

## Fundamentals of High Temperature Processes

### Wettability and interfacial permeability between prereduced ilmenite and molten pig iron

Z. YUAN *et al.*

The interfacial phenomena between molten pig iron and ilmenite prereduced with carbon and hydrogen were investigated. The wettability of prereduced ilmenite discs by molten pig iron was evaluated through measuring contact angle by the sessile droplet method. The effects of melting temperature, prereduction temperature and reduction degree on contact angle were examined. Molten pig iron could not be wetted by ilmenite prereduced with carbon and hydrogen, and the contact angle increases with increasing prereduction temperature. The contact angles of ilmenite prereduced with hydrogen are higher than that of ilmenite prereduced with carbon at the same reduction degree. The permeation of molten pig iron into porous ilmenite was observed at the interface of specimens prereduced with carbon and enhanced at higher reduction temperature. On the other hand, in the case of ilmenite prereduced with hydrogen, the permeation was not observed. These results indicate clearly that the separation between molten pig iron and reduced ilmenite can be enhanced by hydrogen reduction process, even at lower temperature than that of carbon reduction process.

(cf. *ISIJ Int.*, **49** (2009), 323)

### The Influence of Ash Impurities on Interfacial Reactions between Carbonaceous Materials and EAF Slag at 1550°C

M. RAHMAN *et al.*

sCarbon/slag interactions have been investigated between two carbonaceous materials, metallurgical coke (18.3% ash) and natural graphite (2.1% ash), and an EAF slag (34.8% FeO) using a sessile drop arrangement at 1550°C to determine the role played by carbonaceous materials in carbon/slag interactions and interfacial phenomena. Gaseous emissions from metallurgical coke in the presence of slag were much higher than those from coke alone indicating significant carbon/slag interactions with the system exhibiting extensive/rapid iron oxide reduction, very high rates of gas generation but poor gas entrapment within the slag phase. High levels of gas generation led to a strong likelihood of convective transport of reactants and products across the metallurgical coke/slag interface with oxides present in coke ash partially dissolving in molten slag and modifying slag composition. Natural graphite on the other hand showed slow rates of gas generation, slow reduction of iron oxide and excellent gas entrapment. Gases generated from graphite alone were comparable to those generated in the presence of slag and were an order of magnitude lower than the corresponding emissions from metallurgical coke/slag system. Slag interactions with metallurgical coke resulted in lower surface tension due to pickup of silica from coke ash and small droplet volumes; slag interactions with natural graphite gave rise to minor changes in slag properties due to low ash levels and increases in droplet volumes. The rate of gas gener-

ation was found to be an important parameter in carbon/slag interactions and needs to be maintained at optimum levels for sustaining gas entrapment within slag over extended periods. This study highlights significant differences in the carbon/slag interactions of two carbonaceous materials with an EAF slag, and the important role played by ash impurities.

(cf. *ISIJ Int.*, **49** (2009), 329)

### Thermodynamics of titanium oxide in CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO<sub>satd</sub>-CaF<sub>2</sub> slag equilibrated with Fe-11mass%Cr melt

J.H. PARK *et al.*

Thermodynamic equilibrium between the CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO<sub>satd</sub>-CaF<sub>2</sub>(-TiO<sub>2</sub>) slags and the Fe-11mass%Cr melt was investigated at 1873 K in order to understand the thermodynamic behavior of titanium oxide in the refining slags. The equilibrium between silicon and titanium in steel melts and their oxides in the slags are theoretically expected and experimentally proved well. The activity coefficient of TiO<sub>2</sub> increases by increasing the basicity in logarithmic scale. By combining this with the previous results, in multi component calcium(-magnesium) silicate slags containing Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>, TiO<sub>2</sub> could be considered as a basic oxide in silicate base melts, while it could be the relatively acidic oxide in the aluminate (very low silica) base melts. The activity coefficient of TiO<sub>2</sub> gradually decreases by substituting silica for alumina, indicating that the attraction between TiO<sub>2</sub> and SiO<sub>2</sub> is greater than that between TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in the present slag system. This could be explained from the electronegativity difference between each cation involved. The activity of TiO<sub>2</sub> in the 10%MgO slag system shows a negative deviation from an ideality, while that in the CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO<sub>satd</sub>(-CaF<sub>2</sub>) system is relatively close to the ideal behavior up to about 10 mol% TiO<sub>2</sub>. This is mainly due to the relatively basic characteristic of TiO<sub>2</sub>.

(cf. *ISIJ Int.*, **49** (2009), 337)

### A model for estimating the viscosity of molten aluminosilicate containing calcium fluoride

Y. MIYABAYASHI *et al.*

A model to estimate the viscosity of aluminosilicate melts including alkali oxides, as derived in our previous study, was extended to molten slag systems containing CaF<sub>2</sub>. The bonding state of oxygen in molten silicate and the flow mechanism of melts with a network microstructure were considered for this model. To evaluate the bonding states of oxygen for given chemical compositions, a model proposed by Susa *et al.* was applied. Their model is easily extended to any multi-component molten slag system without the need to consider thermodynamic parameters. In this work, the above-mentioned viscosity model was applied to evaluate the effect of CaF<sub>2</sub> on the viscosity of molten SiO<sub>2</sub>-CaO-CaF<sub>2</sub>, SiO<sub>2</sub>-CaO-Al<sub>2</sub>O<sub>3</sub>-CaF<sub>2</sub> and SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO-CaF<sub>2</sub>-Na<sub>2</sub>O-MgO systems.

(cf. *ISIJ Int.*, **49** (2009), 343)

## Ironmaking

### The effect of lime coating of hematite pellets on soot formation and reduction rate with CH<sub>4</sub>/H<sub>2</sub>

A. RABIEIFAR *et al.*

In some of the Direct Reduction (DR) methods, the iron oxides would be reduced with reformed natural gas. Due to the reduction conditions, temperature, gas flow, pressure, and the component of the reducer gas, the decomposition of CH<sub>4</sub> and CO to C<sub>soil</sub>/soot formation on the surface of the pellets is possible. Consequently, the rate of reduction, and the consumption of the reducer gas could be changed. In this research, the effect of coated hematite pellets with lime on the reduction rate of iron oxides, and also C<sub>soil</sub>/soot formation, is reported. By increasing of 3-8% lime on the surface of the pellets with a diameter of 10 mm, 15-40% H<sub>2</sub> in H<sub>2</sub>/CH<sub>4</sub> mixture, and 1050 to 1250°C reaction temperature, the rate of C<sub>soil</sub>/soot formation is decreased, and the rate of reduction is increased.

(cf. *ISIJ Int.*, **49** (2009), 349)

## Casting and Solidification

### The effect of alloy solidification path on sulfide formation in Fe-Cr-Ni alloys

K. NAKAMA *et al.*

Solidification and sulfide precipitation in Fe-Cr-Ni alloys were investigated. Five samples containing 0.3 wt% S and 1.5 wt% Mn were investigated and each sample chemistry was adjusted to solidify as primary austenite, primary ferrite or eutectic by tailoring the Cr and Ni contents. The solidification was observed *in-situ* in a Confocal Scanning Laser Microscope (CSLM) equipped with a gold-image furnace, and the morphology and distribution of sulfides were characterized through SEM and Optical Microscopy. For high Ni and low Cr alloys which solidified in the primary austenite field, sulfides formed in the enriched melt ahead of the dendrite fronts as a eutectic metal-sulfide structure. On the other hand, for high Cr and low Ni alloys which solidified in the primary ferrite field, liquid sulfide phase precipitated in the inter-dendritic enriched melt first, and it appeared to deform and to concentrate in the dendrite boundaries during cooling. In the near eutectic intermediate Cr and Ni containing alloy, sulfides formed in the solid state. The sulfides formed in all the alloys were Mn and/or Cr rich.

(cf. *ISIJ Int.*, **49** (2009), 355)

### Growth of solidified shell just below the meniscus in continuous casting mold

M. HANAO *et al.*

In the high speed continuous casting of hypoperitectic steel slabs, growth of solidified shell just below the meniscus in the mold was researched. Rate of cooling and solidification was estimated on the basis of experimental results of thickness profiles of solidified shell, heat flux in the mold and dendrite arm spacing in the solidified structure beneath the surface of the slabs cast at 3-5 m/min.

As a result, it was found that there is a delaying period of solidification growth at the beginning, till the shell grows up to about 1 mm thick. After that, it grows in linear relation to square root of solidification time. Dealing with the thickness profiles as a function of solidification time and solidification rate was obtained by differential. Furthermore, cooling rate of the shell was estimated with this solidification rate and heat flux in the mold. This cooling rate showed good agreement with that estimated from dendrite secondary arm spacing of the slabs.

Estimated cooling rates were compared with the slabs by conventional continuous caster at 1.1–1.6 m/min and the influence of casting speed was discussed. It resulted in that cooling rate increases with casting speed and the difference by casting speed begins to be remarkable in the period of 0.1–1 s for solidification time, when uneven solidification just begins to occur. This agreement was considered to be one of the reasons why uneven solidification or longitudinal surface cracking of slabs tends to occur with increase of casting speed.

(cf. *ISIJ Int.*, **49** (2009), 365)

### Instrumentation, Control and System Engineering

#### A modelling and tabu search heuristic for a continuous galvanizing line scheduling problem

*L. TANG et al.*

In this paper, we address a continuous galvanizing line scheduling problem in a steel plant. The continuous galvanizing line we research produces principally two kinds of coils, inner galvanized coils and outer galvanized coils. Due to the technical constraints, outer coils can not be produced continuously more than a specified number and some inner coils must be inserted between outer coils. The problem is to find a schedule of all coils that minimizes the sum of changeover costs, the number of inserted inner coils and the number of transition coils used. The difficulty of solving the problem lies in the interrelation of sequencing these two kinds of coils. We formulate the scheduling problem as an integer programming model by considering above practical requirements. A heuristic based on tabu search is developed to solve the problem. The matching algorithm is used during the procedure of generating an initial solution where the optimal assignment of candidate inner coils to be inserted and positions is found by Hungarian method and then the saving step is used to reduce the number of inner coils inserted. In implementing the tabu search heuristic, intensification search, diversification search and path relinking are used to improve the effectiveness of the algorithm. 28 instances of four sizes are randomly generated to simulate the actual production data. For all the instances with 20 and 30 coils and four out of five instances with 50 coils, the tabu search heuristic finds the optimal schedules as CPLEX does. For other instances, the heuristic always obtains the better schedules and consumes much less computation time than CPLEX. In real production environment, hundreds of coils are needed to be scheduled, and the heuristic is able to gen-

erate high quality schedules within reasonable computation time.

(cf. *ISIJ Int.*, **49** (2009), 375)

### Forming Processing and Thermomechanical Treatment

#### The Theoretical and Experimental Research on the Forward Slip Coefficient in Rail Universal Rolling

*Y.-g. DONG et al.*

In rail universal rolling, the forward slip coefficient is one of the most important parameters. For simplifying the analytic model, the vertical roll with box pass has been simplified as an equivalent flat roll firstly. Then the equation of neutral line and the area of forward slip zone on the flank of horizontal roll have been derived. Furthermore, the horizontal resultant force acting on the rolled workpiece has been obtained and the forward slip coefficient of rail universal rolling has been proposed. For verifying the theoretical model, the universal rolling process of 60 kg/m heavy rail and 18 kg/m light rail have been simulated by the Rigid-Plastic FEM software DEFORM-3D V5.0, and the universal rolling experiments of 18 kg/m light rail has been accomplished in the Yanshan University Rolling Laboratory. Moreover, the theoretical results and numerical simulation results of forward slip coefficient are in agreement with the experimental data basically. So, this theoretical model can be applied in rail universal rolling.

(cf. *ISIJ Int.*, **49** (2009), 385)

#### Towards improved reliability of the analysis of factors influencing the properties on steel in industrial practice

*T. VECKO-PIRTOVSEK et al.*

Due to the large number of parameters that influence the properties of steel and lower accuracy of some measured data, it can take several years for industry to collect a database large enough to carry out reliable analysis. In this paper a new approach is presented to overcome these problems. The Latin hypercube sampling technique (LHS) was used for modelling of the uncertainty of measured industrial data and CAE NN was used for analysis of the mutual dependence of influencing parameters. Using the example of AISI D2 tool steel, the proposed method was applied for determination of relationships between chemical composition and yield  $\delta$  by considering the corresponding coefficients of variation. New insights of relationships were directly applicable in the industrial practice. Despite using small database, it was possible to considerably increase the yield  $\delta$  for industrial hot rolling.

(cf. *ISIJ Int.*, **49** (2009), 395)

#### An approximate model for local strain variation over material thickness and its applications to thick plate rolling process

*C.-H. MOON et al.*

This paper presents a three-parameter approximate model which computes the local strain varia-

tion over thickness direction of thick material in the roll gap. The three parameters were determined through finite element analysis. With the proposed model, we then carried out a series of plate rolling simulation to examine the effect of the arithmetic average aspect ratio (ratio of contact length between work roll and material to mean material thickness in the roll gap) and reduction ratio on the local strain variation over material thickness as material goes through rolling at many passes.

Results reveal that a certain amount of relative difference between local strain at center and that at surface always exists as arithmetic average aspect ratio increases for whole plate rolling process. It also shows that the magnitude of local strain at material center is only 69% of that at surface during rolling no matter how we regulate incoming material thickness, radius of work roll, reduction ratio, roll speed and friction condition when arithmetic average aspect ratio is greater than 1.0. It has been found that the heavy reduction with arithmetic average aspect ratio, 0.9 might be an optimum condition for refining grain size over material thickness in an approximately uniform manner.

(cf. *ISIJ Int.*, **49** (2009), 402)

#### Analysis of longitudinal buckling in temper rolling

*K. KOMORI*

In temper rolling, a shape defect known as longitudinal buckling sometimes occurs, resulting in the formation of wrinkles like those on a washboard. The direction of the crest line of the longitudinal buckling is parallel to the rolling direction. In this study, the analysis of longitudinal buckling is performed using the elementary theory of buckling. First, we calculate the material-roll contact ratio by Hertz's formulae. Next, we calculate the stress distribution in the material at the roll gap using the punch pressure during indentation by Prandtl's formulae. Furthermore, we calculate the wavelength of the waves formed by longitudinal buckling using the elementary theory of buckling. We find that the wavelength calculated from the analysis is in good agreement with that obtained experimentally in the literature. We conclude that the method of analysis is valid, and that the cause of longitudinal buckling is the surface roughness of the roll.

(cf. *ISIJ Int.*, **49** (2009), 408)

### Welding and Joining

#### Analysis of fume formation rate and fume particle composition for Gas Metal Arc Welding (GMAW) of plain carbon steel using different shielding gas compositions

*K. R. CARPENTER et al.*

The present paper compares thirteen shielding gases and their impact on FFR and fume particle while welding in the spray transfer regime. There was no obvious influence from the shielding gas on particle composition and fume particles were identified as  $(\text{Fe}, \text{Mn})_3\text{O}_4$ . There was a slight peak shift that indicated that small levels of Mn, as detected by TEM-EDS, substituted for Fe in the  $\text{Fe}_3\text{O}_4$  phase.

Shielding gas composition is an important para-

meter for successful gas metal arc welding (GMAW) and has been shown to affect the fume formation rate (FFR). In Ar-based mixtures, increasing CO<sub>2</sub> had a greater impact than raising O<sub>2</sub> on FFR. When O<sub>2</sub> was increased in ternary mixtures, the FFR increased for Ar–5%CO<sub>2</sub> but no discernable increase was observed for the Ar–12%CO<sub>2</sub> mixtures. Results indicate that CO<sub>2</sub> additions in Ar-based shielding gases are the controlling factor in determining FFR due to the effect of CO<sub>2</sub> on welding arc characteristics. Ar–He–CO<sub>2</sub> mixtures had the most stable FFR's.

(cf. *ISIJ Int.*, **49** (2009), 416)

## Surface Treatment and Corrosion

### Enhancement of erosion resistance on AISI H13 tool steel by oxynitriding treatment

S.-H. CHANG *et al.*

In order to effectively improve erosion resistance and evaluate the effects of an oxy layer on AISI H13 tool steel after the oxynitriding process, this study used three different nitriding surface treatments, namely oxynitriding process 1 (using air), oxynitriding process 2 (using steam) and normal gas nitride. To evaluate the effects of microstructure and the erosion resistance of AISI H13 tool steel after different nitride processes, evaluated micro hardness, erosion tests and SEM microstructure inspections were conducted. Experimental results showed that the oxide layer can protect and improve the aluminum erosion for AISI H13 tool steel. Erosion tests of 2 and 4 h for oxynitriding process 1 could produce a thicker and complex oxide layer, which has higher hardness (HV 1021.9) and optimal weight loss (0.16%). This procedure is proven to effectively reduce the ratio of Al–Fe–Si compounds during the A380 alloy erosion test.

(cf. *ISIJ Int.*, **49** (2009), 421)

### Electrochemical and erosion corrosion aspect on main gas pipe line in sinter plant: for raw petroleum coke as trimming fuel

M. MANNA *et al.*

Different corrosion mechanisms and their extent within a sinter plant gas pipeline were studied. The low temperature-high moisture at the charging side and high temperature-low moisture at the discharging side exist in the main gas pipeline. The waste gas velocity is maximum near charging side and minimum near discharging side. Aggressive conditions near the charging side resulted in faster corrosion than the discharging side. The electrochemical effect was predominant at the wall while erosion effect was significantly higher at the center of the pipe. The overall effect however turns out to be same at wall and center for a particular location of the pipeline. This has been attributed to the velocity effect of waste gases.

The corrosion products were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction XRD techniques. The low temperature-high moisture at the charging side promotes electrochemical

reaction with formation of bigger goethite and hematite. Only, hematite was detected in between charging and discharging side where as different iron oxides and alkali compounds were detected near the discharging side.

(cf. *ISIJ Int.*, **49** (2009), 425)

### Influence of the crack spacing in the coating layer on the progress of interfacial debonding in galvanized steel pulled in tension

S. IWAMOTO *et al.*

The coefficient of thermal expansion of the coating layer of the hot-dipped galvanized steels is higher than that of the substrate steel. Accordingly, the coating layer shows multiple cracking during cooling due to the thermally induced stress. When tensile stress is applied externally on the coated steels, the coating layer exhibits further multiple cracking perpendicular to the tensile direction. As a result, the coating layer is multiply-cracked both in tensile and sample width directions. Under such existent cracks, the coating layer is spalled due to the interfacial debonding induced by the buckling of the coating layer in the sample width direction. In the present work, the influences of the crack spacing in the tensile direction and that in the sample width direction on the spalling process of the coating layer were studied with the finite element stress analysis. The results of analysis showed that (a) the larger the crack spacing of the coating layer in the tensile direction, the more occurs the interfacial debonding and (b) the crack spacing in the tensile direction affects especially on the initial spalling behavior; the larger the spacing, the more the spalling is enhanced. On the other hand, the crack spacing in the sample width direction affects only slightly on the initial spalling behavior, while the larger the spacing, the larger becomes the debonding area in the later stage.

(cf. *ISIJ Int.*, **49** (2009), 431)

## Transformations and Microstructures

### Prediction of precipitation sequences within grains in 18Cr–8Ni austenitic steel by using system free energy method

Y. TODA *et al.*

The applicability of theoretical energy analysis to the evolution of microstructures in heat-resistant steels was explored by using the system free energy method to predict the precipitation of M<sub>23</sub>C<sub>6</sub> and  $\sigma$  phase within grains in 18Cr–8Ni austenitic steels. The chemical free energy of Fe–Cr–Ni quaternary steel and the interfacial and elastic strain energies between austenitic ( $\gamma$ ) matrix and the M<sub>23</sub>C<sub>6</sub> and  $\sigma$  phase were estimated for the system free energy of microstructures wherein coherent or incoherent M<sub>23</sub>C<sub>6</sub> and the incoherent  $\sigma$  phase precipitated within  $\gamma$  grains. By identifying the minimum-energy path through a determination of system free energy hierarchies, the precipitation initiation curves of precipitates in Fe–0.07C–18.95Cr–9.57Ni steel for temperatures between 823–973 K were theoretically predicted. The calculated curves agreed well with experimental results for Type 304H austenitic

steels; this suggests that the system free energy method is suitable for predicting the evolution of microstructures in heat-resistant steels.

(cf. *ISIJ Int.*, **49** (2009), 439)

## Mechanical Properties

### Characterization of grain-boundary precipitates after hot-ductility tests of microalloyed steels

M. VEDANI *et al.*

The hot ductility of microalloyed steels was investigated by interrupted tensile tests at the temperatures of 850 and 950°C. Analyses of microstructural damage during plastic straining of the steels were performed using an experimental setup that allowed rapidly quenching the tensile specimens after straining to a predefined level. Microstructural investigations on the materials were carried out on longitudinally sectioned samples. Further analyses on crack surfaces were performed by fracturing the strained specimens in liquid nitrogen and by analyzing the surfaces formed by high-temperature decohesion through conventional and field emission SEM. It was demonstrated that AlN and Nb(C,N) precipitates, in isolated or combined form, affected the prior-austenite grain boundaries. Differences in hot cracking sensitivity among the steels was accounted for by modifications of the precipitate size and volume fraction.

(cf. *ISIJ Int.*, **49** (2009), 446)

## New Materials and Processes

### Self-propagating high-temperature synthesis of Ti<sub>5</sub>Si<sub>3</sub>/TiAl<sub>3</sub> intermetallics

M. ZHA *et al.*

The products with designed volume ratio of Ti<sub>5</sub>Si<sub>3</sub>:TiAl<sub>3</sub>=1:0, 3:1, 1:1, 1:3 and 0:1 prepared by self-propagating high-temperature synthesis (SHS) were investigated in the present study. The differential thermal analysis (DTA) results suggested that the Si in reactants had a significant influence not only on the melting of Al, but also on the subsequently exothermic reaction between Ti and Al. Also, the presence of Al altered the reaction process of Ti<sub>5</sub>Si<sub>3</sub>. The phase composition of SHS reaction product agreed well with the designed volume ratio of Ti<sub>5</sub>Si<sub>3</sub>:TiAl<sub>3</sub>. Compared with those apparently coarse ones in monolithic Ti<sub>5</sub>Si<sub>3</sub> or TiAl<sub>3</sub>, the grains were considerably refined and microcracks gradually disappeared in products with coexistence of Ti<sub>5</sub>Si<sub>3</sub> and TiAl<sub>3</sub>.

(cf. *ISIJ Int.*, **49** (2009), 453)

### Synthesis of niobium carbides from ferroniobium by mechanochemical method

J. KANO *et al.*

A feasible study on a novel production process of niobium carbide (NbC) from its low cost resource ferroniobium at low temperature was examined by means of mechanochemical and subsequent chemical separation method. NbC powders as well as metallic iron (Fe) were synthesized from ferroniobium (FeNb) and graphite (C) powders by using a

planetary mill at milling speed of more than 600 rpm with FeNb:C molar ratio of 1:1. Then NbC particles were effectively separated from the

mixed powders by leaching with HCl after the heat-treatment of the mixed powders in argon. These experimental results were consistent with the thermo-

dynamic prediction.

(cf. *ISIJ Int.*, **49** (2009), 458)