

Fundamentals of High Temperature Processes

A physical modeling study of fluid-flow phenomena in gas-slag systems

A.KAPOOR *et al.*

With the advent of more intense iron and steelmaking processes like smelting-reduction, gas evolution in the slag reaches unprecedented levels. In order to understand the fluid mechanics associated with gas-slag systems, gases were injected into a variety of room-temperature liquids at high superficial velocity (0.18 to 4.0 m/s). The void fraction was measured with a gamma ray densitometer. A churn-turbulent flow behavior was obtained in all gas-liquid systems. Drift-flux models proved useful in the analysis of flow behavior. Drift-flux analysis of available smelter data is also consistent with the present data.

Thermodynamics of the MnO-SiO₂-TiO₂ system at 1 673 K

M.ITO *et al.*

The isothermal phase relations for the MnO-SiO₂-TiO₂ system have been investigated at 1 673 K by a chemical equilibration technique. Iso-activity curves for MnO are drawn from the experimental results of the activities as well as those for SiO₂ and TiO₂ calculated by Schuhmann's tangent intercept procedure. Iso-activity curves are nearly parallel to the saturation lines for the respective components. In this system, the interaction between MnO and SiO₂ is the strongest of all.

The sulfide capacity mostly depends on MnO content in this ternary system. It slightly increases when SiO₂ is replaced by TiO₂ at a constant (MnO+MnS) content by molar basis, whereas γ MnS considerably decreases.

Ironmaking and Reduction

Stepwise reduction of CaO and/or MgO doped-Fe₂O₃ compacts to magnetite then subsequently to iron at 1 173 - 1 473 K

A.A.EL-GEASSY *et al.*

Compacts of pure Fe₂O₃ and Fe₂O₃-doped with either of 1.0 %CaO and/or 1.0 % MgO, sintered at 1 473 K for 20h, were isothermally reduced at 1 173-1 473 K with 10%CO-90%CO₂ to magnetite then to metallic iron with purified CO. The oxygen weight-loss resulted from Fe₂O₃, Fe₃O₄ or Fe₃O₄-Fe reduction steps was continuously recorded as a function of time. Chemical and X-ray analyses, microscope examination and pore size analyzer were used to characterize the fired and reduced compacts. The influence of CaO and/or MgO on the reduction behaviour of Fe₂O₃ and Fe₃O₄ was intensively studied. The reduction mechanisms predicted from both of apparent activation energy values and heterogeneous gas-solid mathematical models were correlated with the microstructur-

es of partially reduced samples. The results obtained showed that the doping of these fluxing oxides promoted the reduction of Fe₂O₃ at 1 173 - 1 473 K. The reduction of pure and doped Fe₂O₃ compacts is controlled by gaseous diffusion at early stages and by interfacial chemical reaction at later stages. In Fe₃O₄-Fe reduction step, the doping of CaO and/or MgO enhanced the reduction at early stages which is temperature and compact composition dependent. With progress in reduction, the presence of MgO retarded the reduction of Fe₃O₄ at \leq 1273 K resulting a slowing down in the rate at latter stages. This was attributed to formation of entrapped lower oxide relics which hindered gaseous diffusion. At early stages, the reduction of pure and doped Fe₃O₄ compacts is controlled by mixed control reaction mechanism. At latter stages, interfacial chemical reaction is the rate determining step for pure and CaO-containing samples, whereas solid-state diffusion is the rate controlling step for MgO-doped compacts.

Oxidation and sintering of magnetite ore under oxidising conditions

L.YANG *et al.*

The oxidation of magnetite mineral in a wide range of iron ore concentrates has been studied at sintering conditions. The degree of oxidation for a given ore closely depends on temperature, oxygen partial pressure of the atmosphere and reaction time. With increasing temperature the degree of oxidation increases and then declines because of particle coalescence and melt formation resulting in reduced porosity. For the same reasons, a lower oxidation degree is observed for more fusible ores at high temperatures. The oxygen partial pressure in atmosphere has been shown to strongly influence oxidation, particularly when p_{O_2} is below air. Measures to enhance magnetite oxidation during sintering are discussed based on current understanding of the fundamentals that control the sintering process.

The study has also shown that during sintering, the preferred bonding phase in sinter, *i.e.*, SFCA, forms readily once magnetite is oxidised. The sintering behaviour of the oxidised magnetite is essentially similar to that of a natural hematite ore. However, silicates are still the dominant bonding phase for highly fusible magnetite ore sintered in an oxidising atmosphere.

Casting and Solidification

Recovery of hot ductility by improving thermal pattern of continuously cast low carbon and ultra low carbon steel slabs for hot direct rolling

M.SUZUKI *et al.*

Continuously cast low carbon and ultra low carbon steel slabs with low Mn/S ratio processed *via* hot direct rolling route tend to form more surface defects than those *via* hot charge

rolling route. The slabs low in Mn/S ratio exhibit poor ductility during hot direct rolling at elevated rolling temperatures and high strain rates. Various thermal patterns have been investigated with hot tensile test to find a suitable one to recover the hot ductility of these slabs in hot direct rolling. When they are once cooled to α -ferrite temperature range and subsequently reheated to austenitic temperature range, remarkable recovery of the hot ductility has been found to occur. This recovery is attributed to the recrystallization of prior austenitic grains which transform first into α -ferrites then to new austenitic grains, leaving manganese sulfides which existed at the prior austenite boundaries not to precipitate at the recrystallized new austenite boundaries.

Analysis and Characterization

Analysis of grain colonies in type 430 ferritic stainless steels by Electron Back Scattering Diffraction (EBSD)

M.BROCHU *et al.*

Ridging is a serious problem which occurs in ferritic stainless steel after tensile deformation in the rolling direction, and steel makers are attempting to respond to the surface deterioration caused by this phenomenon. Many authors have suggested that a long distance order in the lattice orientation of a sheet could be responsible for ridging. In this study, the authors performed local orientation measurement on two samples of type 430 stainless steel using orientation imaging microscopy, and analyzed the grain orientations of the RD and ND planes. Data on areas covering more than 5mm² are presented here for the first time in the literature. The experimental results prove the existence of grain bands in the ND plane of samples with severe ridging. However, the texture maps for the RD planes are in disagreement with many previously proposed theories. Therefore, a new mathematical model is introduced. The simulated ridging profiles of the samples calculated with this new model agree with the experimental results. Finally, a method of amelioration that can significantly improve the theoretical model is proposed.

Determination of phosphorus content in steels using flow injection into an argon - water carrier for inductively coupled plasma - mass spectrometry

A.G.COEDO *et al.*

This paper describes a methodology for the determination of phosphorus in steels using argon-water carrier, in flow injection with Inductively Coupled Plasma Mass Spectrometry. ³¹P is a monoisotopic element and its determination, at very low levels, is very difficult due to contamination problems and interference inherent in the plasma and solvents. These unduly raise the background being necessary a sample pretreatment step to achieve the

required contents. To dissolve the samples in this study, a microwave digestion system was applied to minimize the sample preparation time, the quantities of reagents and the risk of contamination. A mercury cathode electrolysis was used for performing a rapid matrix removal/analyte enrichment. The use of an argon-water stream as carrier yielded an enhancement in sensitivity and the detection limit was improved by a factor of approximately 2.5 when compared with that experienced using flow injection into an all-water carrier. Optimization of the operating conditions for the argon-water flow injection and for Inductively Coupled Plasma Mass Spectrometer, were selected with respect to the peak height sensitivity and were found to be dependent on the nebulizer gas flow-rate, the radio frequency power, the sample injection volume and the carrier flow-rate. The detection limit based on a sample rate of 0.02 g ml^{-1} was $0.4 \mu\text{g.g}^{-1}$ and the relative standard deviations at 100 and $10 \mu\text{g.l}^{-1}$ P solution were 2.0 and 15 % respectively. Data are presented for 3 Standard Reference Materials: JSS 003-2, (from Japanese Iron and Steel Certified Reference Materials); and ECRM_s 285-1 and 278-1 (from Bureau of Analysed Samples LTD).

Rapid analysis of steel by inductively coupled plasma-atomic emission spectrometry and -mass spectrometry with laser ablation solid sampling

Y. ISHIBASHI

A cw-Q-switched Nd-YAG laser was used in a laser ablation (LA) system for rapid analysis of steel using inductively coupled plasma atomic emission spectrometry (I CP-MS) and inductively coupled plasma mass spectrometry (I CP-MS). The problems accompanying laser ablation (LA) such as the quantity of fine particles produced by LA for different steel samples were resolved by adjusting the laser radiation conditions. Also the contamination from impurities in the argon gas used to produce the I CP was reduced by a zirconium purifier. A rapid method with high accuracy was established for steel analysis. The LA-I CP-A ES is very useful for rapid and direct steel analysis in the process of steel production control.

On the other hand, the LA-I CP-MS has precisions ranging from 5% to 10% in relative standard deviation ($n=10$) for most of the elements in steels. LA-I CP-MS without standards was found to have sufficient accuracy for semi-quantitative and direct steel analysis.

Forming Processing and Construction

Influences of expansion ratio on inside bore and lamination defects in expansion piercing for materials with poor hot workability

C. HAYASHI *et al.*

In addition to the super piercer, "the new super piercer" which allows expansion pier-

cing has been developed by the authors. The development of the skewing technology for disc roll axes and its application to the cone-type piercing technology results in remarkable increase in the expansion ratio. And then, studied in detail were the influences of the expansion ratio, feed and cross angle on the rotary forging effects, redundant shear deformations and power consumption in the expansion piercing process. Noteworthy is that the larger the expansion ratio is, the more remarkable is the decrease in the number of rotary forging times. Accordingly, the larger the expansion ratio is, the more remarkable is the increase in the elongation value at the tip of the plug. Namely, when the expansion ratio becomes larger, any possible cause of the initiation of inside bore defects can be eliminated.

In order to confirm the above mentioned effects, the influences of the expansion ratio, heating temperature, feed and cross angle on the inside bore defects in materials with poor hot workability were studied in this paper.

Development of manufacturing process of clad bar by rotary rolling

K. NAKASUJI *et al.*

Recently, it has become increasingly difficult for only a single metallic material to meet the demand, which asks for the effective characteristics, such as corrosion resistance, high strength and electric conductivity, at the same time. The development of clad material, which consists of two or more metals, has arisen, in which the clad material has developed advanced functional properties by utilizing the property of each metal. Clad bars have been manufactured by swaging, hydrostatic extrusion, groove rolling methods and so on. However, it is difficult to produce the clad bar with a high dimensional accuracy efficiently. The development of a method for manufacturing a clad bar with diffusion bonding interface by hot rolling has been highly sought.

In this paper, first, the diffusion bonding methods by hot working, such as hydrostatic extrusion, groove rolling and rotary rolling methods, are studied comparatively. Next, in the diffusion bonding method by rotary rolling, the combination of metallic material, the feature of diffusion bonding, the metal flow at bonding portion and the thickness of the diffusion layer are investigated in detail. This diffusion bonding method can be carried out by the heavy reduction rolling which is a symmetric working method.

Surface Science and Technology

Characterization of the Fe-Al inhibition layer formed in the initial stages of hot-dip galvannealing

Y. MORIMOTO *et al.*

Due to the lack of detailed information surrounding the inhibition phenomenon in hot-dip galvannealing, and the microstructure and

morphology of the Fe-Al inhibition compounds, this study was initiated to investigate the inhibition layer formed under the low bath Al content, short-dipping-time conditions typical of commercial galvannealing. In this paper, Fe-Al inhibition layer formation on samples produced in a 0.14 mass% Al-bearing Zn bath on a Ti IF, Ti-Nb IF and a P-added Ti IF substrate was studied using a technique combining both SEM and TEM. The inhibition layer on the Ti IF and Ti-Nb IF substrates was composed of both Fe_2Al_3 and FeAl_3 . The inhibition layer on the P-added substrate was composed primarily of FeAl_3 with individual grains of Fe_2Al_3 observed between the FeAl_3 and the steel substrate. A possible formation mechanisms for the inhibition layer is speculated based upon the TEM observations of the morphology of the layer.

Microstructure

Crystallographical analysis on martensite transformation and martensitic morphology

G. NANJU *et al.*

The strain tensor and displacement vector associated with lattice deformation were introduced to discuss the effect of self-accommodation and plastic accommodation in martensitic transformation and the crystallographic models for $\{557\}f$, $\{225\}f$ and $\{31015\}f$ martensite were proposed, along with their experimental verification. According to the present work, the authors suggest that the invariant habit plane is formed by the self-accommodation between different martensite variants with the internal twin substructure, when the strength of parent phase is so high that the plastic accommodation is difficult to occur. However, when the strength of parent phase is low enough for the plastic accommodation to occur, the dislocation substructure in the martensite may be produced, and the invariant habit plane is difficult to form, so the $\{225\}f$ and $\{557\}f$ habit planes are difficult to be explained by phenomenological crystallographical theory.

Furthermore, the authors suggest that the martensitic morphology depends on the competition between self-accommodation and plastic accommodation, and also depends on the factors of not only the strength but also the fine structure of the parent phase. For instance, when the density of crystal defects is high, the dislocation rather than twinned martensite will be introduced in spite of the very high strength of the parent phase.

Analysis of kinetics of diffusion induced recrystallization in Fe phase of Cu/Fe/Cu diffusion couples

Y. KAWANAMI *et al.*

Combining the columnar-geometry and boundary-diffusion model by Li and Hillert with the energy balance model by Kajihara and Gust, a new kinetic equation has been

obtained to describe the growth rate of the alloyed region due to diffusion induced recrystallization (DIR) in a solution phase of binary systems. The equation has been utilized to analyze quantitatively the kinetics of DIR in the Fe(Cu) system observed by the present authors using Cu/(Fe-4.8mass%)/Cu diffusion couples. The notation Fe(Cu) means that Cu atoms diffuse into a pure Fe or binary Fe-Cu

phase. The observations indicate that the thickness l of the DIR region formed in the Fe phase from each interface increases with increasing annealing time t according to the relationship $l=1.58 \times 10^{-6} (t/t_0)^{0.29}$ at 1323 K. Here, l and t are measured in m and s, respectively, and t_0 is unit time, 1s. Using the mobility M as the fitting parameter of the kinetic equation, we have obtained the value of $M = 8.64$

$\times 10^{-13} \text{ m}^4/\text{Js}$ and the relationship $l=1.08 \times 10^{-6} (t/t_0)^{0.33}$ on the basis of the observations. Through the analysis, it is concluded that l increases in proportion to \sqrt{t} for DIR in a pure metal whereas l increases proportionately with $3\sqrt{t}$ for DIR in a solution phase even with a low solute concentration.