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Fundamentals of High Temperature Processes

Deoxidation equilibrium of vanadium in the Iron–Nickel melts

V.Y.Dashevskii et al.

Thermodynamic analysis of oxygen solutions in the Fe–Ni melts with vanadium has been carried out. The deoxidizing ability of vanadium decreases slightly with the nickel content up to 20% but it considerably rises with a further increase in its concentration. The oxygen solubility curves pass through a minimum, which shifts to the lower vanadium concentrations with a rise in the nickel content, *i.e.*, from 2.32% for pure iron to 0.77% for pure nickel. The further vanadium addition causers an increase in the oxygen concentration in melt. For pure nickel or alloys with the nickel concentration higher than 60%, the deoxidizing ability of vanadium is lower in comparison with that of manganese and is close to the deoxidizing ability of chromium. This can be explained by the fact that although the bond strength of oxygen with nickel is appreciably weaker as compared to that with iron ($\gamma^{\circ}_{O(Fe)}$= 0.0105; $\gamma^{\circ}_{O(Ni)}$=0.357), but the vanadium bond strength with nickel is much stronger than that with iron ($\gamma^{\circ}_{V(Fe)}$=0.1; $\gamma^{\circ}_{V(Ni)}$=0.011). The deoxidation of iron–nickel melts with vanadium was experimentally studied by the example of the Fe–40%Ni alloy.

(cf. *ISIJ Int.*, **49** (2009), 149)

Sulphide capacities of $CaO–Al_2O_3–SiO_2–MgO–MnO$ slags in the temperature range 1 673–1 773 K

Y.Taniguchi et al.

With a view to estimate the sulphide capacities of slags used in hot metal pretreatment processes, the sulphide capacities of $CaO–Al_2O_3–SiO_2–MgO$, $CaO–Al_2O_3–SiO_2–MnO$ and $CaO–Al_2O_3–SiO_2–MgO–MnO$ slags in low SiO_2 concentration region were measured in the temperature range 1 673–1 773 K. The gas–slag equilibrium technique has been used for these measurements.

From the results obtained, it was found that the MgO and MnO increased the sulphide capacity values of slag. A new empirical model based on optical basicity for sulphide capacity prediction of these slags was developed using the measured values of the present work and literature.

(cf. *ISIJ Int.*, **49** (2009), 156)

Reduction extraction kinetics of titania and iron from an ilmenite by H_2–Ar gas mixtures

Y.Wang et al.

Reduction of an ilmenite concentrate as a natural concentration containing 49.78 mass% TiO_2 and 27.96 mass% total Fe by H_2–Ar gas mixtures was investigated from 1 073 to 1 273 K. Both the reduction degree and reduction rate increased with the increase of temperature and hydrogen content. The reduction degree of the ilmenite decreased due to the presence of impurities including manganese and silicon oxides in the ilmenite concentrate. During the reduction, the formation of manganese and silicon oxide-enriched zones prevented complete reduction of Fe^{2+}. The reduction products were characterized using X-ray diffraction, scanning electronic microscope with energy disperse spectroscopy and optical microscopy analysis, respectively. The main phases in reduced products were reduced iron, rutile, reduced rutiles, pseudobrookite and Ti_3O_5. The reduction reaction proceeded topochemically and the reduction kinetics was discussed. It was found that the rate controlling step was the diffusion of hydrogen gas in the reduced layer.

(cf. *ISIJ Int.*, **49** (2009), 164)

Thermodynamics of copper dissolution into $MnO–SiO_2–MnS$ inclusion system

J.G.Park et al.

Recently, a new process has been introduced to control α–γ transformation of steels using cuprous sulfide precipitates on the MnO bearing oxide inclusion as well as MnS. In order to optimize the composition of inclusions, the solubility of copper into $MnO–SiO_2–MnS$ system were measured at 1 523 K and a dissolution mechanism of copper into an oxy-sulfide melts and cuprous sulfide capacity has been proposed. The solubility of copper increased with an increase in $MnO/(MnO+SiO_2)$ ratio and showed a maximum solubility at MnO and MnS doubly saturated composition, which was consistent with the maximum precipitation ratio of CuS. Deliberating an oxy-sulfide melts saturated with MnS, MnO, and SiO_2 at 1 523 K, the phase diagram for the $MnO–SiO_2–MnS$ system indicated that the activity coefficient of cuprous sulfide in an oxy-sulfide melts was dependent on the activity of MnO and the activity coefficient of MnS. The dissolution mechanism of copper into a MnO–MnS inclusion system was confirmed and cuprous sulfide capacity was also defined from the reaction mechanism. Cuprous sulfide capacity for the MnO–MnS bearing inclusion strongly depends on stability of sulfide and basicity. The experimental results revealed that the optimized composition for inclusion to dissolve copper was (mass% MnO)=43.6, (mass%SiO_2)=1.8 and (mass%MnS)= 54.6. Consequently, it could be proposed that the harmlessness of copper in scrap and dispersion strengthening could be possible by using the non-metallic inclusion technique.

(cf. *ISIJ Int.*, **49** (2009), 171)

Simulation of the impingement of a liquid jet on a molten iron bath by using a particle method

M.Asai et al.

For the development of a kinetic model of the reactions occurring during refining processes, the MPS method that can easily calculate the large deformations in free surfaces and interfaces was used as a simulation tool for a high temperature fluid flow. A two-dimensional simulation of the impingement of a liquid jet on a liquid bath was performed for a slag–metal system, and the interfacial area between a slag and a metal was estimated. The possibilities and problems encountered in the application of the MPS method to the simulation of practical steelmaking processes are discussed.

(cf. *ISIJ Int.*, **49** (2009), 178)

Water model experiments on the effect of an argon bubble on the meniscus near the immersion nozzle

T.Watanabe et al.

Water model experiments have been carried out to understand the behavior of the meniscus of molten steel near the immersion nozzle. The meniscus is disturbed by a large argon bubble rising along the immersion nozzle. Water, silicone oil, and air are used as models for molten steel, mold powder, and argon, respectively. A cylindrical rod is used as a model for the immersion nozzle. The contact angle of a water droplet on the rod is adjusted to become nearly the same as that of a molten steel droplet on the immersion nozzle by coating repellent on the rod surface. A single air bubble of a predetermined volume is released from a cap-shaped container to attach to the bottom of the rod. The behavior of the bubble passing across the interface between water and silicone oil is observed with a high-speed video camera to make clear the entrapment of silicone oil droplets into the water bath.

(cf. *ISIJ Int.*, **49** (2009), 182)

Flooding diagram for multi-phase flow in a moving bed

X.F.Dong et al.

Flooding phenomena typically represent liquid overflow and unstable flow behaviour in a gas–liquid counter-current flow within a fixed bed. Understanding the flooding phenomena in a multi-phase flow system is important to process design and optimization. This paper presents a study of the flooding phenomena in two systems: gas–liquid flow and gas–liquid–powder flow in moving beds. Experiments are conducted in a one-dimensional column involving upward gas and powder flow and downward solids and liquid flow. Based on the experimental findings, a correlation is formulated which considers the effects of particle moving and the presence of powder phase. The correlation can be used to generate flooding diagrams to depict flooding and non-flooding regions for multi-phase flow in a moving bed.

(cf. *ISIJ Int.*, **49** (2009), 189)

Ironmaking

The influence of fuel reactivity on iron ore sintering

R.R.Lovel et al.

An experimental program conducted at CSIRO showed that flame front speed (ffs) and sinter productivity increased with fuel reactivity (r) while sinter strength and fuel utilisation fell. The following relationship modelled flame front speed for a JSM style sinter mix over a fuel reactivity range of 1×10^{-5} g/g/s to 4×10^{-3} g/g/s:

$$ffs=0.2014\cdot\ln(r)+4.039, \quad R^2=0.997$$

where ffs is flame front speed (cm/min) and r is fuel reactivity (g/g/s).

The CSIRO research concluded that rapid heating of the lower bulk density green granules and en-

dothermic fuel gasification preceding the flame front are dominant factors that contribute to changes in flame front speed. While productivity was significantly increased, less time at temperature reduced sinter strength and gasification contributed to poor fuel utilisation under the standard sintering conditions used for the experiments. A range of changes to sinter mixes and sintering conditions are proposed to overcome the negative aspects of increased fuel reactivity while continuing to exploit the positives.

(cf. *ISIJ Int.*, **49** (2009), 195)

Assessment of the state of the blast furnace high temperature region by tuyere core drilling

M.HELLE et al.

Tuyere core drillings give a unique opportunity to probe the blast furnace and detect changes in both physical and chemical conditions of its high-temperature region. In this paper the findings from drill cores taken from a blast furnace are used to characterize the internal state of the furnace hearth, quantified by an erosion model estimating the available hearth volume. The complex relation is studied by entertaining neural network models using different combinations of inputs consisting of the extent of the distinct tuyere-level zones (raceway, bird's nest, dead man, *etc.*) of the core samples. The resulting model can be used to gain knowledge of the relation between tuyere level conditions and hearth states, and to classify the findings from future core drillings. The results also throw light on possible reasons for thermal cycles observed in the hearth of the furnace studied.

(cf. *ISIJ Int.*, **49** (2009), 203)

Steelmaking

Development of fluxed iron oxide pellets strengthened by CO_2 treatment for use in basic oxygen steel making

J.PAL et al.

Lump lime is presently being used as a fluxed material in basic oxygen steel making process. However, problems are often encountered in operation due to its high melting point, poor dissolution property, fines generation and hygroscopic property. Binary CaO–Fe_2O_3 has a eutectic point (1 200°C) with 22% CaO. An agglomerate of lime and iron oxide under BOF condition would thus generate low melting oxidizing slag at the beginning of blow and would facilitate refining process. In this study, lime fluxed iron oxide pellet has been prepared using waste iron oxides and its strength was developed by CO_2/CO_2 containing gas treatment at room temperature, while no separate binders has been used. Industrial waste gases, say, blast furnace gas (BFG) may suitably be used as CO_2 source. The pellet making process parameters such as composition of pellets, grain size of iron ore fines, treatment conditions *etc.* have been optimized through physical characterization like cold compressive strength, drop strength, abrasive resistance *etc.* Up to around 30 kg/pellet cold compressive strength, around 150 drop strength and excellent abrasive property have been achieved.

(cf. *ISIJ Int.*, **49** (2009), 210)

Casting and Solidification

Thermal analysis of the formation of chunky graphite during solidification of heavy-section spheroidal graphite iron parts

J.SERTUCHA et al.

Analysis of cooling curves recorded at the centre of large blocks cast with near-eutectic spheroidal graphite cast irons prone to give chunky graphite has been checked against microstructure observations. It has been observed that solidification proceeds totally at temperatures lower than the stable eutectic temperature and the following solidification sequence could be proposed: 1) nucleation of primary graphite in the liquid; 2) initial eutectic reaction processing by growth of austenite-like dendrites encapsulating the primary nodules; 3) bulk eutectic reaction related to nucleation and then growth of CHG cells and of secondary nodules, these latter giving spheroidal graphite eutectic cells. It was found that the maximum recalescence during the eutectic reaction first increases with the volume of the block affected by chunky graphite, and then decreases when most of the material is affected. Interestingly enough, a relationship between the volume of the blocks affected by CHG and the recalescence measured on TA cups has been observed.

(cf. *ISIJ Int.*, **49** (2009), 220)

Nitride precipitation on particles in Fe–10mass%Ni alloy deoxidized with Ti, *M* (*M*=Mg, Zr and Ce) and Ti/*M*

A.V.KARASEV et al.

The precipitation of nitrides on deoxidation particles in an Fe–10mass%Ni alloy containing 30~130 ppm O and 14~22 ppm N has been studied in the experiments where an alloy was deoxidized with Ti, *M* (*M*=Mg, Zr and Ce) and Ti/*M* at 1 600°C and then cooled to 1 200°C, followed by holding for 0 and 60 min before quenching. The total number of particles above 0.1 μm remains almost constant except in Ti deoxidation, but the proportion of cubic particles which correspond to nitrides and oxide-nitrides increases during holding at 1 200°C in Ti, Zr, Ti/Mg and Ti/Zr deoxidations. In 0.05% (initial amount) Ti deoxidation, the number of TiN and TiN+TiO_x particles increases during holding. In 0.03%Ti/0.1%Mg deoxidation, the number of TiO_x–TiN particles decreases and the number of MgO–TiN and MgO–Ti(O, N) particles increases during holding due to the reduction of TiO_x with soluble Mg. In 0.03%Ti/0.04%Zr deoxidation, the number of ZrO_2 particles significantly decreases during holding due to the precipitation of TiN and ZrN on Zr oxides and formation of Zr(O, N)–Ti(O, N) particles. In the cases of Ti/0.08%Zr and Ti/0.12%Ce deoxidations, most of particles at 0 and 60 min of holding are ZrO_2 and Zr(O, N) and Ce_2O_3 oxides, respectively, because the FeO–TiO_x oxide in particles is reduced completely by dissolved Zr and Ce.

(cf. *ISIJ Int.*, **49** (2009), 229)

Forming Processing and Thermomechanical Treatment

Outflow characteristics of a pressure medium during sheet hydroforming

T.HAMA et al.

The outflow characteristics of a pressure medium during a sheet hydroforming process have been studied experimentally by directly measuring the hydraulic pressure distribution. Initial measurements of the outflow of the pressure medium though the gap between two clamped dies were carried out in order to verify the method for measuring the hydraulic pressure distribution employed in this study and to investigate the basic properties of the outflow. The upward forces were calculated by integrating the measured hydraulic pressure during the outflow and found to be in satisfactory balance with the clamping forces, which demonstrates the validity of this measurement method. Furthermore, we suggest that the critical outflow pressure can be predicted by considering the force equilibrium. An experimental investigation of a square-cup sheet-hydroforming process was then carried out. The upward forces were calculated by integrating the measured hydraulic pressure and again found to be in satisfactory balance with the forming forces at the beginning of the process, which shows that the critical outflow pressure can also be predicted for square-cup deep-drawing processes. The hydraulic pressure distributions in the flange area and the chamber change significantly as the sheet conforms to the die shoulder, and the friction loss of hydraulic pressure at the die shoulder becomes large. These results indicate that the magnitude of the fluid-lubrication effect on the drawability of the sheet can vary with the stage of sheet deformation.

(cf. *ISIJ Int.*, **49** (2009), 239)

Possibility of applying superplastic forging to the microforming of SUS304

M.KATOH et al.

The microforming of SUS304 stainless steel with fine grain structures is carried out utilizing superplastic phenomenon. The configurations of superplastic-formed structures are studied, and possibility of superplastic microforming with SUS304 stainless steel is discussed. The microforming utilizing superplastic phenomenon successfully achieved the formation of pyramid-shaped projection with 50 μm base and 10 μm height on SUS304 specimen with ultra-fine grain structure averaging to 250 nm. It is believed that the surface roughness of the micron-sized structure, obtained in this experiment, may have reached below the Rayleigh limit for light with wavelength of at least 2 μm where the surface becomes smooth enough for infra-red (IR) device applications.

(cf. *ISIJ Int.*, **49** (2009), 247)

Welding and Joining

Pulsed current gas metal arc welding under different shielding and pulse parameters; part 1: arc characteristics

P.K.Ghosh et al.

The variation of arc characteristics with respect to its profile under Ar+2%CO_2, Ar+18%CO_2 and argon gas shielding and the stability of gas shielding with variation in pulse parameters have been studied by video-graphy of arc environment during P-GMA weld deposition using mild steel filler wire. The effect of pulse parameters has been studied by considering their hypothetically proposed summarized influence defined by a dimensionless factor, $\phi = [(I_b/I_p)/t_b]$, mean current and arc voltage. The measured root diameter, projected diameter and length have been measured and correlated to the P-GMA welding parameters under different gas shielding. It has been observed that the arc characteristics with respect to its shape and spreading over the weld significantly vary with ϕ, mean current and arc voltage giving due consideration to the shielding environment. The use of Ar+2%CO_2 gas shielding appreciably reduces the D_R but enhances the D_P and L with respect to that observed under Ar+18%CO_2 and argon gas shielding.

(cf. *ISIJ Int.*, **49** (2009), 251)

Pulsed current gas metal arc welding under different shielding and pulse parameters; part 2: behaviour of metal transfer

P.K.Ghosh et al.

The behaviour of metal transfer and arc stiffness understood in terms of arc pressure (P_a) of pulsed current gas metal arc welding (P-GMAW) using mild steel filler wire have been studied with respect to change in pulse parameters under different gas shieldings of Ar+2%CO_2 and Ar+18%CO_2. The arc environment revealing the droplet transfer from electrode to weld pool in bead on plate weld deposition has been studied by high speed video graphy. Effect of pulse parameters has been considered by their hypothetically proposed summarized influence defined by a dimensionless factor ϕ, mean current (I_m) and arc voltage. The droplet diameter and velocity of droplet at the time of detachment are found to vary significantly with the variation of ϕ. At a given ϕ the experimentally measured behaviour of metal transfer is found well in agreement to their corresponding theoretical estimates. The average droplet diameter transferred per pulse predominantly reduces but, the velocity of metal transfer at the time of detachment enhances with the increase of ϕ under both the shielding gases. In general a higher values of ϕ, I_m and arc voltage enhances the P_a depending upon type of gas shielding. The use of Ar+2%CO_2 shielding gas reduces the droplet diameter and enhances the arc pressure than that observed under Ar+18%CO_2 gas shielding.

(cf. *ISIJ Int.*, **49** (2009), 261)

Hot cracking of metal inert gas arc welded magnesium alloy AZ91D

D.X.Sun et al.

The microstructure and cracking characteristics of MIG welded magnesium alloy (AZ91D) joint, and the effect of welding speed on cracking susceptibility have been investigated. The welded joint consists of primary α-Mg and divorced phases (eutectic α-Mg+eutectic β-$Mg_{17}Al_{12}$), the latter mainly distributing along the α-Mg grain boundaries. Solidification cracking often occurred in the crater and was also observed at weld center line when welding speed was 300 mm/min, which are associated with segregation of Mn, Al and Zn and high tensile stresses in the welds. Liquation cracking appeared in HAZ immediately adjacent to the fusion line when low welding speed was used (300 mm/min). It is mainly related to the low welding speed resulting in increasing heat input and tensile stresses in the HAZ. It is favorable to decrease heat input for improving the susceptibility of hot cracking during MIG welding of magnesium alloys.

(cf. *ISIJ Int.*, **49** (2009), 270)

Surface Treatment and Corrosion

Formulation of a nondestructive technique for evaluating steel corrosion in concrete structures

Y.C.Lim et al.

This paper reports on a fundamental study on the formulation of a corrosion evaluation system for reinforced concrete structures based on a completely nondestructive technique. This is a method of evaluating the polarization resistance produced at the boundary between concrete and steel, without destroying the concrete surface, to estimate the resulting corrosion current density. A method of estimating the resistivity distribution within concrete to examine the steel corrosion environment is also proposed. This system consists of an equivalent circuit, a resistivity model for examining the effect of concrete, *i.e.*, the measurement medium, and an actual measurement system. The equivalent circuit takes account of the electrode arrangement for resistivity measurement, and the impedance within concrete. This technique was verified by tests using specimens with controlled corrosion losses and was proven to express corrosion deterioration within concrete well. Evaluation by this technique was thus found feasible.

(cf. *ISIJ Int.*, **49** (2009), 275)

Transformations and Microstructures

Thermomechanical processing of pipeline steels with a reduced Mn content

T.Schambron et al.

Lowering the Mn content of hot rolled pipeline steel can economise the steel making process by eliminating costly desulphurisation and at the same time improve product properties, particularly toughness, by reducing the formation of MnS inclusions. Lower Mn contents, however, have significant implications for austenite recrystallization and austenite (γ) to ferrite (α) transformation behaviour. This study compares the recrystallization start (T_{nr}) and γ to α transformation (A_{r3}) temperatures of two low Mn steels to those of a commercial steel with a conventional Mn content. Simulations of thermomechanical processing were carried out using a Gleeble 3500 testing machine. The T_{nr} for the low Mn alloy design were found to be ~25°C higher than for the high Mn steel, while the A_{r3} of the low Mn steel were some 50°C higher. The higher A_{r3} promotes the formation of coarse ferrite grains, thus reducing the strengthening and toughening benefits of a fine grain size. However, this could largely be overcome by maximising the degree of deformation performed between T_{nr} and A_{r3} and maintaining a combination of high cooling rates and low stop cooling temperatures below A_{r3}, thus providing a high density of nucleation sites and promoting high nucleation rates. Production trials validated the results from the Gleeble tests.

(cf. *ISIJ Int.*, **49** (2009), 284)

Fine grained austenitic stainless steels: The role of strain induced α' martensite and the reversion mechanism limitations

A.Poulon-Quintin et al.

The effect of the α'-strain induced martensite on the austenitic stainless steel grain size reduction is analyzed. Rolling with a 80% reduction factor at 200°C and at 20°C resulted respectively in a heavily deformed austenite and a mixed deformed austenite with α'-martensite bands. Further annealing treatments were carried out in the range of 20–880°C. Fine grains of about 1 μm can be obtained in the metastable austenitic stainless steel by a repetitive cold rolling and annealing thermomechanical process. In this case, reversion mechanism from deformation induced martensite α' to austenite γ is responsible for grain refining. Non-conventional warm rolling and annealing process can also lead to fine grains of 2 μm without a reversion stage and as a result of recrystallization process of the strain-hardened austenite. The cold rolling route gives rise to a bimodal grain size distribution with a "lamellar" structure resembling to a "composite" material.

(cf. *ISIJ Int.*, **49** (2009), 293)

Design of composition in (Al/Si)-alloyed TRIP steels

M.Gomez et al.

There is an increasing interest in the progressive substitution of Si by Al in TRIP steels in order to obtain alloys with excellent mechanical properties and improved coatability. In this paper, thermodynamic calculations have been carried out with the help of JMatPro™ software in order to assess and compare the effects that Si and Al additions exert on the phase transformation, carbon enrichment and alloying element content of phases during continuous galvanizing of multiphase steels. These simulations have provided important implications regarding the optimal combination of Si and Al. It has been found that Al causes a more pronounced increase of A_3 temperature and a wider extension of the intercritical range than Si. For a constant volume fraction of

phases, the carbon content in austenite is similar for Al and Si-alloyed steels. However, ferrite in Al-alloyed is richer in carbon and consequently an increase in its strength could be expected. The hardenability of intercritically annealed austenite has been estimated for alloys with different combinations of Mn, Al and Si. Finally, simulated CCT diagrams predict for Al-alloyed steels a higher amount of new ferrite formed during cooling from intercritical annealing and the need of shorter isothermal holding times at 460°C. However, Si-TRIP steels would need faster cooling rates to prevent pearlite formation and longer isothermal holding times to complete the bainitic transformation and to obtain a microstructure with high retained austenite.

(cf. *ISIJ Int.*, **49** (2009), 302)

Mechanical Properties

Evolution of dislocation structure and fatigue crack behavior in Fe–Si alloys during cyclic bending test

K. USHIODA et al.

The evolution of dislocation structures was investigated by TEM in Fe–Si alloys with 0, 0.5 and 1.0 mass% Si during a cyclic bending test in conjunction with fatigue crack behavior. The addition of Si increased the fatigue strength. The evolution of dislocation structures was signifiicantly inflluenced by t Si addition. Namely, in the steel without Si the dislocation cell structure develops, whereas in the steel with 1 mass% Si the vein structure develops, which is considered to lead to increased fatigue strength. The dislocation cell structure observed in the steel without Si is postulated to be caused by the easy cross slip of dislocations during cyclic deformation, whereas the vein structure that developed in the steels with Si is inferred to be caused by the diffiicuty in cross slip due to the decrease in stacking fault energy. Furthermore, the Si added steel shows a characteristic structure in a manner such that the dislocations are free in approximately 0.5 μm zones along grain boundaries. The examinations of the fatigue fracture surface revealed that transgranular fracture takes place in steel without Si, whereas in steel with 1 mass% Si many intergranular cracks were observed just beneath the top surface. The intergranular cracks in the 1 mass% Si steel were thought to be caused by the fact that a) strains are dispersed within grains owing to the vein structure and b) micro cracks are initiated and propagated along grain boundaries due to the dislocation free zones.

(cf. *ISIJ Int.*, **49** (2009), 312)