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Biochemical Analysis of Microbial Adsorption Behavior on Iron and Steel Slag Using DNA-specific Fluorescent Reagent, and the Effect of Microbial Biofilm Attached to Slag on pH Buffering Action

T. TAKAHASHI
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Regular Articles

Fundamentals of High Temperature Processes

Dominant factor affecting reducibility of calcio-wüstite originating from silico-ferrite of calcium and aluminum

M. HAYASHI et al.

The reducibility of calcio-wüstite (CW) originating from silico-ferrite of calcium and aluminum (SFCA) has been investigated from the perspectives of the morphology of SFCA and the concentration of FeO in CW. Two types of SFCA sample were prepared: columnar SFCA and acicular SFCA. The former was synthesized from reagent grade powders of Fe₂O₃, CaCO₃, SiO₂ and Al₂O₃, and contained columnar SFCA grains covered with slag. The latter was synthesized from iron ore and reagent gradeCaCO₃, and contained acicular SFCA grains, which were smaller than the columnar SFCA grains and had fine pores nearby. These samples were reduced in an XRD apparatus for high temperature use in a condition simulating a blast furnace where the oxygen partial pressure was controlled by CO–CO₂ mixtures. The microstructures of the samples before and after reduction were observed by electron probe microanalysis (EPMA). XRD profiles indicated: (i) both SFCA samples were reduced to Fe via CW at 1000°C and (ii) acicular-SFCA-origin CW was reduced to Fe earlier than columnar-SFCA-origin CW, which suggests that the reducibility of acicular-SFCA-origin CW is higher than columnar-SFCA-origin CW. EMPA indicated: (i) most residual parts of acicular-SFCA-origin CW phase kept the morphologic feature of having fine pores as acicular SFCA during reduction as well and (ii) the FeO concentration in acicular-SFCA-origin CW was lower than that in columnar-SFCA-origin CW. Hence, it is concluded that the reducibility of SFCA-origin CW is dominated by the morphology of CW but not by the concentration/activity of FeO in CW.

Ironmaking

Review on the phase equilibria in iron ore sinters

E.-P. HEIKKINEN et al.

Sintering process is a commonly used pre-treatment process for iron containing burden materials with an aim to produce porous, agglomerated sinter material with suitable properties to be charged into the blast furnace. During the sintering process the material undergoes a series of reactions, during which the conditions vary considerably. These changes in temperature and state of oxidation cause changes in the mineralogical composition of the material and although the sintering process does not completely reach the chemical equilibrium, it is important to understand the phase equilibria of the sinter system in order to analyze and control the effect of various factors on the sintering process.

The purpose of this paper is to give a review on the research related to phase equilibria in iron ore sinters. The main components of the sinter are FeO, Fe₂O₃, SiO₂, CaO, Al₂O₃ and MgO and by studying the phase equilibria of this system, the behaviour of sinters can be evaluated. Based on the experimental data, oxide databases have been created to provide thermochemical data of all the necessary compounds within this system. Concerning the solutions, more research is required related to SFCA phases. These databases are commercially available with thermochemistry software and can be used to compute phase diagrams illustrating the effect of different factors on the phase equilibria within the FeO–Fe₂O₃–SiO₂–CaO–Al₂O₃–MgO system. Phase diagrams provide a useful tool to study the behaviour of the material in both sintering process itself as well as in the following reduction processes such as the blast furnace.

Effect of hydrogen concentration in reducing gas on the changes in mineral phases during reduction of iron ore sinter

T. MURAKAMI et al.

In order to decrease CO₂ emission from the ironmaking process, an increasing use of hydrogen in blast furnace (BF) ironmaking is a promising way. In this case, the properties of iron ore sinters such as reducibility and strength need to be optimized because hydrogen reduction of iron oxide is an endothermic reaction and temperature distribution in BF drastically changes. In this study, the effect of hydrogen concentration in the reducing gas on the changes in mineral phases during reduction of iron ore sinter is evaluated. Mineral composition of the ten types of sinter samples was analyzed by XRD and image analysis. Sinter sample was reduced under the simulated conditions such as Low-H₂ (N₂ = 48%(CO + CO₂) – 5.8%(H₂ + H₂O)) and SFCA > L that would occur under equilibrium cooling. The SFCA and SFCA-I phases appear to nucleate preferentially at the interfaces between the magnetite and liquid phases; this phenomenon appears to be associated with common crystallographic features in the magnetite and the SFCA phases. It has also been shown that rapid formation of secondary hematite can take place through the liquid phase assisted oxidation of the primary magnetite grains. The mechanism of this reaction has not been previously reported.

Numerical analysis of raceway formation in isothermal and non-reactive packed bed

S. TAN et al.

Raceway zone is one of the most important regions in blast furnace, since it governs efficiency and stability of the blast furnace process through combustions of coke and auxiliary reducing agent, burden descent, fines generation, and so on. Therefore, quantitative understanding of the raceway behavior is indispensable to designing and realizing highly efficient operation of blast furnace. In the raceway, a cavity is formed and the coke particles circulate due to interaction between particles and high velocity blast gas, and consumption of coke. Thus, two-phase flow behavior is one of the key features of the raceway formation. In this study, the formation behavior of the raceway in the isothermal and non-reactive packed bed was numerically discussed from the viewpoint of two-phase flow. The mathematical model used in this study consisted of Lagrangean particle tracking method and Eulerian computational fluid dynamic technique, and these were combined through voidage distribution and momentum exchange. This model was applied to a small-scale cold-model condition. The simulation results revealed the raceway formation behavior such as variations of cavity size, particle motion, gas flow field, and contact force network. Furthermore, the effects of the initial packing structure on the raceway formation, the differences in the raceway formation behavior in the fixed bed and the moving bed and their mechanisms were clearly elucidated.
High-$H_2$ ($N_2 - 48% (CO + CO_2) - 13% (H_2 + H_2O)$). After reduction, microstructure of the sample was observed. Iron ore sinters usually consist of mineral phases such as hematite, magnetite, calcium-ferrites and slag. Furthermore, calcium-ferrite phases are roughly divided into four types: 1) acicular texture coexisted with primary hematite (1H-ACF), 2) columnar texture coexisted with secondary hematite (2H-CF), 3) small and 4) large columnar textures coexisted with magnetite (M-FCF and M-CCF). An increase in hydrogen concentration of reducing gas accelerates the reduction of hematite, 1H-ACF, and 2H-CF in all sinter samples, while it does not affect the reduction of magnetite, and calcium-ferrite coexisted with magnetite.

(c.f. ISIJ Int., 60 (2020), 2678)

Numerical investigation for the temperature dependency of coke degradation by $CO_2$ gasification reaction in a blast furnace

YUMAZAWA et al.

To quantitatively evaluate the temperature dependency of coke degradation by $CO_2$ gasification reaction in a blast furnace, kinetic analyses of gasification reaction with mass transfer for the coke model with approx. 200 million voxels developed from X-ray CT images at the reaction temperatures of 1,373, 1,573, 1,773 and 1,973 K were performed. At high reaction temperature, the gas concentration of $CO_2$ was high in the external area of the coke model, and the coke matrix voxels vanished mainly around the external surface. Distinguishing surface area of interface between a carbon matrix voxel and a pore voxel with the gas concentration of $CO_2$ at a neighbor pore voxel, although the surface area with the high gas concentration of $CO_2$ accounted for the majority of the total surface area at 1,373 K, the ratio was lower at over 1,573 K than at 1,373 K. In addition to this, from the effectiveness factor of catalyst, the initial rate-controlling step was chemical reaction at 1,373 K but pore diffusion at over 1,573 K. Also, although the frequency distribution of local porosity showed unimodal regardless of the progress of reaction, the standard deviation calculated from the distribution was changed by reaction. The reaction rate of the standard deviation by reaction seemed to be larger at high reaction temperature than at low reaction temperature. The logarithm of the change rate hardly depended on reaction temperature under 1,573 K but was proportional to the inverse of the temperature over 1,573 K. This study quantitatively showed that the rate-controlling step affects the coke structure after reaction largely.

(c.f. ISIJ Int., 60 (2020), 2686)

Numerical approach to comprehend for effect of melts physical properties on iron-slag separation behaviour in self-reducing pellet

K-I. OHNO et al.

A smooth iron-slag separation during ironmaking process is necessary for the steel refining process, even in case of “Zero Carbon Ironmaking”. For a fundamental comprehension of the effect of the physical properties of the melts on the iron-slag separation behaviour, a numerical approach with a practical multi-interfacial smoothed particle hydrodynamics (SPH) simulation for the tracking of the iron-slag separation behaviour is undertaken in this study.

Experimental values for iron-slag separation conditions from a previous work and estimated physical properties from literature were used for the numerical analysis. The CLS-SPH method was able to reproduce the iron-slag separation behaviour where iron aggregated in a unitary sphere and the slag discharged onto the iron surface. A less viscous slag may reduce the negative impact on the separation. A slag with a high surface tension enables the slags to agglomerate and decreases the number of elements that may disturb the iron agglomeration. A highly dense slag has a strong influence on the variation of the iron-slag interface due to a larger momentum. The interfacial tension showed no obvious effect on the separation behaviour in the range of experimental values considered in this study.

(c.f. ISIJ Int., 60 (2020), 2695)

Review Articles

Fundamentals of High Temperature Processes

Review on the high-temperature thermophysical properties of continuous casting mold fluxes for highly alloyed steels

ZHANG et al.

Several recently developed highly alloyed steel grades have shown unsurpassed performance in terms of physical, chemical, and electromagnetic properties. However, broader commercialization of these steels has been hampered by limitations in mold flux performance. Newly developed steels containing considerable amounts of dissolved Al, Mn, and Ti actively react with typical CaO-SiO$_2$-based mold fluxes, which severely changes the composition and subsequently the thermophysical properties of the mold flux that determine the external and internal quality of the as-cast steels. These dynamic changes result in nonuniform heat transfer, lubrication issues, surface defects, and caster breakouts. This work critically assesses the current status of the high-temperature thermophysical properties of CaO-SiO$_2$-based and CaO-Al$_2$O$_3$-based mold fluxes intended for use in casting highly alloyed steel grades. Thermophysical properties, including viscosity, crystallization, thermal conductivity, and heat flux, have been evaluated. The effect of compositional variables including CaO-SiO$_2$, CaO/Al$_2$O$_3$, and Al$_2$O$_3$/SiO$_2$ mass ratios and the additions of CaF$_2$, B$_2$O$_3$, Li$_2$O, K$_2$O, Na$_2$O, TiO$_2$, and BaO on these high-temperature thermophysical properties are discussed.

(c.f. ISIJ Int., 60 (2020), 2705)

Thermodynamic modeling of liquid steel

Y.-B. KANG

Thermodynamic property of liquid steel is often described by activity coefficient of solute in the steel (the other form of partial excess Gibbs energy of the solute). Reliable description of the activity coefficient is required in order to predict equilibrium content of the solute as accurately as possible. In the present article, a number of such approaches are reviewed, with emphasis on basic assumption and inherent character of each formalism/model, and on its applicability at high alloyed liquid steel. Chemical interaction between elements was categorized as weak interaction (i.e., between metal and metal) and strong interaction (i.e., between metal and non-metal). Each formalism/model was analyzed in the view of thermodynamic consistency (Gibbs-Duhem equation and Maxwell’s relation). It is concluded that two issues should be explicitly and simultaneously considered: obeying thermodynamic consistency and treating strong chemical interaction. The former ensures its applicability at higher solute content, and the latter is necessary to properly handle the strong interaction between metallic elements and non-metallic elements, contrary to conventional random mixing assumption.

(c.f. ISIJ Int., 60 (2020), 2717)

Steelmaking

Development and prospects of refining techniques in steelmaking process

N. KIRUCHI

The development of refining techniques in the steelmaking process over the last 60 years and the prospects for the future were reviewed. In Japan, hot metal pretreatment started in the 1960s with the aim of reducing refining costs and improving quality, and its purposes have now transitioned to meeting new requirements for reduced treatment time, reuse of steelmaking slag and use of diverse iron sources. In converter refining, in addition to high speed decarburization, visualization of phenomena and sensing modeling techniques are becoming more important for combined use with data science techniques. In ladle metallurgy (secondary refining), techniques to realize high speed treatment and heating of the molten steel are key issues. The necessity of process revolution to contribute to a sustainable social environment is also discussed briefly.

(c.f. ISIJ Int., 60 (2020), 2731)

 Casting and Solidification

Quantitative phase-field modeling and simulations of solidification microstructures

M. OHNO

This review presents the development of quantitative phase-field models for simulating the formation processes of solidification microstructures, with particular attention to the theoretical foundation and progress in modeling. The symmetry of interpolating functions required to reproduce the free-boundary problem in the thin-interface limit and the necessity of antitrapping current in the diffusion equation are discussed. In addition, new cross-coupling in the phase-field equation for two-sided asymmetric diffusion is briefly described. Recent achievements of large-scale simulations using high-performance computing techniques are explained. Furthermore, some important applications of quantitative phase-field simulations such as investigations of cellular and dendritic growth, microsegregation, and peritectic reaction in carbon steel are discussed.

(c.f. ISIJ Int., 60 (2020), 2745)
Transformation from ferrite to austenite during solidification in peritectic steel systems: an X-ray imaging study

H. Yasuda et al.

X-ray transmission imaging with X-ray diffraction and time-resolved tomography with three-dimensional X-ray diffraction microscopy have been used to observe solidification and transformation in carbon steel and other Fe-based alloys. The imaging techniques showed a massive-like transformation, in which multiple austenite grains were produced in a single δ grain through a solid–solid transformation. The critical velocity from the diffusion-controlled growth of the γ phase to the massive-like transformation was as low as 5 μm/s. X-ray imaging indicated that the δ phase transforms massively to the γ phase in the conventional solidification processes, such as continuous casting. The massive-like transformation and multiple γ grains that were produced in the transformation were related to the subsequent microstructure evolution and casting defect formation. Solidification model including the massive-like transformation is expected to improve our understanding of the solidification and the related phenomena in the peritectic steel systems.

(c.f. ISIJ Int., 60 (2020), 2755)

Regular Articles

Fundamentals of High Temperature Processes

Phase identification of crystal precipitated from molten CaO–SiO2–Fe2O3–P2O5 slag by high-temperature in-situ X-ray diffraction

M. Suzuki et al.

For the first time, we have succeeded in directly identifying the crystalline phase precipitated from the fully liquid slag of the CaO–SiO2–Fe2O3–P2O5 system by high-temperature in-situ X-ray diffraction analysis. Dephosphorization from molten iron can be promoted by 2CaO·SiO2 precitates in molten P2O5 containing slag because they form a solid solution with 3CaO·P2O5. Knowledge of the crystal structure of the 2CaO·SiO2 precipitate is important because it strongly affects the phosphorus solubility. Although it is believed that the α phase of the 2CaO·SiO2–3CaO·P2O5 solid solution precipitates from the molten slag, the crystal structure of the precipitate has not been identified because the crystal structure of the 2CaO·SiO2 compound rapidly changes by phase transition when cooled from high temperature. In this study, slag samples were aerodynamically levitated and completely melted by laser heating under an Ar atmosphere, and then the diffraction patterns were obtained by transmitting a high-energy and high-intensity X-ray beam into the molten slag. We verified that the α→2CaO·SiO2 phase precipitated as the primary crystal phase from molten slag containing 10–30 mass% FeO and 5 mass% P2O5, whereas nagleischmidite precipitated for the molten slag with high P2O5 content. The α→2CaO·SiO2 precipitates contained much higher FeO content than the reported solubility limit, which was supported by the diffraction angles positively deviated from those of the FeO-free α phase in the CaO–SiO2–P2O5 system and chemical analysis of the quenched slag sample.

This excess FeO solute may influence the phosphorus distribution in the α→2CaO·SiO2 precipitates.

(c.f. ISIJ Int., 60 (2020), 2765)

Determination of thermal diffusivity/conductivity of oxide scale formed on steel plate by laser flash method through thermal effusivity measurement by transient hot-strip method

R. Endo et al.

Thermal diffusivity and conductivity were determined for oxide scale formed on steel plate by the laser flash method in combination with thermal effusivity measurement by the transient hot-strip method. The thermal effusivity measurement technique was confirmed by measurement of silica glass, and the value was determined to be 2.52 kJ m−2 s−1/2 K−1 for the oxide scale formed on an ultra-low-carbon steel plate by oxidation at a temperature of 900°C for 360 s. Thermal diffusivity measurements were also conducted for 1 mm-thick steel plates oxidized in air at 900°C for 770–3600 s by the laser flash method. The apparent thermal diffusivity of samples provided the thermal diffusivity of the oxide scale based on three-layered analysis by inputting the measured value of the thermal effusivity. The measured values suggested that no significant boundary resistance exists between the oxide scale and the steel plate. The thermal conductivity and diffusivity of the oxide scale were calculated to be 1.6 W m−1 K−1 and 4.0 × 10−1 m2 s−1, respectively.

(c.f. ISIJ Int., 60 (2020), 2773)

Activities of P2O5 in solid solutions between Dicalcium silicate and Tri-calcium phosphate at 1573 K

T. Saito et al.

The knowledge of P2O5 activities in solid solutions between Ca2SiO4 and Ca3P2O5 is required for more effective dephosphorization. In the present study, firstly, the P2O5 activities in these solid solutions coexisting with CaO or CaSiO3 at 1573 K were measured by equilibrating molten copper with phases under a stream of Ar + H2 + H2O gas mixtures. Subsequently, the sub-regular solution model was applied to Ca2SiO4–Ca3P2O5 solid solutions. The P2O5 activities calculated with the solution model agreed well with the present experimental results and the literature data. It was found that the P2O5 activity in Ca2SiO4–Ca3P2O5 solid solution coexisting with CaO was about 7.5 times lower than that coexisting with CaSiO3.

(c.f. ISIJ Int., 60 (2020), 2780)

Deoxidation equilibria of Fe–Mn–Al melt with Al2O3 or MnAl2O4 at 1573 and 1773 K

R. Nishigaki et al.

Deoxidation equilibria of Fe–Mn–Al melt with Al2O3 or MnAl2O4 were measured at 1573 K. Composition of melts doubly-saturated with Al2O3 and MnAl2O4 were also measured using a crucible comprising these two phases at 1573 or 1773 K. Equilibria with each solid oxide were analyzed using Wagner’s Interaction Parameter Formalism (WIPF).

(c.f. ISIJ Int., 60 (2020), 2794)

Viscosity of slag suspensions with a polar liquid matrix

N. Saito et al.

Under the operating temperatures employed in steelmaking, most slags and fluxes often contain solids, such as undissolved CaO and its reaction products; thus, they are more viscous than their fully liquid states. However, few studies have considered the dielectric interactions of solid particles with the liquid matrix in such systems. In the present study, the viscosity of suspensions of dispersed particles consisting of polyethylene beads in a matrix of silicone oil or aqueous glycerol at room temperature was 1/3 of that at 1873 K. The deoxidation equilibria were reproduced using WIPF at the composition range above 0.1 mass%Al by using α0.1 and 10−1.4 as the equilibrium constant of Al2O3 dissolution reaction, both of which were determined through analysis of measured results for Fe–(20 to 30) mass%Mn melt. In the case of MnAl2O4 saturation, accurate values of equilibrium constant were not obtained because of the relatively significant influence of oxygen analysis error. On the contrary, using compositions doubly-saturated with Al2O3 and MnAl2O4, valid values of the equilibrium constant of MnAl2O4 dissolution reaction, 10−13 and 10−12 at 1873 K and 1773 K, respectively, could be determined.

(c.f. ISIJ Int., 60 (2020), 2787)
measured. Then, empirical models for estimating the viscosity based on the Einstein–Roscov equation were proposed. Furthermore, the viscosity of suspensions of CaO and MgO particles dispersed in a matrix of CaO–Al2O3–SiO2–MgO slag at 1773 K was measured, and the feasibility of the proposed viscosity equations was investigated. As expected, the viscosities of the suspensions of polyethylene beads dispersed in silicone oil and glycerol increased with an increasing bead volume fraction. Under comparable measurement conditions, the viscosities of the glycerol suspensions were higher than those of the silicone oil suspensions. The proposed viscosity models based on the Einstein–Roscov equation and the capillary number reproduced the viscosity of the silicone oil suspensions but underestimated that of the glycerol suspensions. The trend of increasing viscosity of the molten slag suspensions with dispersed CaO and MgO particles was similar to that of the room-temperature suspensions, exhibiting Bingham non-Newtonian behavior. The viscosity model composed with the results from the glycerol aqueous suspensions underestimated the slag viscosity, which can be attributed to the repulsive forces in the high-polarity liquid matrix.

(c.f. ISIJ Int., 60 (2020), 2807)

Steelmaking

Characterization of non-metallic inclusions and clusters during production of low-carbon IF steel

D. GORKUSHA et al.

One of the quality criteria for Interstitial Fee (IF) steels is the metal purity with respect to non-metallic inclusions (NMI), which are harmful for the plastic properties of the material. Furthermore, they cause a formation of surface defects in flat rolled products and reduce the rate of steel casting due to nozzle clogging. This article presents the results of studies on the content, composition, size and morphology of non-metallic inclusions and clusters in steel samples taken during ladle treatment, casting as well as from slabs and steel sheets after rolling of IF steel. The characteristics of NMI and clusters were determined by using conventional two-dimensional quantitative metallographic investigations of polished sections of steel samples (2D method), electrolytic extraction (EE method) of samples followed by investigations of inclusions and clusters by using scanning electron microscopy and energy dispersive spectroscopy and fractional gas analysis (FGA method). By using EE method, different types of inclusions and clusters, their formation, growth and behavior during different stages of IF steel production were studied. The results obtained by the EE method agreed well with the results of the quantitative determination of oxide NMI by using the FGA method. The method of fractional gas analysis shows the dynamics of changes in the content of various types of oxide non-metallic inclusions during ladle treatment and casting of steel. The obtained results can be used to analyze the causes of the formation of harmful NMI in the metal and to optimize ladle treatment of IF steel grades.

(c.f. ISIJ Int., 60 (2020), 2819)

Casting and Solidification

Effect of deoxidizing element on the hot ductility of boron-containing steel

K. TAGUCHI et al.

Improving the surface quality of the casting slab increases the productivity of steel. The slab surface sometimes has transverse cracks along a grain boundary when the slab is bent and/or unbent around the transformation temperature from the austenite to ferrite phases. In particular, for boron-containing steel, the defect is strongly influenced by the precipitation of BN on the grain boundary. In this study, the effect of deoxidizing elements, such as aluminum, calcium, and zirconium, on the hot ductility of boron-containing steel have been investigated fundamentally. The addition of zirconium or calcium improves the hot ductility of boron-containing steel by comparing with that of aluminum. An oxide containing calcium or zirconium acts as a more effective nucleus for BN precipitation than alumina, and the excess precipitation of BN on the grain boundary is suppressed. This improves the hot ductility of boron-containing steel in the region of single-phase austenite. Moreover, the hot ductility of Al2Zr-added steel is the best, even in the region of coexisting austenite and ferrite phases, because the precipitation of ferrite (α-Fe) on the grain boundary is suppressed compared to that of Al1Ca-added steel.

(c.f. ISIJ Int., 60 (2020), 2829)

Optimization of the interfacial properties between mold flux and TiN substrate through the regulation of B2O3

L. ZHOU et al.

Titanium nitride (TiN) inclusions are easy to precipitate in the high temperature processing of titanium alloying steels, which tends to introduce numerous surface defects on the final continuous casting slabs. This study utilizes B2O3 to regulate the interfacial properties between the designed mold fluxes and TiN, with the aim to resolve above problems. The results show that the spreading behavior of the mold flux on the TiN substrate is enhanced, and the interfacial contact angle starts to drop at a lower temperature (from 1473 K for Sample 1 to 1343 K for Sample 4) with the addition of 0–9 wt.% B2O3, as the melting behavior of the designed mold fluxes has been improved. The interfacial reactions between the TiN substrate and molten fluxes are also promoted with the addition of B2O3, where more bubbles are observed in the tested mold flux samples. For Sample 1 without B2O3, quite a few TiN particles couldn’t be dissolved and remain in the matrix phase, where the major formed phase is perovskite (CaTiO3) that would deteriorate the high temperature properties of mold flux severely. However, most TiN particles have been dissolved in the optimized mold fluxes, as major of them have reacted with mold fluxes, resulted in the more generation of titanium oxides phase in the samples. In addition, the calculated phase diagram of CaO–SiO2–TiO2 slag system under different B2O3 contents indicates that the formation and precipitation of CaTiO3 can be effectively inhibited by the addition of B2O3.

(c.f. ISIJ Int., 60 (2020), 2838)

Chemical and Physical Analysis

Control of laser focal point by using an electrically tunable lens in laser-induced plasma optical emission spectrometry

Y. FUGANE et al.

This paper suggests a method to control the focal point of laser on the focus position of a sample surface automatically in laser-induced breakdown spectrometry (LIBS). For this purpose, an electrically tunable plano-convex lens was installed in a laser irradiation system, where it could vary the focal length of laser with a long working distance and a rapid response time, and the focal length could be periodically varied with a triangle waveform. Because the tunable lens was easily handled and inexpensive, the laser system could be modified with a low cost, as compared with commercial apparatuses having complicated optics to control the position of laser irradiation. A piece of scrapped stainless steel was selected to determine whether it was and had some roughness was investigated as a test specimen. A satisfactory result was obtained such that the plasma could be generated uniformly and firmly along a laser trace on the sample surface and thus could give the emission signal with a sufficient precision. The driving frequency of the tunable lens, which controlled a repetition period of the laser beam, was optimized to be 10 Hz when the scan rate of laser was fixed at 3.0 mm/s. As a result, it is expected that the LIBS system with the tunable lens can be applied to actual on-site/in-line analysis in material production.

(c.f. ISIJ Int., 60 (2020), 2845)

Accuracy improvement of the XRD–Rietveld method for the quantification of crystalline phases in iron sintered ores through the correction of micro-absorption effects

T. HIRANNO et al.

The mass fraction of each crystalline phase in inorganic materials can be investigated using the Rietveld refinement of the X-ray diffraction (XRD) patterns. For quantitative analysis, differences in the values of the linear absorption coefficient, μ, among the crystalline phases must be considered when certain X-ray sources are used, because such differences often affect their mass fractions. Herein, we evaluate the effects of the differences between the Cu and Co Kα X-rays on the mass fractions of the crystalline phases in iron sintered ores using the XRD–Rietveld method by performing two types of XRD measurements. Type 1 samples modeled materials with two different particle size combinations of α-Fe2O3 and ZnO. Type 2 samples used powder mixtures to simulate iron sintered ores composed of α-Fe2O3, and synthesized SFCA and SFCA-1 in various mass fractions. Moreover, a correction method was developed using the Taylor–Matulis (TM) correction that considers the μ of each phase and the average particle diameter R of each crystalline phase determined by scanning electron microscopy with energy dispersive spectroscopy. For type 1 samples, results that were in good agreement with the initially-charged mass fractions could be obtained using the TM correction, even in the presence of significant differences in R between α-Fe2O3 and ZnO. The results for type 2 samples confirmed...
that quantitatively accurate mass fractions could be obtained using the TM correction with an accuracy of approximately ±3 mass% for Cu and Co sources, whereas the error was greater than ±3 mass% for Cu source when the TM correction was not applied. (cf. ISIJ Int., 60 (2020), 2851)

Social and Environmental Engineering

Kinetic analysis considering particle size distribution on Ca elution from slags in CaO–SiO2–MgO–Al2O3–Fe2O3 system
Y.KASHIWAI et al.

The understanding of the behavior of alkali elution from slags is important for their effective recycling and utilization. In a previous study, it was reported that the addition of iron oxide to steel slags significantly inhibited alkali elution. A lower modified basicity, i.e., CaO/(SiO2 + Fe2O3) ratio, indicates a lower alkali elution from the slags. In addition to the effect of Fe2O3 content, particle size distribution is an important parameter to determine the elution of Ca quantitatively.

In this study, a kinetic model considering particle size distribution is developed and applied to the results of a dissolution experiment using samples of a slag, which is designated as SlagF4, with different particle size distributions. In the new kinetic model, an effective surface area \( S_{\text{eff}} \) and the effectiveness of total surface area \( \alpha \) are introduced, and a kinetic analysis is performed.

\[
\left[ \frac{\text{Ca}^{2+}}{\text{L}} \right] \text{mod} = \sum \left( \frac{n_a}{\left( \text{Ca}^{2+} \right)^{-1}} \right) \\
= \sum \left( \frac{\text{Ca}^{2+}}{16.0 \left( 1 - \exp \left(-S_{\text{eff}} / k_{\text{L}} \right) \right)} \right) \\
S_{\text{eff}} = \exp \left( \beta_1 n_a S_a \right) \quad \beta_1 = 23.4 \\
\text{Ca}^{2+} = \alpha n_a S_a, \quad \alpha = 3 \times 10^{-6}
\]

The rate constant \( k \) obtained is a fixed value for one sample type; \( k \) decreased with the increase in Fe2O3 content.

The values of \( \alpha \) increased significantly with the increase in the total surface area \( S_T \) (i.e., a decrease in particle diameter) in different particle size distributions. It is discovered that \( \alpha \) typically represents the Ca elution tendency. Additionally, the change in \( \alpha \) is small with the change in the Fe2O3 content. It is demonstrated that the developed kinetic model is valid for the analysis of Ca elution for samples with different particle size distributions. (cf. ISIJ Int., 60 (2020), 2859)

Steelmaking

Development of metal supported SOEC for carbon recycling iron making system
H.TAKASU et al.

A new solid oxide electrolysis cell (SOEC)—a metal-supported SOEC (MS-SOEC) where the SOEC is structured on a metal support and is capable of extending the cell surface area to a wider extent than a conventional ceramics base SOEC—was designed for carbon dioxide (CO2) recycling in the ironmaking process. The MS-SOEC demonstrated CO2 reduction and CO and oxygen production properties. The possibility of carbon cycling was examined with the CO2 resource utilization technology using MS-SOEC and its application to the iron-making process. The required cell area of MS-SOEC for an iACRES, which combines an active carbon recycling energy system (ACRES) and a steelmaking process, was estimated using experimental results. By improving the performance of the cell, MS-SOEC was expected to be applied to a carbon recycling ironmaking system that could contribute to the establishment of a zero-emission CO2 iron making system. (cf. ISIJ Int., 60 (2020), 2870)

Instrumentation, Control and System Engineering

Development of a shape meter employing the LED dot pattern projection method for a hot strip finishing mill
Y.ISEI et al.

In recent years, to improve the fuel efficiency of automobiles by reducing their weight while maintaining their strength, smaller-thickness and higher-strength steel sheets tend to be used as automobiles’ construction materials. For stable and accurate production of these sheets, it is crucial for them to be flattened through the hot strip rolling process. Therefore, to realize accurate automatic flatness control (AFC), a new shape meter that employed the light-emitting diode (LED) dot pattern projection method was developed. This consists of an LED dot pattern projector that can project the staggered periodic dot pattern, made of 1 200 power LED chips, on the rolled strip and area camera that captures the image of the projected pattern. Then, instantaneous strip flatness is measured to analyze the pattern pitch correlation with inclination angle. The shape meter was installed at the hot strip finishing mill’s exit, and its measurement accuracy and stability were evaluated. As a result, its inclination angle measurement error was within 0.45 degrees (two sigma) when compared to the set angle of the standard target, and the measured flatness of the rolling strip was consistent with the visually observed one. Its measurement success rate per entire coil was above 98.5%. These results indicated that the developed shape meter could be applied to the AFC. In addition, applying the measured flatness to the AFC of the work roll bender and leveling, it was confirmed that the strip flatness was improved in a short time. (cf. ISIJ Int., 60 (2020), 2876)

Dynamic control of flatness and elongation of the strip in a skin pass mill
T.OGASAHARA et al.

This paper proposes a dynamic control method for strip flatness and elongation in a skin pass mill. In conventional feedback control, the target values of flatness and elongation are fixed. However, elongation control to a fixed target value is often insufficient to achieve strip flatness when rolling force manipulation for elongation control causes work roll deflection. To improve flatness control performance, we propose an elongation control method which considers flatness. In the proposed method, an optimization problem is solved periodically. The objective function, including flatness control error, is minimized subject to constraints such as the range of elongation and strip thickness and the control outputs. A feedforward control method which suppresses elongation deviations during mill speed changes is also proposed. Accurate online prediction of rolling force using a physical model is difficult due to the heavy computational load. The proposed method utilizes the relationship between the strip strain rate and deformation resistance, which is measured offline, and the designated rolling force change from low to top rolling speed, and does not require a physical model or heavy computation load. An evaluation by simulation and experiments showed that the proposed method improves flatness and elongation control performance. (cf. ISIJ Int., 60 (2020), 2886)

Formulation and Processing Thermomechanical Treatment

Formulation of a generalized flow curve for 0.2% carbon steel under high-speed hot forming conditions by a regression method
H.-W.PARK et al.

A precise flow curve for a wide range of forming conditions is important for accurately predicting forming force. Moreover, since the flow curve reflects microstructural changes, its accurate description must be obtained under various temperatures and strain rates up to 300 s⁻¹. For practical forming processes such as hot strip rolling and wire rod rolling, the deformation behavior at high strain rates (50–200 s⁻¹) must also be studied. However, a uniform axial high strain rate is difficult to achieve. Hence, a new deceleration method is developed. Also, the compression test at high strain rates is accompanied by marked internal heat generation. Therefore, temperature and deformation are highly inhomogeneous compared with those in tests at lower strain rates. In addition to this problem, heat conduction to the die and friction should be corrected using inverse analysis. By considering the internal temperature increase effect at high strain rates, the uniaxial flow curve obtained using inverse analysis is shown to be greater than the experimental apparent stress–axial strain curve. And then, a regression method is applied to obtain a generalized flow curve at high strain rates, which can cover wider ranges of strain rates and temperatures. Finally, they are compared with an extrapolated flow curve that is regressed using an intermediate strain rate in our previous research. By comparing those results, the extrapolated flow curve is greatly different from the flow curve obtained in the current research. To find the reason for the difference, a microstructure analysis using EBSD is implemented. (cf. ISIJ Int., 60 (2020), 2896)

Uniform hot compression of nickel-based superalloy 720Li under isotothermal and low friction conditions
S.HORIKOSHI et al.

Isothermal compression tests at 1000°C and 0.1 s⁻¹ strain rate, in which mica or glass sheets were used as a lubricant, were conducted. Isothermal con-
dation was achieved by placing high-heat-resistant (HHR) alloys between a workpiece and ceramic tools in the induction-heating configuration to prevent heat from escaping to the ceramic tools. To perform high compression tests, it was necessary to increase the diameter of the HHR alloy, for which a new single-turn coil was designed using FEM calculation coupled with deformation-temperature electromagnetic fields. In order to obtain the correct flow stress, inverse analysis was conducted using the FEM calculation, in which temperature and strain rate fluctuations were compensated. However, the compensation was insufficient when the distribution of temperature and strain rate was large. The use of glass sheets as a lubricant considerably reduced friction and uniform deformation was achieved. Thus, flow stress obtained using the inverse analysis became extremely reliable. The flow stress of mica obtained using inverse analysis with a constant friction coefficient was different from that of glass. Introducing new friction model that the friction coefficient changed from 0.02 to 0.3, the flow stress of mica was consistent with that of glass. Therefore, the flow stress obtained using the inverse analysis for the new configuration proposed in this study proved to be reliable.

(After) Impact of the different friction coefficients on the tools on the mechanics of the Mannesmann 2-roll tube piercing M. FERNANDES et al.

A numerical parametric study on friction in cross-roll tube piercing is reported in this paper, in order to assess the role of the different friction coefficients on the different parts of the complex tooling of this process (friction on cross-rolls, Diescher disks, piercing plug). Their effects on entrainment speed, state of strain and stress are quantitatively evaluated using the 3D Finite Element Method (FEM). This knowledge allows measures to be taken in case of friction-dependent defects occurring on the piercing mill. Simple regression formulae are proposed which highlight which friction coefficient (s) most impact feed efficiency, twist angle, piercing plug force and torques on the different tools. Based on these relations, a strategy is developed, involving measurements to be performed and equations to be used for an unambiguous friction coefficients identification procedure.

(Cf. ISIJ Int., 60 (2020), 2917)

Calibration of distortional plasticity framework and application to U-draw bending simulations S.-Y. LEE et al.

A new version of a distortional plasticity framework, the so-called homogeneous anisotropic hardening (HAI), was investigated regarding its calibration and its application to the numerical analysis of the U-draw bend test. First, the mechanical properties of a dual-phase steel sheet sample, DP780, were characterized using uniaxial tension, bulge and elastic loading-unloading tests to provide the data for the calibration of a conventional constitutive description assuming isotropic hardening. Then, tension-compression with different load reversal numbers and two-step tension tests were performed to produce strain path changes that lead to anisotropic hardening effects. The coefficients of this new model, called HAH, were determined with an optimization procedure. Several sets of coefficients were identified depending on the number of reversals considered in tension-compression. U-draw bend test simulations were also carried out to validate the HAH model and its implementation in a finite element code, to compare the results with an older version of the HAH model family, and to assess the influence of the input data used in the coefficient calibration. In addition, the influence of the cross-loading effect on the U-draw bending predictions was examined. The main conclusion of this work is that the HAH coefficients calibrated for the description of reverse loading depend on the number of reversals during the tension-compression tests. The results of the U-draw bending simulations indicate that this dependence may lead to significant differences in the predicted amounts of springback.

(Cf. ISIJ Int., 60 (2020), 2927)

Review Article

Transformations and Microstructures

Interaction of alloying elements with migrating ferrite/austenite interface G. MIYAMOTO et al.

Alloying elements greatly influence phase transformation kinetics in steel due to local partitioning and interfacial segregation to migrating interface and quantitative understanding of the alloying effects is a key for tailoring mechanical properties of modern high strength steels. Energy dissipation for interface migration is an important concept to understand the alloying effects and also to control carbon enrichment in untransformed austenite in multiphase high strength STRIP or DP steels. In this review, possible sources causing energy dissipation for interface migration are introduced and the energy dissipation for interface migration in formations of grain boundary ferrite (gb), Widmanstatten α and bainitic α investigated in various systems are summarized.

(Cf. ISIJ Int., 60 (2020), 2942)

Regular Articles

Transformations and Microstructures

Control of core-shell type second phase formed via interrupted quenching and intercritical annealing in a medium manganese steel T. TSUCHIYAMA et al.

Medium manganese steel (Fe-5.0%Mn-1.2%Si-0.10%Al alloy) was subjected to interrupted quenching from the austenite single-phase region to a temperature between Mₐ and Mₜ followed by intercritical annealing in the ferrite and austenite dual-phase region at 923 K. As a result, a core-shell type second phase, which consisted of a fresh martensite core surrounded by a film-like retained austenite shell, was formed. The mechanism and kinetics of reversion for the interrupted-quenched specimens were analyzed with DICTRA simulation and TEM observation. With regard to the effect of the core-shell type secondary phase on mechanical properties, it was inferred that the fresh martensite core functioned as a hard second phase and enhanced work hardening by stress partitioning similar to DP steel, while the film-like retained austenite contributed to improved ductility due to the TRIP effect. As the interrupted quenching temperature decreased, the volume fraction of the fresh martensite core decreased, while the stability of the retained austenite shell increased. This showed potential for controlling the strength and ductility balance of medium manganese steel. A possible beneficial effect of the core-shell type second phase on the ductile fracture behavior was also discussed in terms of stress/strain relaxation at the interfaces between hard martensite and ferrite matrix.

(c.f. ISIJ Int., 60 (2020), 2954)

Electron theory calculation of thermodynamic properties of steels and its application to theoretical phase diagram of the Fe–Mo–B ternary system M. ENOKI et al.

In this study, the ground structures of the Fe–Mo–B ternary systems were estimated by first-principles calculations based on genetic algorithm, and the free energies of their structures are evaluated by electronic calculations and statistical thermodynamic techniques. In addition, the phase diagram at finite temperature was theoretically constructed using the calculated free energies, and the result was compared with the experimental knowledge. The space groups and compositions of many ground structures obtained by the calculations correspond well with the experimental findings, but the agreement is not perfect. However, by including metastable structures by only a few kJ/mol than the ground state, it becomes clear that the appearance of almost all structures can be predicted based on this technique. The new calculation technique of such theoretical phase diagrams suggested in the present study is expected to open up the possibility of estimation of unknown phase diagram, reexamination of experimental phase diagrams and discovery of new phases. On the other hand, examining the calculation conditions for improving the accuracy of energy calculation, consideration of the anharmonicity of atomic vibration, magnetic entropy effect, handling of solid solution, etc. are mentioned as problems requiring further consideration.

(c.f. ISIJ Int., 60 (2020), 2963)

Texture formation in a polycrystalline Fe–Ni–Co–Al–Ti–B shape memory alloy D. LEE et al.

In polycrystalline Fe-Ni-Co-Al-based shape memory alloys, control of the recrystallization texture is significantly important to improve the ductility by suppressing the brittle precipitates of the β-2 phase at grain boundaries during aging treatment. In this paper, the texture evolution in the recrystallization process was systematically investigated by means of the electron backscatter diffraction (EBSD) method in an Fe–Ni–Co–Al–Ti–B polycrystalline alloy. The development of a [110]<112> texture was confirmed in the 98.5% cold-rolled sheet specimen. After primary recrystallization annealing, the γ matrix containing the β phase showed the continuous recrystallization remaining in the same orientation with a
deformation texture after annealing at 1 000°C. Grain growth of the γ phase was interrupted by the β phase. Then, abnormal grain growth of {210}<001> grains began occurring in concurrence with the dissolution of the β phase and the main recrystallization texture changed from {110}<112> to {210}<001> at temperatures higher than 1 100°C. (cf. ISIJ Int., 60 (2020), 2973)

Reduction of thermal expansion of ferritic/martensitic heat resistant steels -Alloying effects on thermal expansion of α-Fe phase-
S.KOBAYASHI et al.

Alloying effects on the thermal expansion of the α-Fe phase in connection with the magnetic states were investigated with a final goal to design ferritic/martensitic heat resistant steels with a reduced thermal expansion coefficient at high temperatures. The thermal expansion coefficient decreases with increasing the content of the alloying elements M (M = Co, Cr and V) regardless of the types of the elements in the ferromagnetic state well below the curie temperature in each alloy. The thermal expansion coefficient increases with temperature more significantly in the paramagnetic states compared to the ferromagnetic states. The temperature dependence of the thermal expansion coefficient in the paramagnetic states is not much influenced by the type of alloying element. As a result, the lower the Tc is, the higher the thermal expansion coefficient tends to become in the para-magnetic states at high temperatures. Based on the results obtained, a way is proposed to design heat resistant ferritic steels with reduced thermal expansion coefficients; to add both Cr and Co as major alloying elements such that the Tc and the A3 temperatures are placed above their service temperature. (cf. ISIJ Int., 60 (2020), 2983)

Limiting retained austenite decomposition in quenched and tempered steels: Influences of rapid tempering and silicon
V.K.EUSER et al.

Tempering reactions are critical to microstructure and property control in martensitic steels. Here, retained austenite decomposition and cementite precipitation are monitored using Mössbauer spectroscopy in 4340 and 300-M steel under conventional and rapid tempering conditions. Tempering times are compared at a constant tempered hardness by increasing tempering temperatures associated with short time conditions to achieve equivalent matrix softening to that of longer tempering times. Time-temperature combinations that provide equivalent tempered hardness generated microstructures with similar dislocation densities and cementite precipitation fractions; these mechanisms are controlled by self-diffusion. However, systematic differences in retained austenite content were observed at a given degree of softening, where shorter tempering times exhibited higher levels of retained austenite compared to more conventional conditions. At low temperatures, the differences in retained austenite preservation between explored time-temperature conditions are attributed to corresponding differences in carbon diffusion distance (in austenite), the controlling diffusional process of retained austenite decomposition. At higher temperatures, retained austenite decomposition exhibits C-curve kinetic behavior in 4340. Thus, reduced thermodynamic driving force for cementite and ferrite formation at higher temperature is believed to play a role in restricting retained austenite decomposition within some short-time, high temperature tempering regimes. The addition of silicon pushes cementite precipitation and retained austenite decomposition to higher temperatures, although retained austenite decomposition is suppressed to a greater extent than cementite precipitation. Potential is illustrated for coupling rapid tempering with silicon alloying to produce appreciably tempered martensite (~490 HV) with relatively less retained austenite decomposition compared to conventional tempering conditions. (cf. ISIJ Int., 60 (2020), 2990)

Editors' postscript
M.SUSA and J.YANAGIMOTO

(cf. ISIJ Int., 60 (2020), 3001)
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【研究内容】

1. 常見 祐介
2. 堤 成一郎
3. 土山 聡宏

【分析方法】

1. DF/mWH (寄) 真空浸炭およびFe-N二元フェライト鋼における相分率解析
2. 肌焼鋼の潤滑下における摩擦特性
3. ロセスにおけるスラグの亀裂生成挙動
4. 焼戻し軟化抵抗のピーク分離手法の汎用性と水素存在状態の定量的解析
5. 伸線パーライト鋼の水素脆化の異方性評価
6. 素添加の影響
7. 有中炭素マルテンサイト鋼の機械特性に及ぼす
8. 材の微細組織と機械的性質

【結論】

1. 設計された0.3 mass%炭素鋼の微細組織
2. 剛性の高次応力と弾性の関係
3. 弹性引き裂の影響
4. 焼戻し軟化抵抗のピーク分離手法の汎用性と水素存在状態の定量的解析
5. 伸線パーライト鋼の水素脆化の異方性評価
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なお、本特集号は、「腐食劣化解析に基づく鋼構造物維持の最適化」研究会の研究成果を広く発信する場にもさせて頂くことを申し添えます。

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様々な環境における腐食挙動解析や電気化学計測、環境センシング技術、数理解析法を含めた鋼構造物維持の最適化に関する最新の研究を広く取り上げる。

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