent and it enabled us to confirm dynamic ferrite transformation occurrence during high reduction low temperature rolling at the actual tandem hot strip mill.

(cf. *ISIJ Int.*, **47** (2007), 1475)

Welding and Joining

Numerical modeling of pulse MIG welding

A.MANDAL et al.

A new heat source model for pulse MIG welding is developed which enables estimation of weld bead width and central penetration obtained with industrially pure Argon, Argon+5%, 25% CO_2. The heat source model takes into account effects of various pulse current parameters such as pulse peak time, base time and mean current. Finite difference method with explicit time stepping technique is used to solve the two dimension Fourier's equation on non-uniform meshes generated by K-Method. Enthalpy method is used along with Kirchhoff variable transformation to solve the moving boundary problem. Relationship between enthalpy and temperature is used to include non-linear thermal properties of the material. The computed values of weld bead width and central penetration are within 8% and 5% of the experimental values respectively.

(cf. *ISIJ Int.*, **47** (2007), 1485)

Surface Treatment and Corrosion

Microstructure and growth kinetics of nitrided-zone in plasma-nitrided Fe–Cr alloys

G.MIYAMOTO et al.

Microstructure and growth kinetics of the nitrided zone have been examined in various Fe–Cr binary alloys ranging from 1 to 30 mass% Cr that had been plasma-nitrided at temperatures between 743 and 943 K. CrN precipitates form under all the nitriding conditions. With increasing Cr content or decreasing nitriding temperature, the morphology of CrN precipitates changes drastically from continuous precipitation with a fine disk-shaped morphology to CrN lamellar precipitation. In addition, small amounts of Cr_2N form when the Cr content and the nitriding temperature are high. In spite of the different manners of Cr–nitride precipitation, the thickness of the nitrided zone is approximately proportional to the square root of the nitriding period. Furthermore, in most of the alloys the activation energies for the overall growth kinetics of the nitrided zone are nearly constant.

(cf. *ISIJ Int.*, **47** (2007), 1491)

Potential and current flow of double steel bars in concrete by 2-D BEM

J.-W.KYUNG et al.

This study performs the numerical analysis of electric potentials and currents in reinforced concrete to achieve the nondestructive evaluation of corrosion using a half-cell potential measurement and polarization resistance method. Electric fields around single and double reinforcing steel bars were analyzed using a 2-D Boundary Element Method (BEM). In the half-cell potential measurement, the potential distribution in double reinforcing steel bars in concrete was represented as a singular shape rather than a circle shape. It means that the potential value inside concrete and current ratio in rebar are closely related to the size and the number of rebars. In the polarization resistance method, the polarized area of rebar is associated with the current density and current ratio.

(cf. *ISIJ Int.*, **47** (2007), 1497)

Effect of steel composition on iron dissolution in molten zinc and development of Fe–Zn phases on steel surface

R.MISHRA et al.

The effect of steel composition on iron solubility in molten zinc and coating microstructure was studied by immersing test coupons of four different steel grades in zinc bath for 20 min at 470°C. The amount of iron dissolved in the molten zinc depends upon the steel composition. The coating was characterized by energy dispersive spectroscopy, X-ray diffraction and galvanostatic methods. The coating consists of mainly ζ (zeta) and δ (delta) phases along with zinc layer at the top, irrespective of the steel composition. The presence of a thin layer of gamma phase has also been confirmed by XRD and galvanostatic analysis. However, the proportion and distribution of the coating phases varies with the steel chemistry.

(cf. *ISIJ Int.*, **47** (2007), 1504)

Effect of coating thickness on formability of hot-dip galvanized low carbon steel sheet

M.R.TOROGHINEJAD et al.

In this study, formability of hot-dip galvanized low carbon steel sheets has been evaluated. The effect of coating thickness on formability has been analyzed by using galvanized sheets with five different coating weights and a comparison has been made with an uncoated sheet. The crystallographic orientation of the coatings was determined using X-ray diffraction and texture parameters were calculated. Mechanical properties of samples were determined by uniaxial tensile tests and formability of the sheets was evaluated using forming limit diagrams (FLD). From the experimental results, it was concluded that by increasing the coating thickness, the texture parameter of basal planes component was decreased. Such variations caused the yield strength of the coated sheet to increase, but ductility was reduced. The overall evaluation of the results indicated that the formability of galvanized steel with thinner coating thickness is better than uncoated and other samples with thicker coatings.

(cf. *ISIJ Int.*, **47** (2007), 1510)

Transformations and Microstructures

Evaluation of the hot ductility of a C–Mn steel produced from scrap recycling

J.CALVO et al.

The use of scrap for the production of steel has been related to a high incidence of transverse cracking during continuous casting. This phenomenon is linked to the high contents on residual elements and impurities present in recycled steels. In order to understand the embrittlement mechanisms that can take place at high temperatures, the hot ductility of a C–Mn steel with residual contents of Cu and Sn was analyzed. The steel was evaluated in two as-received conditions, *i.e.* as-cast and as-rolled, and samples were subjected to different reheating and *in-situ* melting cycles prior to tensile tests. Testing temperatures ranged from 650 to 1 100°C and strain rate was $5 \cdot 10^{-3} s^{-1}$. Results show that the width and depth of ductility troughs depend on both the reheating cycle and the as-received condition of the material. According to the fractographical analysis, a reheating of the samples at 1 100°C leads to a combined intergranular and interdendritic fracture while samples reheated at 1 330°C exhibit mainly intergranular features at temperatures in the ductility trough. The interdendritic brittleness for samples reheated at the low temperature is related to the microsegretation taking place during solidification as it can be demonstrated with samples *in-situ* melted. On the other hand, intergranular brittleness is related to the segregation of impurities and residuals to grain boundaries. The recovery of the ductility in the high temperature range is associated to the appearance of D-REX. The differences observed between the as-cast and as-rolled as-received condition of the steel are associated to differences in the austenite grain size and its effect on dynamic recrystallization.

(cf. *ISIJ Int.*, **47** (2007), 1518)

Variant selection of reversed austenite in lath martensite

N.NAKADA et al.

The microstructure of partially reversed lath martensite in 13%Cr–6%Ni steel was examined by electron backscatter diffraction, and the crystallo-

graphic character of the reversed austenite is discussed in relation to the mechanism of 'austenite memory'. Most of the reversed austenite grains had the same orientation as the original austenite matrix before martensitic transformation. However, some austenite grains had a different orientation in a twin relationship to the other major austenite grains, although all the reversed austenite grains retained a Kurdjumov–Sachs relationship to the martensite matrix. On the basis of the crystallographic relationships among the habit plane, the close packed direction of austenite and the martensite lath boundary, we suggest that the austenite variants are theoretically limited to two kinds within one packet and five kinds within one original austenite grain. In addition, we found that internal stress introduced by martensitic transformation plays an important role in determining the austenite variant: internal stress operates so that reversed austenite selects the same variant as that present in the original austenite matrix before martensitic transformation. This phenomenon is understood as the austenite memory.

(cf. *ISIJ Int.*, **47** (2007), 1527)

Physical Properties

Viscosity measurements of some mould flux slags

M.PERSSON et al.

Continuous casting has been the dominating process for steel casting over the past decades. During the process, mould fluxes are added to enable a smooth functioning of the process, enabling better process performance and products with less defects. The viscosity of the mould flux slag is a key parameter determining the optimum casting conditions.

Several experimental studies have earlier been carried out in order to determine viscosity data for mould flux slags, both industrial ones as well as synthetic slags with compositions close to industrial mould fluxes. However, the continuous evolving of new steel grades, casting dimensions and product quality in the steel industry also demands better control and development of the mould fluxes. In industrial practice for clean steel production, the Al_2O_3 pick up has generally been observed to be about 2–4%. In view of this, the present study was initiated to experimentally investigate the viscosity of mould fluxes used in Swedish steel industry and the effect of dissolution of alumina in the same. The industrial implications of the slag viscosities measured in the present work are discussed.

Viscosities of mould fluxes for continuous casting in steel production have been measured by the rotating cylinder method. Seven industrial mould fluxes, with different compositions, used were included in the study. The effect of the Al_2O_3 content in the mould fluxes was also investigated. Even relatively small additions of Al_2O_3 show a significant increase in viscosity. The measurements were carried out in the temperature range of 1 373 to 1 673 K.

(cf. *ISIJ Int.*, **47** (2007), 1533)

Social and Environmental Engineering

Separation and recovery of phosphorus from steelmaking slags with the aid of a strong magnetic field

K.YOKOYAMA et al.

In order to make sure the great potential of steelmaking slag as a new phosphorus resource, domestic phosphorus material flow in Japan including iron and steel industry has been investigated based on the statistical data on 2002. It has been demonstrated that phosphorus in the steelmaking slag is almost equivalent with that in the imported phosphate rock in the view points of the amount and the concentration. Phosphorus exists mainly in the form of calcium-phosphate or its solid solution with calcium-silicate rather than the Fe_tO rich liquid phase in the slag and exhibits remarkable segregation in the solidified slag. If the strong magnetic field is applied to the crushed slag, precipitated calcium-phosphate solution phase can be separated from Fe_tO matrix phase due to the large difference of each magnetic property. It has also been indicated by the Waste Input–Output model that the phosphorus recovery from steelmaking slag by the new process proposed in the present work has great environmental and economical benefits.

(cf. *ISIJ Int.*, **47** (2007), 1541)