Fundamentals of High Temperature Processes

Modification of bubble-driven liquid metal flows under the influence of a DC magnetic field

C.ZHANG et al.

The paper presents an experimental study devoted to a bubble-driven liquid metal flow under the influence of an external DC magnetic field. Experiments have been performed on laboratory scale at ambient temperature using the ternary alloy GaInSn. Measurements of the bubble-driven liquid metal flow have been carried out using the Ultrasound Doppler Velocimetry (UDV). The magnetic field has been imposed either in vertical direction parallel to the main bubble motion or in horizontal direction, respectively. Whereas a global damping of the flow field was generally observed in the case of the vertical aligned magnetic field, the application of a horizontal magnetic field can provoke a restructuring of the flow pattern with strong, non-steady vortical structures. This finding could attain relevance for metallurgical engineering, for instance the control of the mould flow during the continuous casting of steel by means of an electromagnetic brake.

(cf. *ISIJ Int.*, **47** (2007), 795)

A proposal for evaluation method of energy parameter values in cell model for thermodynamics of refining slag

T.MATSUMIYA et al.

In the cell model, a thermodynamic model of slag, the fractions of heterogeneous cation pairs are calculated which compose cells with an oxygen atom in between by the minimization of free energy of slag with energy parameters. On the other hand, by using NMR the fractions can be measured. Therefore, a new method can be proposed to evaluate the energy parameter values used in the cell model so that the calculated fractions in the slag with its minimum of free energy coincide with the measurement. The possibility of this evaluation method of energy parameter values was examined by the comparison of bridging oxygen/non-bridging oxygen (BO/NBO) ratio calculated by the cell model with energy parameter assessed elsewhere with measured BO/NBO ratio by NMR. The BO/NBO ratios of three SiO_2–Al_2O_3–CaO slags with different basicities calculated by the use of cell model with energy parameter values assessed elsewhere were 42.5/57.5, 45/55 and 70/30, respectively. On the other hand, from ^{17}OMAS spectrum and $^{27}Al \rightarrow {}^{17}O$ CP/MAS spectrum of the same slags fully labeled and calcinated at 1 600 centigrade degree, the ratios of BO/NBO in the slags were determined as 48.5/51.5, 42/58 and 76/24, respectively. This agreement supports the energy parameter values assessed elsewhere and gives a good expectation to the proposed method.

(cf. *ISIJ Int.*, **47** (2007), 802)

Activities of SiO_2 in some CaO–Al_2O_3–SiO_2(–10%MgO) melts with low SiO_2 contents at 1 873 K

Y.J.KANG et al.

The activity of SiO_2 in the CaO–Al_2O_3–SiO_2 (–10%MgO) systems was measured at the region of %SiO_2<10%. The melts were equilibrated with a reference metal of Cu in a controlled oxygen partial pressure at 1 873 K. Iso-SiO_2 activity contours were drawn from the measured values. For the CaO–Al_2O_3–SiO_2 system, the measurement result showed positive deviation from the previous data. It was also found that the SiO_2 activity decreased as CaO content increased. In the CaO–Al_2O_3–SiO_2–10%MgO system, the composition dependence of the SiO_2 activity seemed to be similar as the ternary system. On the other hand, the activity values were much lower than that of the ternary system. It could be attributed to the strong basic behaviour of MgO. Based on the result of the SiO_2 activity measurement, the equilibrium between the inclusions and liquid steel during ladle treatment was discussed.

(cf. *ISIJ Int.*, **47** (2007), 805)

Ironmaking

Selectivity of microwave energy consumption in the reduction of Fe_3O_4 with carbon black in mixed powder

K.ISHIZAKI et al.

In the present work, mixtures of magnetite and carbon black powers were irradiated by a constant microwave power supply of 2.8 kW at 2.45 GHz. The temperature was measured as a function of the irradiation time from 220°C. The progress of the reduction was examined by XRD analyses of reaction products at various temperatures in the range 400–1 200°C. Under the present experimental conditions magnetite selectively absorbs microwave energy below approximately 650°C; meanwhile carbon black absorbs it in the whole temperature range. The reduction of magnetite to wustite was observed to occur from at least a measured temperature of 400°C but did not occur homogenously inside the mixture. At 800°C, the reduction of wustite to iron started and occurred almost homogeneously inside the reacting mixture mass.

A general conclusion of this work is that microwaves are absorbed and used in their path within the irradiated mass by the material of highest absorbing microwave power.

(cf. *ISIJ Int.*, **47** (2007), 810)

Localized heating and reduction of magnetite ore with coal in composite pellets using microwave irradiation

K.ISHIZAKI et al.

Magnetite iron ore-coal composite pellets were microwave irradiated in N_2 atmosphere with a power supply gradually increased in 2 min intervals from 0.2 up to 2 kW at 2.45 GHz. Electron probe microanalysis (EPMA), X-ray image of SEM micrographs, back scattered electron image (BSE) and Fe, O, S and C mappings of composite pellets irradiated up to different temperatures were obtained. Under the present experimental conditions, pellets heated up to about 800°C without reduction. Above this temperature the reduction occurred stepwise; Fe_3O_4 reduced to FeO between about 800°C and 1 000°C and then FeO reduced to Fe from about 1 000 to 1 250°C average temperatures. The measured temperature appears to reasonable represent the average temperature of the pellet. However inside of the reacting mass localized slightly different temperatures may exist. Point carbon contents of reduced iron inside the composite pellet irradiated up to 1 150°C were from almost 0% to 2% with no correlation between with their spatial location inside the pellet. It is concluded that carbon in the microwave irradiated pellet acting as a reducing and heating agent at the same time generates localized reduction microenvironments.

(cf. *ISIJ Int.*, **47** (2007), 816)

Some fundamental aspects of highly reactive iron coke production

S.NOMURA et al.

It is important to develop the production and utilization technology of highly reactive coke in order to improve the efficiency of blast furnace reactions. In this study, some fundamental aspects of highly reactive iron coke produced in a coke oven chamber were investigated. First, the effects of catalytic Fe powder addition to coal before carbonization on coke strength were investigated. The addition of Fe powder decreased the coal caking property and hence the resultant coke drum index (DI^{150}_{15}). On the other hand it increased coke reactivity (JIS coke reactivity index and CRI) to a great extent. This means that the caking property of blended coals needs to be adjusted higher to produce iron coke with proper strength and high reactivity. Secondly, it was shown that the iron ore reacts with silica brick at 1 200°C in a condition similar to that in a coke oven chamber. The iron ore and silica reacted to produce fayalite ($2FeO \cdot SiO_2$) and the brick was damaged. On the other hand, it was proven that the iron ore does not react with silica brick at 1 100°C in the above condition. Based on this fundamental study, iron coke with proper strength and high reactivity was successfully produced in a coke oven chamber on a commercial scale by adjusting the coal blend composition and the coke oven temperature. Furthermore it was revealed that about 70% of iron in iron ore powder added to coal was reduced to metallic iron during carbonization.

(cf. *ISIJ Int.*, **47** (2007), 822)

Post-reaction strength of catalyst-added highly reactive coke

S.NOMURA et al.

The development of production and utilization technology for highly reactive coke is significant in order to improve blast furnace reaction efficiency. In this report, the post-reaction strength of catalyst-added highly reactive coke was investigated. The reaction between coke and CO_2 was stopped at a weight loss of 20% and the reaction temperature was adjusted so that the reaction lasted for a constant period. In this experimental condition, the reaction temperature of highly reactive coke was lower than that of normal coke, which corresponds to the decrease in the thermal reserve zone temperature in a blast furnace. First, a decrease in reaction temperature made the reaction of the catalyst added coke more homogeneous, which increased the post-reac-

tion strength of the highly reactive coke produced by the post-addition of catalyst to coke method; however, it decreased that of the highly reactive coke produced by the pre-addition of catalyst to coke method. Secondly, the post-reaction strength of catalyst-added highly reactive coke produced by the post-addition of catalyst to coke method became equal to or greater than that of normal coke. The types of catalysts and the catalyst adding method affect the porosity distribution in the coke after reaction and hence the post-reaction strength of coke to a great extent. Suitable selection of catalysts and its addition method to coke leads to highly reactive coke with post-reaction strength greater than that of normal coke. Catalyst-added highly reactive coke is a promising material to improve blast furnace reaction efficiency.

(cf. *ISIJ Int.*, **47** (2007), 830)

Casting and Solidification

Fluid flow behavior in submerged entry nozzle of continuous casting

T.KATO et al.

A fluid flow control of molten steel in continuous casting mold directly leads to surface and internal quality of slab and product. However, few studies have looked at fluid flow in submerged entry nozzle even when it is a source of flow in the mold. In this study, fluid flow in submerged entry nozzle is *in-situ* observed through transparent immersion nozzle by a fusible alloy model and a steel flow model. Subsequently, argon gas extract from mold meniscus is also measured at commercial caster.

In the fusible alloy model experiment, both potential flow and plug flow emerged depending on argon flow rate, metal flow rate and nozzle diameter. Decreasing argon flow rate, increasing metal flow rate and reducing nozzle diameter leads to a rise of meniscus height in nozzle.

Molten steel flow was observed through transparent silica glass nozzle. Both potential-flow and plug-flow also emerged depending on argon flow rate.

According to measurement of net argon flow rate through mold meniscus, about 20% of argon gas injected from upper sliding plate is brought into nozzle at molten steel system.

Taking these experimental results into consideration, it is concluded that in conventional slab caster, submerged entry nozzle must be filled with molten steel and steel flow must be like plug-flow during usual operation.

(cf. *ISIJ Int.*, **47** (2007), 839)

Solidification of iron and steel on single-crystal oxide

T.SUZUKI et al.

The effect of oxides as an agent of ferrite solidification of iron and steel was investigated by measuring melt undercooling for solidification on single-crystal oxide substrates. Four kinds of single-crystal oxide plates, Al_2O_3(0001), Al_2O_3(11-20), MgO(100), MgO(111), were employed. Poly-crystal oxide substrates of Al_2O_3, MgO, Ti_2O_3 and V_2O_3 were also used for comparison. Undercooling for solidification of Ti-free C–Mn steel was dependent on the kind of oxide used, and seemed to increase with increasing the lattice misfit between the oxide and delta-ferrite, the primary phase to solidify. On the other hand, undercooling for the solidification of 1% Ti-added C–Mn steel was not so affected by the kind of oxide, and was lower than that of Ti-free steel solidified on the same oxide substrate. Titanium oxide or titanium-enriched oxide was formed at the interface between Ti-added steel and oxide substrates used, and this is expected to be the reason of decrease in undercooling. The undercooling of Ti-free steel on poly-crystal Ti_2O_3 substrate also supported this. Steel-oxide interfacial energy was estimated based on the undercooling measurements, and the result suggests that the interfacial energy between delta-ferrite and titanium oxide should be much smaller than that between the ferrite and other oxides, being close to metal–metal interfacial energy rather than metal-oxide interfacial energy. This metallic characteristic of titanium oxide is thought to be the primary cause to enhance the ferrite nucleation at the interface.

(cf. *ISIJ Int.*, **47** (2007), 846)

Chemical and Physical Analysis

Chemical analysis of zinc electroplating solutions by X-ray fluorescence spectrometry

S.-M.JUNG et al.

A quantitative analysis method used to analyze chlorine, iron and zinc in electroplating solutions, using X-ray spectrometry in atmospheric He mode, is proposed. The present research concerns the replacement of the conventional analyses of electroplating solutions with rapid and reproducible quantification using X-ray fluorescence spectrometer. An in-depth investigation conducted in the present study identifies the species present in the real electroplating solutions. XRD patterns and semi-quantitative results for the electroplating solutions show synthetic standards based on the compositional range of solutions by analyzing the electroplating solutions obtained in real processes. 28 calibration standard solutions are prepared by diluting liquid standard solutions certified by titration and ICP-OES analyses used to construct the XRF calibration curves for Cl, Fe and Zn. The suggested method showed satisfactory precision and accuracy in the analysis of electroplating solutions. The present study provides evidences that the proposed XRF spectrometry could be an alternative analytical method to replace the conventional techniques by comparing the uncertainties estimated for each method.

(cf. *ISIJ Int.*, **47** (2007), 852)

Forming Processing and Thermomechanical Treatment

Effect of Mo on dynamic recrystallization of Nb–Mo microalloyed steels

B.PEREDA et al.

The influence of the Mo addition on the dynamic recrystallization behavior of Nb microalloyed steels was studied. The initial austenite grain size, the amount of microalloying elements in solid solution and the deformation conditions (temperature and strain rate) affect dynamic recrystallization kinetics of both Nb and Nb–Mo steels. Continuous torsion tests were carried out to characterize the dynamic recrystallization behavior of the microalloyed austenite, after reheating the specimens at different temperatures between 1 100 and 1 460°C, this brought a wide range of initial grain sizes, from 22 to 805 μm. It was observed that decreasing the values of the Zener–Hollomon parameter and of the initial grain size promotes dynamic recrystallization. Mo in solid solution produces a large retardation effect in the dynamic recrystallization and brings higher values in the characteristic critical, ε_c, and peak, ε_p, strains, being this effect independent of the Mo content. A corrective factor has been applied to quantify the retardation produced by Mo in solid solution. This means that it was possible to propose a unique relationship to predict the peak strain for Nb, Nb–Ti and Nb–Mo steels.

(cf. *ISIJ Int.*, **47** (2007), 859)

Welding and Joining

Microstructural analysis of infrared brazed Ti–6Al–4V and Nb using the Ti–15Cu–25Ni foil

D.W.LIAW et al.

Microstructural analysis of infrared brazed Ti–6Al–4V and Nb at 970°C using the clad Ti–15Cu–25Ni filler metal has been performed in the experiment. For the 180 s brazed specimen, at first primary Ti_2Cu, Ti_2Ni as well as eutectic microstructure are obtained upon cooling cycle of brazing, and maximum amounts of Ti_2Cu and Ti_2Ni are observed at the brazed joint. The amount of transient Ti_2Cu and Ti_2Ni intermetallics is decreased with increasing the brazing time due to depletion of Cu and Ni from the braze into the Ti–6Al–4V substrate. Diffusion of Cu and Ni into Ti–6Al–4V substrate results in isothermal solidification rather than eutectic solidification of the molten braze, and the region of transformed β-Ti is broadened as the brazing time increased to 600 s and 1 200 s. For the 3 600 s brazed specimen, Ti_2Cu, Ti_2Ni and transformed β-Ti are vanished from the infrared brazed joint. Disappearance of Ti_2Cu and Ti_2Ni intermetallics for the longer brazing time makes Ti–15Cu–25Ni filler metal with a great potential in future applications.

(cf. *ISIJ Int.*, **47** (2007), 868)

Surface Treatment and Corrosion

Service life estimation of concrete structures reinforced with Cr-bearing rebars under macrocell corrosion conditions induced by cracking in cover concrete

S.-H.TAE et al.

Cracked concrete members reinforced with Cr-bearing rebars were assumed for the purpose of developing Cr-bearing rebars having the required corrosion resistance in macrocell corrosion environments induced by cracking in cover concrete. The service life of concrete structures reinforced with

Cr-bearing rebars was then estimated based on a macrocell corrosion rate model, and their Cr content to achieve a service life of 100 years was calculated. As a result, the service life of concrete reinforced with Cr-bearing rebars was found to increase as their Cr content increased regardless of the corrosive environment type. The calculation also revealed that Cr contents of 16% or more and 13% or more would lead to a service life of over 100 years in harsh and moderate chloride attack zones, respectively. In a carbonation zone, the Cr content to achieve a service life of over 100 years was calculated to be 9% or more.

(cf. *ISIJ Int.*, **47** (2007), 874)

Transformations and Microstructures

Effect of alloying additions on the SFE, Neél temperature and shape memory effect in Fe–Mn–Si-based alloys

N.E.STANFORD et al.

A range of Fe–Mn–Si-based shape memory alloys has been investigated to examine the interplay of composition, stacking fault probability (SFP) and Neél temperature on the shape memory effect (SME). It has been found that the SFP (inversely proportional to stacking fault energy) showed little correlation to the SME for the range of alloy compositions examined. Further, the Neél temperature was not found to exhibit a significant effect on the SME. The addition of interstitial elements, however, was found to markedly decrease the SME.

(cf. *ISIJ Int.*, **47** (2007), 882)

Grain boundary characteristics of isolated grains in conventional grain oriented silicon steel

T.KUMANO et al.

During the secondary recrystallization of GO, isolated grains are often observed in secondary-recrystallized Goss grains (matrix). In an extraction experiment, the crystallographic relations between single isolated grains and matrix grains respectively were investigated in the case of CGO. Although the $\Sigma 5$ grain boundary to the Goss orientation was the most frequent in the primary recrystallization texture, no $\Sigma 5$ boundary could be observed at the onset temperature of secondary recrystallization; hence it was concluded that the $\Sigma 5$ boundary was most mobile in this case. On the other hand, in the case of HGO, it has already been reported that the $\Sigma 9$ is most mobile. The reason why the dominant grain boundary may differ for the same Goss orientation could be the temperature dependency of the grain boundary features, *i.e.*, mobility and/or grain boundary energy. Furthermore, the $\Sigma 3$ was more mobile than GGB (general grain boundary) at the higher temperature, which corresponded to the lower temperature range of HGO.

(cf. *ISIJ Int.*, **47** (2007), 889)

Phases equilibrium study in quaternary iron-rich Fe–Al–Mn–C alloys

V.RIGAUD et al.

High aluminium low manganese steels were elaborated in order to study the phase equilibrium in the range of 900–1 100°C. Two different quaternary alloys have been studied (Fe–9 to 10wt%Al–1.7wt%Mn–0.2wt%C and Fe–9 to 10wt%Al–8wt%Mn–0.2wt%C). Equilibrium phase's nature was identified at four temperatures for two alloys. Equilibrium phase fraction and chemical compositions were determined at 900°C. Thanks to these equilibrium results, we tried to understand the non-equilibrium microstructure observed after different thermal histories (DTA cycles, isothermal treatment followed by quench, reception state) and identify DTA events in a coherent way.

(cf. *ISIJ Int.*, **47** (2007), 897)

High temperature flow stress and recrystallization behavior of high-Mn TWIP steels

A.S.HAMADA et al.

The flow stress behavior and recrystallization kinetics in the hot rolling temperature range have been investigated in five Fe–Mn–Al (Mn: 25 wt%, Al: 0–8 wt%) TWIP steels by compression testing on a Gleeble simulator. Results were compared with corresponding properties of carbon and austenitic stainless steels. Microstructures were examined by electron microscopy. The results show that the flow stress level of the TWIP steels is considerably higher than that of low-carbon steels and depended on the Al concentration close to 6 wt%, while the structure is austenitic at hot rolling temperatures. At higher Al contents, the flow stress level becomes significantly lowered due to the presence of ferrite. The static recrystallization kinetics is slower compared to that of carbon steels, but it is faster than typical of Nb-microalloyed or austenitic stainless steels. High Mn content is a reason for the high flow stress as well as for slow softening. Al has a minor role only, but in the case of austenitic–ferritic structure, softening of the ferrite phase occurs very rapidly that also contributes to overall faster softening. The grain size is effectively refined by the dynamic and static recrystallization processes.

(cf. *ISIJ Int.*, **47** (2007), 906)

Mechanical Properties

Effect of intergranular ferrite on hydrogen delayed fracture resistance of ultrahigh strength boron-added steel

J.S.KIM et al.

The effect of intergranular ferrite (IGF) on hydrogen delayed fracture of an ultrahigh strength boron added steel with yield strength over 1 GPa was investigated. For this purpose, a series of constant loading tests and thermal desorption analyses was conducted on the hydrogen precharged steel having different volume fractions of IGF, 0%, 6%, and 10%. Tensile strength of unnotched and notched specimens slightly decreased only by 4% with increasing the volume fraction of IGF up to 10%. Time to failure of the steel in the constant loading tests became prolonged with increasing the volume fraction of IGF. Microstructural observation informed that the presence of IGF at prior austenite grain boundaries suppressed the precipitation of film-like carbides, which act as the susceptible crack nucleation sites, at these boundaries. In addition, IGF was very effective on retarding crack propagation. The thermal desorption analyses revealed that hydrogen was trapped mainly at grain boundaries and that the critical hydrogen content causing delayed fracture increased with increasing the volume fraction of IGF, resulting in better hydrogen delayed fracture resistance. The present results shed light on the fact that the presence of IGF is beneficial for improving hydrogen delayed fracture resistance of the steel with little degradation of strength.

(cf. *ISIJ Int.*, **47** (2007), 912)

Numerical simulation and effect of curling on bolted connections in cold-formed stainless steel

T.S.KIM et al.

Finite element (FE) model with three-dimensional solid elements is established for investigating the structural performance of bolted connections loaded in static shear with cold-formed stainless steel (austenitic steel; SUS304) plate, based on the existing test data performed by Kuwamura *et al.* for calibration. From the experimental results, modifications of current Japanese steel design standards for calculating the ultimate strength to account for the mechanical properties of stainless steel and thin-walled steel plates were made. However, curling, *i.e.*, out of plane deformation in connection plate ends with a long edge distance was observed, and led to the strength reduction of bolted connections. In this study, non-linear material and non-geometric analysis are carried out in order to predict the load-displacement curves of bolted connections. Therefore, finite element analysis results are compared with previous experimental results, failure modes and ultimate strengths predicted by recommended procedures of FE method show a good agreement with those of experimental results and it can be also found that curling which occurred in test specimens is also observed in FE model. Failure criterion, which predicts failure mode of bolted connections and curling criterion are proposed considering the stress distribution at the region where initial crack may occur. In addition, influence of curling on the strength reduction is estimated quantitatively and change of in-plane stress of connection plate is investigated according to the advancement of curling.

(cf. *ISIJ Int.*, **47** (2007), 919)

Analysis of buckling and interfacial debonding of galvannealed coating layer on steel substrates under applied tensile strain

S.IWAMOTO et al.

As the hot-dipped galvannealed steels are composed of brittle coating layer with low failure strain and ductile substrate with far higher failure strain. When tensile stress is applied externally on the coated steels, the coating layer exhibits multiple cracking perpendicular to the tensile direction, and then interfacial debonding occurs, following the buckling of the coating layer in the sample width direction. In the present work, the interfacial debonding behavior was observed with the scanning electron microscope and analyzed with 3-dimentional finite element

models, to reveal the influence of the crack spacing on the buckling-induced interfacial debonding. In the analysis, three cases with different crack spacing in the coating layer were used. The results of analysis could account well for the experimentally observed buckling-induced spalling process of the coating layer. Furthermore it was revealed that, the shorter the crack spacing of the coating layer in the tensile direction, the less the interfacial debonding takes place.

(cf. *ISIJ Int.*, **47** (2007), 929)