

Fundamentals of High Temperature Processes**Phase equilibria in the cristobalite, tridymite and pyroxene primary phase fields in the MgO–“FeO”–SiO₂ system in equilibrium with metallic iron***S.CHEN et al.*

Experimental studies have been carried out to determine the phase equilibria for the ternary MgO–“FeO”–SiO₂ in equilibrium with metallic iron. Liquidus isotherms have been determined in the temperature range from 1578 to 1898 K in the silica and pyroxene primary phase fields, using high temperature equilibration, modified quenching and electron probe X-ray microanalysis (EPMA) techniques.

(cf. *ISIJ Int.*, **45** (2005), 791)**Effect of Al₂O₃ and Cr₂O₃ on liquidus temperatures in the cristobalite and tridymite primary phase fields of the MgO–“FeO”–SiO₂ system in equilibrium with metallic iron***S.CHEN et al.*

The effects of alumina and chromite impurities on the liquidus temperatures in the cristobalite/tridymite (SiO₂) primary phase fields in the MgO–“FeO”–SiO₂ system in equilibrium with metallic iron have been investigated experimentally. Using high temperature equilibration and quenching followed by electron probe X-ray microanalysis (EPMA), liquidus isotherms have been determined in the temperatures range 1673 to 1898 K. The results are presented in the form of pseudo-ternary sections of the MgO–“FeO”–SiO₂ system at 2, 3 and 5 wt% Al₂O₃, 2 wt% Cr₂O₃, and 2 wt% Cr₂O₃+2 wt% Al₂O₃. The study enables the liquidus to be described for a range of SiO₂/MgO and MgO/FeO ratios. It was found that liquidus temperatures in the cristobalite and tridymite primary phase fields, decrease significantly with the addition of Al₂O₃ and Cr₂O₃.

(cf. *ISIJ Int.*, **45** (2005), 798)**Copper enrichment behaviours of copper-containing steels in simulated thin-slab casting processes***R.Y.CHEN et al.*

The copper enrichment behaviours of several copper-containing steels under conditions similar to those in the thin-slab casting processes are examined. Formation of a molten copper phase at the scale–steel interface can be avoided when the substrate phase is occluded in the scale during steel oxidation. Significant enrichment of nickel in the surface layer of the substrate is a necessary condition for the occlusion of the substrate phase into the scale before the precipitation of a molten copper phase. The critical nickel content above which the occlusion mechanism would be operative is between 0.02 % and 0.07 %. The critical temperature above which the occlusion mechanism becomes operative in the high nickel steels differs when the isothermal oxidation step, following continuous cooling in ambient air, is conducted in different atmospheres. When conducted in ambient air, this temperature is between 1150°C and 1230°C, whereas when conducted in moist air or in simulated furnace atmospheres, it is lowered to between

1100°C and 1150°C. Preferential oxide growth along the grain boundaries of the substrate is another important occlusion mechanism, however, the deep oxide penetrations in the substrate may lead to descaling difficulty.

(cf. *ISIJ Int.*, **45** (2005), 807)**Ironmaking****Development of coating granulation process at commercial sintering plant for improving productivity and reducibility***N.OYAMA et al.*

An advanced granulation process for treatment of raw mixtures was developed to improve sinter productivity and reducibility through fundamental and applied studies. Proper selection of the coating time is the most important factor for achieving improvement, because destruction of quasi-particles proceeds together with granulation in the drum mixer. The advanced granulation process is characterized by coating coke breeze and limestone on the surface of quasi-particles which have been granulated in the primary part of the drum mixer. Coke breeze and limestone are injected from the end of the drum mixer by the belt conveyor at high speed to achieve coating on the quasi-particles. Because these two materials segregate in the quasi-particles, this process controls the excess melting reaction between iron ore and limestone.

Fundamental studies and commercial plant trials showed that the desirable melt fluidity resulting from the above-mentioned segregation enhanced permeability in the sintering bed. The superior reducibility of the sinter product depends on the diffusible structure retaining micro pores originating in the relict ores.

Commercial sinter plants at JFE Steel's West Japan Works with an annual production capacity of 13.5 million tons have already introduced the new granulation process. The new process remarkably improves both the productivity and reducibility of sinter products in spite of the recent prevalence of inferior ores. In addition to improving the sintering operation, this granulation process also contributes significantly to improve blast furnace operation, including both higher productivity and lower reducing agent rate.

(cf. *ISIJ Int.*, **45** (2005), 817)**Steelmaking****Effect of iron oxide feeding rate and hot metal temperature on dephosphorization rate in torpedo car***Y.HINO et al.*

In this study, the reduction behavior of iron oxide and the effects of the iron oxide feeding rate and hot metal temperature on the dephosphorization rate were investigated in laboratory experiments and industrial plant tests with the aim of increasing the dephosphorization rate. The results are summarized as follows:

(1) When the iron oxide feeding rate was 400 kg/min under a constant lime feeding rate, 61 % of the iron oxide is reduced by a transitory reaction

zone, while 11 % is reduced by a permanent reaction in the effective reaction zone in the top slag. The remainder is not reduced and remains in the dead zone in the torpedo car in the form of iron oxide.

(2) The iron oxide reduction rate in the transitory reaction zone is dependent on the hot metal temperature and iron oxide feeding rate. When the hot metal temperature was reduced from 1653 to 1563 K ($\Delta 90^\circ\text{C}$), the iron oxide reduction rate decreased by 40%. The reduction ratio decreased by 20% when the iron oxide feeding rate was increased from 200 to 400 kg/min.

(3) The apparent activation energy of iron oxide reduction was evaluated as 132 kJ/mol, which is slightly smaller than the temperature dependence of the diffusion coefficient of oxygen in FeO–CaO–SiO₂ slag.

(4) A new mathematical model of hot metal dephosphorization in the torpedo car was developed, which considers the effect of the hot metal temperature and iron oxide feeding rate on the oxygen supply rate. The results with this model showed good agreement with actual data from the torpedo car.

(cf. *ISIJ Int.*, **45** (2005), 827)**Physical modeling of slag foaming for various operating conditions and slag compositions***D.LOTUN et al.*

The present study is concerned with steady-state slag foaming. First, the various models presented in the literature are briefly reviewed and their accuracy in predicting steady-state foam thickness is evaluated by using a wide range of experimental data. Then, a correlation is developed by applying the Buckingham's Pi theorem to variables commonly used in previous studies on slag foaming. However, the concept of foaminess has been disregarded as it is an idealization that is not supported by experimental evidences. A power type of law has been assumed between the four dimensionless numbers derived. The empirical coefficients are obtained using experimental data reported in the literature. They cover a wide range of operating conditions and foaming fluids with appreciably different chemico-physical properties. The predictions of the correlations compares well with experimental data. The dimensionless numbers derived from the Buckingham's Pi theorem and the associated empirical coefficients are found to be similar to those obtained by performing a dimensional analysis of the governing equation for the transient foam thickness which could not be predicted a priori. Finally, the dependency of the average bubble radius with surface tension is discussed. Overall, the predicted thickness decreases with increasing surface tension thus satisfying basic thermodynamics considerations.

(cf. *ISIJ Int.*, **45** (2005), 835)**Instrumentation, Control and System Engineering****Roll speed and roll gap control with neural network compensation***M.MAHFOUF et al.*

In this paper the detailed procedure of roll speed

and roll gap control strategy development for a laboratory scale rolling mill is given. The core of the control strategy is the incorporation of feed-forward compensations based on neural network models for the roll force and roll torque, which are the major disturbances introduced during the rolling operation. An integrated computer simulation model is developed to investigate the performance of the proposed control strategies, and results show significant improvement over the traditional feedback control scheme. Based on the control strategies and the integrated simulation model, a major upgrading scheme is undertaken on an existing laboratory scale rolling mill. The new mill data acquisition and control systems, including the upgrading of the drive and gap motors, are currently under commissioning. After the mill upgrading system is fully commissioned, further work such as online adaptation of the neural network prediction model and the fine-adjustment of the feed-forward compensation need to be investigated for consistent control performance under changing rolling conditions.

(cf. *ISIJ Int.*, **45** (2005), 841)

Forming Processing and Thermomechanical Treatment

Effect of TMCP parameters on the microstructure and properties of an Nb-Ti microalloyed steel

Y. LIU et al.

Thermal mechanical control processing (TMCP), which includes combination of controlled rolling and controlled cooling, provides a powerful means of developing high-strength low alloy (HSLA) steels by intensive microstructural control. In the present investigation, the effects of TMCP parameters, consisting of the finish cooling temperature and the start rolling temperature in non-recrystallization region, on the final microstructure and mechanical properties of an Nb-Ti microalloyed steel has been studied by tensile, Charpy impact tests, optical microscopy and scanning electron microscopy. The TMCP parameters for Q460 grade steel have been optimized by laboratory experiments. The microstructure and properties of industrial product were coincident with the results of laboratory experiments.

(cf. *ISIJ Int.*, **45** (2005), 851)

Regression method of determining generalized description of flow curve of steel under dynamic recrystallization

A. YANAGIDA et al.

A regression method of obtaining a generalized description of a flow curve is proposed in this paper. The flow curve of a metal at a constant temperature and a constant strain rate is precisely obtained by an inverse analysis of hot compression. To eliminate the error in the flow curve induced by the inhomogeneous distribution of temperature in the test piece, an electromagnetic analysis of induction heating is performed with thermal analysis throughout the heating and hot compression of a test piece. The coefficients embedded in the flow curve are determined by the inverse analysis associated with thermomechanical FE (Finite Element) analysis. The

obtained coefficients, which cover wide ranges of hot-forming temperature and high strain rate, are regressed using the proposed regression method, and a generalized flow curve for a C-Si-Mn steel is obtained. Although the proposed regression method is demonstrated for only one type of C-Si-Mn steel, it could also be used to obtain a generalized description of a flow curve for other types of steel under dynamic recrystallization, which could be used to estimate the force characteristics of hot metals in bulk forming processes, such as forging and rolling.

(cf. *ISIJ Int.*, **45** (2005), 858)

TEM characterization of the recrystallization behaviour of warm rolled low carbon steels containing chromium during the early stages of annealing

I.B. TIMOKHINA et al.

The early stages of recovery and recrystallisation were studied using transmission electron microscopy in four warm rolled low carbon steels. Three of these contained additions of chromium and of the latter, one was phosphorus and a second boron modified. Addition of the alloying elements led to the formation of both shear and microbands within some of the grains. The progress of recovery in these areas differed from that applicable to the grains containing only microbands. Two types of carbides were present after warm rolling in the steels containing the alloying additions: (i) coarse carbides; and (ii) fine strain-induced particles. The coarse carbides underwent spheroidization and coarsening during annealing. They, on the one hand, accelerated recrystallisation as the particles stimulated nucleation; on the other hand, they retarded it by pinning both the high and low angle grain boundaries. These carbides also influenced the morphology of the recrystallised grains by restraining their growth.

(cf. *ISIJ Int.*, **45** (2005), 867)

Welding and Joining

Prediction of acicular ferrite from flux ingredients in submerged arc weld metal of C-Mn steel

P. KANJILAL et al.

The prediction model has been developed for low carbon steel weld metal acicular ferrite microstructure as a function of flux ingredients such as CaO, MgO, CaF₂ and Al₂O₃ in submerged arc welding carried out at fixed welding parameters. The results of quantitative measurements of acicular ferrite (AF) on eighteen no. of weld metal samples were utilised for developing the prediction model. Among the flux ingredients, CaO appears to be most important as an individual as well as interaction with other ingredients in controlling the amount of acicular ferrite content in the weld metal. Furthermore, formation of acicular ferrite is also related to the weld bead geometry which is influenced by flux ingredients. The prediction equation for acicular ferrite has been checked for adequacy by performing separate experiments on welding using randomly designed flux. The isoresponse curves were developed to show the level of acicular ferrite content at different percentage of flux ingredients.

(cf. *ISIJ Int.*, **45** (2005), 876)

Surface Treatment and Corrosion

Hot corrosion behaviour of plasma sprayed coatings on a Ni-based superalloy in Na₂SO₄-60%V₂O₅ environment

S. PRAKASH et al.

NiCrAlY, Ni-20Cr, Ni₃Al and Stellite-6 metallic coatings were deposited on a Ni-based Superalloy (18.5Fe-19Cr-0.5Al-0.9Ti-3.05Mo-0.18Mn-0.18Si-5.13Ta-0.15Cu-0.04C-Balance Ni). NiCrAlY was used as bond coat in all the cases. Hot corrosion studies were conducted on uncoated as well as plasma spray coated superalloy specimens after exposure to molten salt at 900°C under cyclic conditions. The thermogravimetric technique was used to establish kinetics of corrosion. X-Ray Diffraction, SEM/EDAX and EPMA techniques were used to analyse the corrosion products. The uncoated superalloy suffered a catastrophic corrosion in the form of intense spalling of the scale accompanied by disintegration of the scale with cracking sounds. NiCrAlY coated specimen showed a minimum weight gain, whereas the Stellite-6 indicated a maximum weight gain among the coatings studied. All the coatings were found to be successful in reducing spalling of the superalloy substrate. It is concluded that the formation of oxides and spinels containing nickel, aluminium, chromium or cobalt contributes to an improved hot corrosion resistance.

(cf. *ISIJ Int.*, **45** (2005), 886)

Correlation between the microstructure of galvanized coatings and the defoliation during press forming

M.-H. HONG

The defoliation properties of galvanized coating, so-called powdering and flaking, in a commercial continuous galvanizing line were investigated by scanning electron microscopy, X-ray diffraction, a roughness profiler and deep drawing test. Both aluminum content in molten zinc and galvanizing temperature were found to be critical factors controlling the microstructure and mechanical properties of galvanized coatings. Below 0.135 wt% Al, the coating surface was composed of a mixture of granular δ_p and columnar ζ phases, while, above 0.155 wt% Al, the coating surface contained the mixture of granular δ_p and pan-cake δ_k phases. The appearance of brittle δ_k phase approximately 10 μ m in size can account for the high amount of defoliation during deep drawing test. The formation of relatively ductile ζ phase and thin Γ phase contributes to improve the press formability. The optimization of the surface microstructure controlling the ratio of granular δ_p and columnar ζ phases was important to reduce the powdering.

(cf. *ISIJ Int.*, **45** (2005), 896)

Transformations and Microstructures

Thermodynamic modeling in reduced activation steels

C.A. DANON et al.

Reduced Activation (RA) martensitic steels of the

9/11CrWVTa type are promising candidates for fusion reactor applications as long as they exhibit good mechanical properties—similar to those of commercial martensitic steels of the 9/12CrMo(NbV) type—and satisfy design criteria related to the behavior under irradiation. A contribution on thermodynamic modeling in RA steels is presented. The current status of the development of a thermodynamic database in the framework of the CALPHAD approach is reported. Thermodynamic calculations concerning RA alloys are discussed which can be helpful to interpret experimental observations on phase relationships, phase compositions, transition temperatures, etc.

(cf. *ISIJ Int.*, **45** (2005), 903)

Low temperature tempering of a medium carbon steel in high magnetic field

Y.ZHANG *et al.*

The effect of low temperature magnetic tempering on carbide precipitation in a medium carbon steel, 42CrMo, has been investigated. As-quenched steel specimens were tempered at 200°C for 60 min without and with a 14-T magnetic field. Results show that under the magnetic field processing there forms the relatively high-temperature monoclinic χ -Fe₃C₂ carbide with a denser distribution and smaller sizes, as compared to the usual orthorhombic η -Fe₃C carbide obtained without the magnetic field. The impact of the external magnetic field refers to a change in the precipitation sequence of the transition carbides by effectively lowering the Gibbs free energy of the high magnetization phases. The denser distribution and smaller size of χ -Fe₃C₂ precipitates are attributed to the increased nucleation rate and the weaker diffusion capacity required for growth as the formation temperature is lower. This offers additional dispersion strengthening to compensate for the decrease in strength and hardness due to the loss of supersaturation of carbon atoms in the matrix and raises the toughness of the steel.

(cf. *ISIJ Int.*, **45** (2005), 913)

Texture effect on magnetic properties by alloying specific elements in non-grain oriented silicon steels

S.K.CHANG *et al.*

The effect of texture on magnetic properties by adding specific elements of tin, antimony and boron was investigated. Texture factor was defined as the ratio of the sum of cube and Goss textures to γ fibre. Tin and antimony showed larger effect in enhancing

grain size than boron for both hot rolled and cold rolled bands. Texture factor increased with an increase in grain size; that is, grain size effect on texture factor appeared larger for the addition of tin and antimony than for boron. Both tin and antimony showed large volume fractions of cube and Goss textures but low volume fraction of γ fibre compared to boron. The effect of texture factor on core loss was larger for tin and antimony added steels than for boron added steel. Magnetic flux density increased proportionally with the texture factor and the effect of texture factor on magnetic flux density was also greater in tin and antimony added steels than in boron steel.

(cf. *ISIJ Int.*, **45** (2005), 918)

Ridging-free ferritic stainless steel produced through recrystallization of lath martensite

T.TSUCHIYAMA *et al.*

Ridging phenomenon was successfully suppressed in a ferritic stainless steel by controlling microstructure through recrystallization of lath martensitic structure. Fe–12Cr–1Ni alloy was quenched after the solution treatment in an austenite single phase region to obtain lath martensitic structure. Cold rolling was performed to the quenched materials up to 80% reduction in thickness before the annealing for recrystallization. With increasing the reduction by cold rolling, the recrystallization was promoted and ferrite grain size was decreased to 20 μ m after recrystallization in the 80% pre-cold-rolled material. A weak $\langle 111 \rangle$ //ND recrystallization texture was formed by the cold rolling, but no grain colonies existed in the microstructure. As a result, the materials produced through the recrystallization of lath martensite did not cause ridging during tensile deformation, although an orange peel appeared when the grain size was not refined enough.

(cf. *ISIJ Int.*, **45** (2005), 923)

Mechanical Properties

Evaluation of plastic deformation limit by circumferentially notched tension test

K.ENAMI *et al.*

Circumferentially notched and smooth round tension tests were carried out at room temperature with and without compressive prestrain in a hot-rolled ferrite–pearlite SM490B steel, and the effects of the compressive prestrain and the notch on the plastic deformation limit (PDL) were investigated. The PDL was defined as a termination point in a true

stress–true strain curve which was caused by a ductile or cleavage fracture. The notch caused variations in the average stress–true strain curve but did not affect the true stress–true strain curve. A true strain at the plastic instability was determined by a work hardening exponent, regardless of the notch. On the other hand, the true strain at the PDL differed according to the notch. An axial stress at the PDL was found to be about 1 300 MPa, regardless of the ductile and cleavage fracture.

(cf. *ISIJ Int.*, **45** (2005), 930)

Social and Environmental Engineering

Hydrothermal synthesis of zeolite A using blast furnace slag

Y.SUGANO *et al.*

Alkali hydrothermal synthesis of zeolite A using blast furnace (BF) slag was investigated. The preliminary experiment was conducted in use of synthetic slag consisting of SiO₂, Al₂O₃, and CaO powders, and it was made clear that the most optimum slag compositions to synthesize zeolite A were the molar ratio of Si to Al (Si/Al) of 1 and reduction of CaO content down to 15 mass%, and that hydrothermal treating conditions were the temperature range from 328 to 358 K, NaOH solution of 1 M (=mol/L) and the ratio of the volume of NaOH solution to total mass of slag ($V_{\text{sol}}/W_{\text{slag}}$) of 15 (mL/g). It was also found that synthetic slag with such a higher content of CaO as 40% resulted in formation of tobermorite and hydrogarnet. In the experiment using BF slag, zeolite A could be successfully synthesized by optimizing both conditions of the compositions of raw material powders and hydrothermal treatment as noted above, where optimization of the compositions of raw material powders such as Si or Al content were performed by the suitable amount of addition of SiO₂ powder or NaAlO₂ powder as a source of Si and Al. The ball milling type reaction vessel containing numerous small SiC balls which was first adopted in this hydrothermal treating study was confirmed to be very effective for acceleration of synthetic reaction rate, shortening markedly the time period needed for fully synthesis of zeolite A. Temperature dependence of heat capacity of zeolite A powder synthesized in use of BF slag was measured after absorption of vapor at the ambient temperature, showing endothermic behavior with the peak at the temperature of around 473 K.

(cf. *ISIJ Int.*, **45** (2005), 937)