

## Ironmaking and Reduction

### Degradation characteristics of iron ore fines of a wide size distribution in fluidized-bed reduction

U.-C. CHUNG *et al.*

Three mechanisms of the degradation behavior of iron ore fines (IOF) sized 1 to 5 mm during fluidized-bed reduction have been investigated: mechanical degradation (MD), thermal degradation (TD) and reactional degradation (RD). The effects of operational parameters such as ore type, temperature, superficial gas velocity, and reducing gas composition have also been studied. The degradation in three mechanisms has been quantitatively evaluated by the amount of IOF smaller than 1 mm which was produced and by the weight change of IOF in the bed during fluidization. The reactional degradation becomes the most predominant aspect among the three and the thermal degradation is the least when the IOF has a low water content, while the degradations of all three aspects are not much different in contribution when the IOF has a high water content. For the effect of operating parameters, the IOF of a lower sphericity showed a higher degradation in the cold fluidization under inert atmosphere but this effect was lessened or negligible under a fluidized-bed reduction at a high temperature. A higher water content in the IOF demonstrated a higher degradation at a high temperature. In the fluidized-bed reduction, the degradation was little affected by the reaction temperature, the superficial gas velocity, and the reducing gas composition. In addition, all three aspects of degradation were mostly completed in the early stage, within 15 min, of the operations.

## Steelmaking and Refining

### Some perspectives on technological exchange and international co-operation in steel related research

A. MCLEAN

In this paper the concept of bridge building is used in order to illustrate the theme "Technological exchange and international co-operation in steel related research". In this context, Mr. Masao Yukawa provides an outstanding example of one who was a great builder of bridges. Bridges of friendship can facilitate communication, bridges of education can enhance knowledge transfer, bridges of innovation are important for the next millennium, and bridges of synergism can span different cultures. The steel used for the hull of the Titanic is taken as an example of how steel chemistry affects steel structure, properties and performance. Some examples are presented to illustrate new technologies for the processing of steels for the next century. In the final analysis, high quality products require high quality processing and both require high quality people. In the coming decades, well-trained people will continue to constitute the critical link in the bridge building process.

### Activity of manganese oxide in CaO-MnO-SiO<sub>2</sub>-PO<sub>2.5</sub>(-MgO, Fe<sub>2</sub>O<sub>3</sub>) slags

A. SOBANDI *et al.*

The thermodynamics of manganese in high MnO slags at hot metal pretreatment temperature were investigated. The CaO-MnO-SiO<sub>2</sub>-PO<sub>2.5</sub>(-MgO, Fe<sub>2</sub>O<sub>3</sub>) slags were equilibrated with liquid copper alloys in a molybdenum crucible under a flowing CO<sub>2</sub>/H<sub>2</sub> atmosphere in the temperature range of 1573 to 1673 K.

The manganese equilibrium quotient,  $K'_{Mn} = (\%Mn)/[\%Mn]p_{O_2}^{1/2}$ , between slag and liquid copper and the activity coefficient of MnO,  $\gamma_{MnO}$ , were obtained as a function of slag composition and temperature. Based on the thermodynamic data obtained in the present study, the manganese distributions between hot metal and slag were estimated. From comparison with the plant data, it was concluded that the thermodynamic data obtained in the present study are satisfactorily applicable to the thermodynamic assessment of hot metal pretreatment with high MnO slag.

## Casting and Solidification

### Experimental studies of interfacial heat transfer and initial solidification pertinent to strip casting

L. STREZOV *et al.*

Melt/substrate contacting experiments have been carried out under controlled laboratory conditions designed to approximate the conditions encountered during the initial solidification period in strip casting. Immersion experiments were performed with various substrates embedded in a moving paddle; the substrates had both smooth and textured surfaces. The melt was 304 austenitic stainless steel, and the substrates were made from copper blocks. The apparatus and instrumentation were designed for 'millisecond' resolution of heat transfer behaviour. The experimental variables chosen for study were substrate texture, gas atmosphere, immersion velocity and melt superheat. The conclusions drawn were based on an analysis of transient interface heat fluxes during the first 50 milliseconds of contact, the observed nucleation behaviour and the resultant surface and internal solidification structures. The aim of the work was to add to the fundamental understanding of melt/substrate contacting in the meniscus region, since this will inevitably be critical to strip surface quality.

### The solubility of Ar in liquid succinonitrile

Y.H. WANG *et al.*

Since a gas bubble formation process ahead of a growing dendritic solid/liquid interface is quite complex, the direct observation of gas bubble formation by using the transparent materials is very useful method to study. For works on the gas bubble formation during the solidification, the information of solubility of gas in the melt was required. The first attempt to measure the solubility of Ar in molten succinonitrile was carried out by using a newly developed method which consists of the injection of He into molten salts through a nozzle,

collection of ascending bubbles of He-Ar gas mixture, determination of concentration of Ar in the He-Ar gas mixture using a high sensitive mass spectrometer. The solubilities of Ar in molten succinonitrile have been determined as a function of temperature from 333 to 348 K and were best expressed by

$$\ln[\text{Ar}] = -7.64 - 1630/T$$

where the unit of solubility is expressed by mol-Ar/cm<sup>3</sup>-succinonitrile. From the temperature dependency of Ar solubility, the enthalpy of solution was found to be about 13.5 kJ/mol.

## Instrumentation and Control System

### A method for measuring particle size in overlapped particle images

X. SONG *et al.*

This paper describes a new method to measure the sizes of particles and their probability distribution function by image processing. In the present research, an image with heavily overlapped particles was processed. The edges of the particles in the image are detected by Canny's method, and the contours are linked from the pixels in the edges by using chain-coding. The contours are subsequently segmented according to the curvature. Constant curvature segments are clustered according to some relations among contour segments which are likely to represent the same circle. The Least Squares Method which provides an accurate parameter estimation is performed to find out the parameters of a circle. The present method can recover most of particles even in a heavily overlapped particle image. It provides a useful way of investigating the structure of material or counting particles in many fields of engineering. Another advantage of the present method is its short computational time. All the computations in this paper were carried out on a personal computer, and the computational time of processing one image was between 4-10 sec on the personal computer (Pentium II, 300 MHz), which depended on the number of particles in the image. It is clear that the present research can be extended to the real-time processing of particle-detecting and particle-measuring in the future.

### Renewal of automatic gage control system for a cold tandem mill with electric screwdowns

Y. WASHIKITA *et al.*

This paper describes renewal of automatic gage control (AGC) system for a cold tandem mill with electric screwdowns. Although gage control methods using high responsive actuators were researched, there is few research on the renewal of an old mill with low response actuators. We studied the way of improving gage accuracy without reconstructing actuators, from the viewpoint of saving cost.

Gage response to manipulation of the actuators depends on the frequency characteristics of the actuators and the rolling process, and the effect of AGC was restricted by these frequency characteristics. We eliminated the frequency dependency of the gage response by inserting a dynamic compensator with re-

verse frequency characteristics in the control loop of AGC, and expanded the controllable frequency range of the feedforward AGC to a higher frequency. Moreover, in order to reduce the tension change caused by the gage control and to stabilize rolling force during acceleration and deceleration, we developed new tension control and rolling force control manipulating reduction in thickness. The combination of these controls and the feedforward AGC reduced gage deviation, keeping stable rolling.

The application of this AGC system to a cold tandem mill with electric screwdowns resulted in a great improvement in gage accuracy, and the accuracy has almost matched that of a newly constructed mill.

## Surface Science and Technology

### Mathematical modelling of Al take-up during the interfacial inhibiting layer formation in continuous galvanizing

P.TOUSSAINT *et al.*

A laboratory scale continuous galvanizing simulator has been designed and used in order to study the effects of control parameters on interfacial Al take-up, with particular emphasis on hydrodynamic effects and very short immersion times, shorter than one second. Effects of Al concentration and process temperatures have also been studied. It was shown that Al take-up is extremely fast at the very early stages of the reaction, leading to the complete coverage of the steel within a tenth of a second, and that the reaction subsequently proceeded at a rate compatible with solid state diffusion through the inhibiting layer. A mathematical model is proposed to describe the take-up curves. Atomic force microscopy was used to analyze the morphology of the inhibition layer and energy dispersive X-ray spectroscopy for thickness measurements.

## Microstructure

### Overview—Five decades of the Zener equation

P.A.MANO HAR *et al.*

The Zener equation was first reported by C. S. Smith in 1948 and since then it has become an integral part of any theory which deals with recovery, recrystallization and grain growth in particle-containing materials. Several modifications to the original equation have been made over the past five decades to improve its applicability to more realistic situations. This paper summarises these modifications and discusses which modifications are reasonable and justifiable based on the analytical models and experimental evidence reported in the literature. Several examples of the applications of the equation are provided to describe annealing phenomena in a wide variety of materials. The paper also examines the impact of the equation in the field of materials science and engineering and suggests a direction for its future development.

### Solubility product of TiN in austenite

K.INOUE *et al.*

The solubility product of TiN in an austenitic steel was experimentally determined using a diffusion couple technique. Diffusion couples consisting of Ti-N steels with various compositions were annealed at temperatures between 1473 and 1623 K, taking special care to exclude the oxygen contamination during the annealing. The solubility limit of TiN was determined from the break points of iso-activity lines for nitrogen. A thermodynamic analysis was also carried out to arrive at solubility products by considering the wide range of temperature dependence of the TiN formation energy. Solubility products in austenite ( $\gamma$ ), ferrite ( $\alpha$  or  $\delta$ ) and liquid phases were determined as  $\log(\text{mass}\% \text{Ti})(\text{mass}\% \text{N})_{\gamma} = 4.35 - 14890/T$ ,  $\log(\text{mass}\% \text{Ti})(\text{mass}\% \text{N})_{\alpha/\delta} = 4.65 - 16310/T$  and  $\log(\text{mass}\% \text{Ti})(\text{mass}\% \text{N})_{\text{liq}} = 4.46 - 13500/T$  respectively.

### Transformations during intercritical annealing of a TRIP-assisted steel

I.SAMAJDAR *et al.*

During intercritical annealing of cold rolled Trip-assisted steel, ferrite matrix recrystallizes and austenite forms. After subsequent quenching, part of that austenite may remain at room temperature as retained austenite, while the other part transforms into martensite. Based on their respective origin, recrystallized ferrite grains were recognized as PSN (particle stimulated nucleation) and non-PSN types. Near randomized PSN grains nucleated somewhat preferentially (*i.e.* at the earlier stages of recrystallization), which may explain the slight drop in non-PSN orientations at higher annealing temperatures. Increased intercritical soaking periods coarsened the austenite grains and increased the austenite volume fraction (till 37%), while the austenite stability dropped (*i.e.*  $M_s$  temperature increased). On the other hand, both the retained austenite volume fraction and its carbon content decreased with prolonged intercritical soaking. Stability of the initial retained austenite colonies was ascertained by their small sizes and high carbon content, while smaller dimensions of the austenitic domains were possibly crucial in their stability/survival after prolonged soaking. At the initial stages of transformation, the retained austenite showed randomized orientations. During the subsequent stages, a weak cube texture was observed. After the transformation was complete and a drop in retained austenite percentage was recorded, again a randomized texture for retained austenite was noted.

## Physical and Mechanical Properties

### Application of band parameters to materials design

Y. MAKINO

Availability of the two band parameters to materials design is reviewed. In order to express the chemical character between two different atoms in solid state, the concept of two band parameters for any bond between two different atoms is proposed on

the basis of the bond orbital model and the empirical relation between observed band gap and pseudopotential radii of Zunger. The two band parameters are called as the hybrid function ( $H$ ) and gap reduction parameter ( $S$ ), which substantially correspond to the energy difference between the bonding and anti-bonding levels and the band widening effect due to the formation of crystalline state. These parameters roughly correspond to ionic and covalent band gaps which are constructed by one gap model. By comparing with the definition of ionicity based on the ionic and covalent band gaps, it is found that the ionicity for any A-B bond can be estimated from the two band parameters. Extensive availability of the band parameters to materials design is suggested by exemplifying several successful results such as the construction of crystal structure maps for compounds, determination of site preference of third alloying elements in intermetallics and prediction of phase changes in pseudobinary nitride films in the energetic processes.

### Notch and fracture toughness studies on stainless steels containing vanadium

R.PATON

One of the shortcomings of ferritic stainless steels is their limited toughness. By modifying the steel chemistry to include vanadium, some improvement is obtained in this regard. In this work, the influence of different stabilizers and thermomechanical treatments on the toughness of an experimental 18Cr-4V alloy have been studied. Some tests were also carried out to assess the influence of welding on the toughness and intergranular corrosion resistance.

It was found that vanadium, alone, is not a potent stabilizer. A small addition of niobium is necessary to prevent intergranular corrosion. High annealing temperatures adversely affect toughness, and the benefit of smaller grain size has not been demonstrated in this work.

A combination of inert-gas shielded welding, relatively low heat input and an austenitic filler provides weldments with adequate toughness.

### Effects of tensile stress on the high-temperature oxidation of an Fe-38Ni-13Co-4.7Nb-1.5Ti-0.4Si superalloy in air

K.KUSABIRAKI *et al.*

The oxidation behavior of an Fe-38Ni-13Co-4.7Nb-1.5Ti-0.4Si superalloy (Incoloy 909 type alloy) under tensile stress conditions has been investigated in the temperature range of 1100 to 1300 K in air. Optical microscopy, X-ray diffraction measurements, and electron probe microanalyses on the oxide scales formed during oxidation indicate that the scales consist of an external scale and an internal scale which has an intergranular scale (above 540 ks at 1100 K and above 1200 K) and an intragranular scale. The oxide phases in each scale are identified as ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and CoO·Fe<sub>2</sub>O<sub>3</sub>) and (FeO·Nb<sub>2</sub>O<sub>5</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), respectively. An effect of tensile stress on the compositions of the scales is not observed. Although the effects of the tensile stress on growths of the external scale and the intra-

granular scale are small, the growths of the thickness and the width of the intergranular scale are remarkably promoted as the tensile stress increases. The stress accelerated grain boundary oxidation in this alloy is confirmed distinctly.

#### **Effect of vanadium and niobium on restoration behavior after hot deformation in medium carbon spring steels**

*M. AYADA et al.*

The effect of vanadium and niobium on the restoration behavior after hot deformation in medium carbon spring steels was investigated by use of a thermomechanical processing simulator. It was clarified that vanadium is effective to inhibit restoration after hot deformation, because vanadium carbides and/or nitrides finely precipitate during and after hot deformation. On the other hand, niobium is not effective to inhibit restoration. This is because the amount of niobium soluble in austenite is essentially

very small in medium carbon steels and that niobium exists as coarse carbides before hot-deformation.

The degree of strengthening in martensite was not so large within the present deformation condition equivalent to Controlled Tapered Leaf Rolling, because the enhanced dynamic recovery and recrystallization caused the steady-state deformation (small work-hardening). Further, the increase in the amount of retained austenite and the decrease in solute carbon resisted to increase the strength. However the martensite structure became fine owing to the refinement of the austenite grains by hot deformation. As a result, the toughness and the fatigue strength increased by hot deformation and direct quenching. The above results indicate that the combination of the present thermomechanical processing and the Controlled Tapered Leaf Rolling would be effective to decrease the weight of the springs. The present investigations have also clarified that the vanadium addition and the low temperature de-

formation is preferable to maximize the effect of the thermomechanical processing.

#### **Social and Environmental Engineering**

##### **The management of research and technology in the German steel industry with respect to the European Union**

*D. SPRINGORUM*

Discussion on frame and targets as well as management structures for research and development of the German steel industry. Product and research strategies. Specific steel research expenditure in Germany. Example for joint research work. The role of the European Coal and Steel Community (ECSC) and of the European framework programmes including remarks about the future of European R & D support.