（＊掲載記事題目及び掲載頁は変更になる場合があります。）

Fundamentals of High Temperature Processes

Crack generation in blast furnace slag bearing high titanium by microwave cyclic heating

Y.CHEN et al.

In order to facilitate grinding a blast furnace (BF) slag bearing high TiO_2 (Ti-BF slag) for extracting $CaTiO_3$ (TiO_2 containing phase) from it, microwave (MW) cyclic heating was attempted to induce cracks. In this study, MW heating behavior of Ti-BF slag, general BF slag and sintered perovskite ($CaTiO_3$) body were examined. Under the same heating conditions, Ti-BF slag was heated better than the general BF slag. And the sintered perovskite powder compact was heated best among them. Considering the measured large permittivity of Ti-BF slag, the perovskite phases in the Ti-BF slag are responsible for heating of the whole slag.

Cracks were induced in the Ti-BF slag by cyclic irradiation of MW. They extended and opened as an increase of the cycle number. In order to examine the effect of the induced cracks on the strength of Ti-BF slags, compression test of the specimens was performed after the treatment. It was demonstrated that MW treatment caused reduction in the compressive strength, such that the strength decreased about 35% after 10 cycles of heating for 5 min.

It was also shown that while the treatment with longer heating time caused a smaller decrease in the compressive strength, increasing of cyclic number decreased the strength more effectively.

(cf. *ISIJ Int.*, **47** (2007), 193)

Effects of N, C and Si contents and MgO on dispersion of TiN particles in Fe–1.5%Mn–0.05(0.15)%C alloy

H.OHTA et al.

The crystallization and precipitation of TiN in an Fe–1.5%Mn–0.05(0.15)%C–0.1%Ti alloy with and without Mg deoxidation have been studied as a function of N (20–130 ppm) and Si (0, 0.5 and 1.1%) contents. The size distribution of TiN particles in the sample of 0.05% C with no Mg deoxidation is bimodal and that in the sample of [%C]=0.15 is a log-normal distribution. This is explained by the difference in the degree of microsegregation which is influenced by the solidification mode. The significant effect of Si on crystallization and precipitation of TiN has not been observed. The number of MgO, TiN+MgO and TiN particles with Mg deoxidation is about two times larger than that of TiN particles with no Mg deoxidation. The number of particles with Mg deoxidation sample at [%C]=0.05 increases gradually with increasing the fraction of solid, but that at [%C]=0.15 increases drastically at interdendritic region.

(cf. *ISIJ Int.*, **47** (2007), 197)

Mass spectrometric study on phosphorus in molten carbon-saturated iron

T.NAGAI et al.

The vapor pressure of phosphorus in molten carbon-saturated iron with phosphorus content of 0.20–0.92 mass% at 1 473–1 673 K was measured by double Knudsen cell mass spectrometry. In the molten carbon-saturated iron phosphorus behaved according to Henry's law in the composition range. The activity coefficient of phosphorus in this conditions relative to infinite dilute solution, f_P, and the interaction parameter of carbon on phosphorus in molten iron, $e_P^{'C}$, were determined to be as follows:

$$\log f_P = -\frac{300}{T} + 0.82(\pm 0.05),$$

$$e_P^{'C} = \frac{120}{T} + 0.061(\pm 0.010)$$

(cf. *ISIJ Int.*, **47** (2007), 207)

Surface tension evaluation of molten silicates containing surface-active components (B_2O_3, CaF_2 or Na_2O)

M.NAKAMOTO et al.

A thermodynamic model for determining the surface tension of molten ionic mixtures, derived by considering the ionic radii, was applied to a molten silicate containing a surface-active component B_2O_3, CaF_2 or Na_2O. The calculated results of the present model were in agreement with the experimental data in these ternary systems over a wide temperature range.

(cf. *ISIJ Int.*, **47** (2007), 211)

Ironmaking

Reduction of iron ore fines with CO-rich gases under pressurized fluidized bed conditions

C.PAWLIK et al.

Pressurized fluidized bed processes for the reduction of iron ore fines using CO-rich reducing gas formed by coal gasification are an upcoming iron-making technology. They operate in a continuous multi-staged countercurrent mode at an absolute pressure of 4 bar.

For process optimization, a laboratory-scale pressurized fluidized bed reactor was built to perform experiments similar to industrial conditions. The reactor is operated as a differential reactor to ensure a constant gas concentration and temperature within the reactor volume.

Three-stage experiments with three different iron ores were carried out at an absolute pressure of 4 bar in batch-wise operation. The reducing gas consisted of a mixture of H_2, H_2O, CH_4, CO_2 and CO. The CO-concentration was varied in a range between 30% and 50%, the residence time in pre-reduction between 30 and 60 min and the operating temperature from 400°C to 850°C. In correspondence to the tests, microscopic techniques were applied to determine the importance of morphological properties of the ores on their reduction behavior.

A higher temperature in the pre-reduction stage causes an increase of porosity as well as an increase of the final reduction degree. Comparing different ore types, a significant correlation between the morphology of the employed ores and the final reduction degree was found.

(cf. *ISIJ Int.*, **47** (2007), 217)

In situ observation of reduction behavior of hematite with solid carbon and crystallographic orientation between hematite and magnetite

Y.KASHIWAYA et al.

Recently, the composite pellets between iron ore and carbon are studying by many researchers because of its high reactivity. However, the reaction mechanism of the composite pellet is not yet clarified. It is obviously determined the reaction mechanism at initial stage as the direct reaction between solid carbon and iron oxide. However, in the course of reaction, the reaction mechanism will change to the indirect reduction of iron oxide with CO gas owing to separation of the interface between solid carbon and iron oxide.

In this study, samples of poly crystalline hematite (grain size was approximately 20 μm) were prepared. The hematite sample was coated by carbon with different thickness. The *in situ* observation by a laser microscope was carried out. The crystal orientation and morphology was investigated before and after the reaction using EBSP, SEM, AFM, XRD and Raman.

The direct reduction began from about 650°C and the reaction proceeded rapidly until carbon was consumed. The behavior of reduction was different from the thickness of carbon layer (amount of carbon) which was related to whether the product gas (CO) could be passed through the carbon layer or not. After Reduction, the lath magnetite having same direction in a grain was observed on the surface. The crystallographic orientation between magnetite and hematite is as follows;

$$(0\,0\,0\,1)_H \parallel (1\,1\,1)_M,\ [1\,\bar{1}\,0\,0]_H \parallel [1\,\bar{1}\,0]_M$$

In addition, the growth direction of the lath magnetite formed on the hematite surface was

$$[2\,1\,\bar{3}\,0]_H \parallel [1\,0\,\bar{1}]_M$$

(cf. *ISIJ Int.*, **47** (2007), 226)

Sintering pot test of manganese ore with addition of manganese furnace dust

R.SHEN et al.

Manganese furnace dust is formed from volatiles and fines collected during wet scrubbing of the off-gas from manganese alloy smelting furnaces. This dust, in the form of slurry, contains tar, alkalies, zinc, manganese oxide and other elements. Impediments to the recycling of the manganese furnace dust back to the ferroalloy furnaces are the potential accumulation of zinc and alkalies, which can cause irregularities in furnace operation, and handling. This paper examines the behaviour of zinc during sintering pot tests of manganese ore with addition of manganese furnace dust. Laboratory reduction experiments showed that at temperatures above 800°C zinc oxide was reduced by carbon in tar to zinc vapour. However, zinc removal in sintering pot tests was below 30%, which was attributed to reoxidation of the zinc vapour. Zinc removal was the highest from the bottom section of the sintering pot bed.

(cf. *ISIJ Int.*, **47** (2007), 234)

Influence of operational conditions on dust emission from sintering bed

M.NAKANO et al.

Dust emission is an environmental concern to iron ore sintering. To suppress it by preventing dust from migrating into gas stream in the sintering bed, the effect of various operational factors was investigated and the site and cause of dust generation in sintering bed were estimated through pot tests with a dust sampler placed between the pot and the wind box. Dust emission increased with increasing coke content or decreasing moisture; suction pressure, bed height and blend ratio of Marra Mamba ore did not affect significantly. Of 750 mg/kg dust emitted from bed, about 150 mg/kg originated in the drying zone, another 150 mg/kg in the dehydration zone and the rest in the coke combustion zone. The dominant causes allowing particles to move into gas stream in the bed was estimated to be granule's breakage in the drying/dehydration zones and extinction of adjacent particles in the combustion zone.

(cf. *ISIJ Int.*, **47** (2007), 240)

The characteristics of catalyst-coated highly reactive coke

S.NOMURA et al.

It is important to develop the production and utilization technology of highly reactive coke in order to improve the efficiency of blast furnace reactions. In this study, the characteristics of catalyst-coated highly reactive coke produced by the 'post-addition of catalyst to coke' method were investigated. First, the catalytic effect of Fe and Ca on graphite and deashed coke during the C–CO_2 reaction was investigated. Fe and Ca addition increased the reaction rate and decreased the reaction beginning temperature. Second, the reaction rate of Fe or Ca coated coke was kept high until the weight loss reached 10%. This means that a high catalytic effect is maintained in the thermal reserve zone of the blast furnace. Furthermore, it was found that 70% of the catalyst coated on the coke surface remains after drop impacts. It is expected that the loss of catalyst during coke handling and transportation is small. Catalyst-coated highly reactive coke is promising for improving the efficiency of blast furnace reactions.

(cf. *ISIJ Int.*, **47** (2007), 245)

Casting and Solidification

Segregation in twin roll strip cast steels and the effect on mechanical properties

Z.LIU et al.

Strips of low carbon steels containing high contents of P and Cu have been produced by twin roll strip casting. The as-cast strips containing more than 0.1 mass% P exhibited obvious inverse surface segregation, which was difficult to be eliminated by the subsequent annealing treatments. After cold rolling and annealing treatment at 625°C for 30 min, the strip containing 0.1 mass% P exhibited similar plastic deformation and rupture behaviors with the strip containing 0.01% P, showing no obvious deteriorating effects of P on the ductility of as-cast strip. With increasing P content in steel, more cleavage cracks, micro voids and inter-granular rapture were observed in the fractured areas after tensile testing, which are believed to have been induced by the segregation of P in as cast strips. These results provide primary guidance for recycling dirty steel scraps.

(cf. *ISIJ Int.*, **47** (2007), 254)

Eutectic cell and nodule count in cast iron Part I. Theoretical background

E.FRAS et al.

In this work, heat balance is incorporated in developing expressions of general validity for the solidification of hypo-eutectic flake graphite cast iron, as well as eutectic and slightly hyper-eutectic nodular iron. These expressions incorporate the active nucleation and growth processes that occur in flake and nodular graphite. The derived analytical equations describe the eutectic cell N and nodule count N_n as a function of the solidification parameters and melt chemistry. From this analysis, eutectic cell or nodule counts can be predicted based on experimental data related to cooling rates and chemical composition. In particular, it has been found that the quality of the liquid cast iron is closely tied to its intrinsic nucleation properties (N_s and b coefficients in the nucleation equation, $N_{nuc}=N_s \exp(-b/\Delta T_m)$), or directly to the graphite nuclei density, N_{nuc}. Moreover, the outcome of the present work is used in providing a rational for the effect of technological factors such as the material mold ability to absorb heat, the casting modulus, pouring temperature, chemistry, inoculation practice, holding temperatures and times on the resultant cell count or nodule count in cast iron.

(cf. *ISIJ Int.*, **47** (2007), 259)

Eutectic cell and nodule count in cast iron Part II. Experimental verification

E.FRAS et al.

In this work the predictions of a proposed theoretical analysis aimed at elucidating eutectic cell counts, N in gray iron cast and nodule counts, N_n in ductile iron were experimentally verified. The work was focused on processing flake graphite and ductile cast iron under various inoculation conditions in order to achieve various physicochemical states in the experimental melts. In addition, plates of various wall thicknesses were cast and the resultant eutectic cell N and nodule N_n count were established. Moreover, thermal analysis was employed for determinations of the maximum degree of graphite eutectic undercooling, ΔT_m. A comparison was made between the theoretical predictions and the experimental determinations of eutectic cells, N, nodule counts, N_n and ΔT_m. It was found that N and N_n can be related to the casting modulus, M and as a consequence to the wall thickness, s in plate shaped castings. In particular, the outcome of this work indicates that the predictions of the theoretical analysis are in good agreement with the experimental outcome.

(cf. *ISIJ Int.*, **47** (2007), 269)

Three-dimensional phase-field simulation of free dendrite growth of iron

K.OGUCHI et al.

The free dendrite growth of iron has been investigated using a three-dimensional phase-field model with thin-interface-limit parameters. The stability parameters are determined for different values of anisotropy intensity of interface energy, and the obtained values are in good agreement with previously reported values. The dendrite growth velocities of iron at different degrees of undercooling are compared with experimental data, and a good agreement between phase-field simulations and experiments are obtained when the kinetic coefficient is assumed to be 20. The validity of the analytical dendrite model by Lipton, Kurz and Trivedi (LKT model) is also examined, and it is shown that the model gives accurate predictions when suitable parameter values are adopted.

(cf. *ISIJ Int.*, **47** (2007), 277)

Chemical and Physical Analysis

Role of ash impurities in the depletion of carbon from alumina–graphite mixtures in to liquid iron

R.KHANNA et al.

Due to their excellent thermal shock and slag resistance at high temperatures, alumina–graphite refractories are used extensively in the steel industry. The degradation of carbon based refractories through carbon depletion is an important issue and a fundamental understanding of refractory behaviour at high temperatures is crucially important. Natural flake graphite, with ash impurity levels ranging from 1 to %, is used extensively in the commercial preparation of alumina–carbon refractories. This study investigates the role played by ash impurities in the depletion of carbon from the refractory composite. Two natural graphites, respectively containing 2.1% and 5.26% ash, were used in this study. Substrates were prepared from mixtures of alumina and carbon over a wide concentration-range. Using a sessile drop arrangement, carbon pick-up by liquid iron from alumina–natural graphite mixtures was measured at 1 550°C and was compared with the carbon pick-up from alumina–synthetic graphite mixtures. These studies were supplemented with microscopic investigations on the interfacial region. Very high and similar levels of carbon dissolution were however observed from both alumina–natural graphite mixtures, with carbon pickup by liquid iron from mixtures with up to 30 wt% alumina reaching saturation. A sharp reduction to near zero levels was observed in the 30 to 40 wt% alumina range. Along with implications for commercial refractory applications, these results are discussed in terms of poor wettability between alumina and liquid iron, interactions between ash impurities and alumina, and formation of complexes in the interfacial region.

(cf. *ISIJ Int.*, **47** (2007), 282)

Forming Processing and Thermomechanical Treatment

Thermal stability of high temperature deformation induced ferrite in a low carbon steel

Z.LIU et al.

A series of unidirectional compression tests for a low carbon steel were performed on a thermal simulating machine Gleeble 3 500, and the thermal stability of high temperature deformation induced ferrite (DIF) was studied systematically through the post-treatments such as isothermally hot holding time above the austenite–ferrite equilibrium transformation temperature Ae_3, cooling rates and annealing temperatures after deformation. The results show that the DIF volume fraction decreases during isothermally hot holding and finally DIF disappears. The DIF grains showed rapid growth at low annealing temperatures (<200°C) and relatively stable at middle annealing temperatures (200–500°C), lastly rapid growing at high annealing temperatures (500–700°C). Compared with the microstructure of DIF with martensite in quenched specimens, the proeutectoid ferrite and pearlite appear in controlled cooled specimens at low cooling rates (*i.e.* 10°C/s and 1°C/s). The present experiments revealed that DIF is not stable at the high temperature and hardly conserved in hot mills.

(cf. *ISIJ Int.*, **47** (2007), 289)

Reverse austenite transformation behavior of equal channel angular pressed low carbon ferrite/pearlite steel

K.-T.PARK et al.

Reverse austenite transformation characteristics of ultrafine grained ferrite/pearlite low carbon steel were examined by using a dilatometry with a heating rate of 10°C/s, and compared with those of coarse grained counterpart. Ultrafine grained steel was prepared by equal channel angular pressing at 500°C, resulting in a microstructure consisting of ultrafine ferrite grains and pearlite with partially dissolved pearlitic cementite. Reverse austenite transformation start and finish temperatures of ultrafine grained steel were lower than those of coarse grained steel. Reverse austenite transformation of coarse grained steel occurred with the two serial stages of pearlite→austenite followed by proeutectoid ferrite→austenite. In the case of ultrafine grained steel, reverse austenite transformation was manifested by the three serial stages of carbon-supersaturated ferrite→austenite, pearlite→austenite, and proeutectoid ferrite→austenite in the order. The formation of carbon-supersaturated ferrite in ultrafine grained steel is associated with carbon dissolution from pearlitic cementite during equal channel angular pressing. The effect of equal channel angular pressing on reverse austenite transformation temperatures can be explained in terms of the difference in shear stress driving transformation between two steels under assumption that the nature of carbon-supersaturated ferrite in ultrafine grained steel is similar to that of conventional martensite.

(cf. *ISIJ Int.*, **47** (2007), 294)

Welding and Joining

Transformation in stir zone of friction stir welded carbon steels with different carbon contents

L.CUI et al.

Five types of ferrite–pearlite structure carbon steels with different carbon contents (IF steel, S12C, S20C, S35C, S50C) were friction stir welded under various welding conditions, and the mechanical properties and microstructures of the FSW carbon steel joints were evaluated. Compared with IF steel, the microstructures and mechanical properties of the carbon steel joints are significantly affected by the welding conditions. When the carbon content is less than or equal to 0.12 mass%, the welding produces ferrite–pearlite structures, and the strength slightly increases compared to the base metal due to the refined microstructure; when the carbon content is above 0.2 mass%, the welding produces ferrite–pearlite plus harder phases like the martensite and bainite microstructures, resulting in a significantly increased strength of the joints. These are dependent on each of the thermal-mechanical cycles.

(cf. *ISIJ Int.*, **47** (2007), 299)

Transformations and Microstructures

The influence of niobium microalloying on austenite grain coarsening behavior of Ti-modified SAE 8620 steel

K.A.ALOGAB et al.

The potential for suppressing unacceptable austenite grain growth during carburizing by Nb microalloying additions in the range of 0.02 to 0.11 wt% to a Ti-modified SAE 8620 carburizing steel were evaluated. Alloys, were designed based on fundamental equilibrium thermodynamic analyses, as part of an extensive study on the effects of alloy composition, thermomechanical history, and pseudo-carburizing conditions on austenite grain coarsening behavior. Laboratory samples were produced to simulate both conventional hot rolling and controlled rolling practices designed to produce different initial precipitate distributions. Pseudo-carburizing heat treatments, *i.e.* without a carburizing atmosphere, were performed in the temperature range of 950 to 1 100°C for holding times of 30 to 360 min. Precipitate distributions, including size, number density, morphology, distribution, and chemical composition in selected samples from the as-rolled and pseudo-carburized conditions were evaluated with transmission electron microscopy on extraction replicas. Results showed that increasing Nb additions to the Ti-modified SAE 8620 steel restrained austenite grain coarsening, and increased the grain coarsening time, especially at temperatures below 1 050°C. The Nb-free (Ti-modified) steel yielded either severely duplex grain structures or pseudo-normal grain growth (with very large mean grain diameter). However, holding a Ti–Nb-modified steel (*e.g.* 0.06 Nb wt%) at 950°C for 6 h or at 1 000°C for 4 h. produced fine and uniform austenite grain structures (with a mean grain diameter less than 20 μm). The finer grain sizes observed in the Ti–Nb-modified steels were due to the presence of Nb-rich precipitates that hinder austenite grain coarsening, and precipitate distributions and grain growth behaviors are also influenced by the steel rolling history. The results indicate that Nb can successfully be used to suppress grain growth in carburizing steels.

(cf. *ISIJ Int.*, **47** (2007), 307)

Tempering process and precipitation behavior of 8%Cr–2%WTa steel

M.TAMURA et al.

In order to clarify the stability of precipitates in 8%Cr–2%W–0.09%Ta steel at high temperatures, tempering and precipitation behavior of the steel has been examined precisely. Tempering was performed mainly at 740°C for 0.5–1 000 h. Hardness peak was found in a tempering curve at 650–740°C for 3.75–8 h. Discontinuous changes in X-ray diffraction intensity of the matrix, apparent lattice strain and apparent crystalline particle size, which were estimated from integral breadth, the amount of extracted residue, and the Cr and Ta content in the residue were observed at 740°C for 2–12 h tempering. These discontinuous changes correspond to the rotation of preferred orientation or the generation of subgrains accompanying the dissolution and the subsequent re-precipitation of $M_{23}C_6$ and TaC. The re-precipitation of fine $M_{23}C_6$ particles is responsible for the hardness peak. Three kinds of TaC with different chemical composition and different size are found. The amount and the size of the coarse and medium TaC particles are affected by the discontinuous changes in microstructure. Fine particles of TaC grow very slowly, which is caused by the continuous dissolution of TaC due to the annihilation of precipitation site, dislocations, and re-precipitation on dislocations. Particle sizes of TaC and $M_{23}C_6$ estimated using X-ray diffraction peak coincide with those observed in transmission electron microscopy.

(cf. *ISIJ Int.*, **47** (2007), 317)

Mechanical Properties

Dependence of magnetic susceptibility on dislocation density in tensile deformed iron and Mn-steel

K.YAEGASHI

Dependence of the coefficient c of magnetic susceptibility on dislocation density was investigated in tensile deformed polycrystalline Fe and Mn-steel (JIS SFVQ-1A). The dislocation density was measured by transmission electron microscopy (TEM). The coefficient c was analyzed from hysteresis loops, and found to be proportional to dislocation density. It is concluded that the observed dependence of c is valid when there exist homogeneously distributed dislocations. The observed dependence of c on dislocation density would be useful for nondestructive evaluation of dislocation structure in practical steel.

(cf. *ISIJ Int.*, **47** (2007), 327)

Creep behavior and rupture life of the simulated intercritical HAZ for 1.25Cr–0.5Mo steel under a multiaxial stress state

S.FUJIBAYASHI

For the life assessment of an elevated temperature

component under complex loading, the effect of a mutiaxial stress state upon creep behavior must be taken into account. From a practical point of view, quantifying the creep life of welds exposed to mutiaxial stresses should be the first priority since welds are often located at the change in geometry. For the components fabricated from low alloy ferritic steels, the most likely failure mode should be Type IV cracking taking place at Intercritical HAZ (ICZ).

Thus, the triaxiality effect upon the creep behavior of ICZ for 1.25Cr–0.5Mo steel was examined using semicircular notched specimens. Though grain boundary damage was accelerated when tested by notched specimens, all the notched specimens showed notch strengthening behavior. To quantify the effect of the equivalent stress and maximum principal stress, stresses given by a code of practice for notched bar creep rupture testing were correlated with rupture lives of notched specimens. It was found that the equivalent stress played a major role in determining the mutiaxial rupture life for ICZ.

The above finding suggests that the Monkman–Grant type relationship between rupture life and Θ_4 of the theta projection, which was found in the uniaxial creep tests, could be also applicable to a multaxially stressed component since creep deformation is uniquely determined by the equivalent stress. As a matter of fact, the rupture lives of notched specimens were also well correlated with Θ_4. As a new scheme for the nondestructive life assessment, measuring the value of Θ_4, which is equivalent to the slope of strain rate versus strain at the tertiary creep regime, is considered to be potential.

(cf. *ISIJ Int.*, **47** (2007), 333)

Effect of solute copper on yield strength in dislocation-strengthened steels

J.SYARIF et al.

The effect of copper atoms in solid solution (solute Cu) on yield strength was investigated in dislocation-strengthened steels such as a martensitic steel and a work-hardened steel, which have high dislocation density. The yield strength of the martensitic steel increases with increasing content of the solute Cu. However, the increment of the yield strength by the solute Cu is smaller in the martensitic steel than in the ferritic steel. Dislocation density of the martensitic steel increases with increasing Cu content and the yield strength is also enhanced depending on the dislocation density. The increment of the yield strength can be reasonably explained by the dislocation strengthening mechanism based on the Bailey–Hirsch relationship. In the work-hardened ferritic steel, the solid solution strengthening by Cu is significant when the dislocation density is low, but it tends to disappear with increasing the dislocation density through cold-rolling. These are indicative of the facts that the yield strength of the dislocation-strengthened steel is determined by the dislocation strengthening and the contribution of the solute Cu on the yield strength disappears owing to high dislocation density.

(cf. *ISIJ Int.*, **47** (2007), 340)