

Fundamentals of High Temperature Processes

Microstructural features of $TiAl_3$ base compounds formed by reaction synthesis

M.SUJATA *et al.*

Microstructural evolution in $TiAl_3$ and $TiAl_3$ base alloys formed by the reaction synthesis between liquid Al and solid Ti or Ti alloys has been studied by carrying out the tests on reaction couples isothermally reacted between 973 and 1373 K for different time intervals. It has been found that the reaction product forms by the exothermic chemical reaction and while $TiAl_3$ is the only reaction product formed up to the reaction temperature of 1273 K, $TiAl_3$ as well as Ti_9Al_{23} form when the reaction is carried out at 1373 K. In all cases the microstructure of the reaction layer comprises titanium aluminide particles dispersed in an Al rich matrix. However, morphological features of titanium aluminide particles been shown to be widely differing with the reaction conditions. In pure Ti-Al reaction couples an increase in the reaction temperature from 973 to 1173 K leads to the formation of particles having a bi-modal distribution. At the reaction temperature of 1173 K a large number of particles formed contain several micro-cracks. In contrast to $TiAl_3$ formed in the case of pure Ti-Al couples, the morphology of the $TiAl_3$ based compounds containing V or Mo, Zr and Si has been found to be significantly different. Instead of being equiaxed, the particles of $TiAl_3$ based compounds have a continuously changing morphology across the reaction layer. The particles away from the reaction interface, in general, are not equiaxed, and have sharp edges and many of them have severe internal micro-cracks.

Ironmaking and Reduction

Gas-particle flow in a De Laval nozzle with curved convergent configuration

Y.PENG *et al.*

An analytical procedure of gas-particle mixture flow in the De Laval nozzle is described for the case where the curved convergent part of nozzle has been prescribed. The effect of particle volume fraction, particle clouds, and nozzle configuration on the gas-particle flow is analytically considered. According to the Mach number checkout method, the critical cross-sectional area of the nozzle and the behavior of the supersonic gas-particle flow were determined and calculated respectively.

Evolution of porosity profiles of magnetite phase during high temperature reduction of hematite

J.JANOWSKI *et al.*

The reduction of initially nonporous hematite to porous magnetite by $CO + CO_2$

(3:97) mixture was monitored thermogravimetrically at 850°C. For the series of six kinetic runs the grains of diameter *ca* 1.5 mm were used. The final reduction degree varied from 13 to 100%. After each kinetic run the microscopic observations of the central cross-section of grains were done in quantitative way. The observations yielded the values of local porosity. The empirical equations were found describing the continuous exponential decrease of local porosity with the distance from the external surface of the grain. The value of total porosity obtained by mercury porosimetry agrees in a reasonable way with microscopic data.

The classical shrinking core model (SCM) was fitted to kinetic data. The model took into account the gas-solid reaction occurring at sharp defined interface as well as the pore diffusion phenomena occurring inside the magnetite layer. The model was also modified. The local value of porosity was introduced to the definition of effective diffusivity (D^{eff}). In this way D^{eff} was allowed to vary with the distance from the external surface of the grain. The corrected three parameter SCM yielded slightly worse results. It implies that apart from the spatial variation of D^{eff} the temporal one should be also considered. Indeed, it was found that the local porosity of the already reduced layer varied also with time. However, the data are not accurate enough to permit the temporal variation of D^{eff} to be included in the model calculation.

The smelting process of titanium-silicon ferroalloy using blast furnace titaniferous slag

L.ZUSHU *et al.*

In this paper the smelting process of titanium-silicon (Ti-Si) ferroalloy using waste blast furnace titaniferous slag containing higher than 20% TiO_2 is introduced. Firstly the principle of smelting Ti-Si ferroalloy by electrosilicothermic method (ESTM) and the effects of DC on the reduction of TiO_2 in molten slag are studied. Ti-Si ferroalloy containing about 20% Ti can be produced by ESTM, and Ti recovery is less than 60%. And then, electrosilicoaluminothermic method (ESATM) is introduced. Ti-Si ferroalloy containing higher than 30% Ti and less than 35% Si can be produced by ESATM, and Ti recovery is higher than 80%. Studies on application of products (Ti-Si ferroalloy and residue slag) of the smelting process have been carried out, and the results show that the electrothermic smelting process can make the waste blast furnace titaniferous slag be used perfectly.

Casting and Solidification

Effect of carbon and sulfur in continuously cast strand on longitudinal surface cracks

K.-H.KIM *et al.*

Effects of carbon and sulfur on the longitudinal surface cracks have been investigated by calculating the non-equilibrium pseudo binary Fe-C phase diagram and introducing the strain in brittle temperature range for continuous casting of steels. The cracking tendency as a function of carbon content was well described by the strain in brittle temperature range. The strain in brittle temperature range was influenced by the other solute elements as well as carbon. The carbon content at which longitudinal surface cracking is maximized decreased with increasing sulfur content. At a given carbon content, the possibility of surface cracking increased with increasing sulfur content.

Analysis and Characterization

Nitrogen trapping to chromium in α iron studied by internal friction and magnetic after-effect techniques

H.NUMAKURA *et al.*

The effect of chromium on the behaviour of nitrogen in α iron has been studied by anelastic and magnetic relaxation measurements on dilute Fe-Cr-N alloys. The presence of chromium is known to give rise to an additional contribution to the Snoek relaxation at a higher temperature than the ordinary Snoek peak. When specimens quenched from a solution-treatment temperature are subjected to isothermal ageing at room temperature, the high temperature component develops while the ordinary component decays. This observation can be explained in terms of redistribution of nitrogen atoms: almost random distribution established at the solution-treatment temperature changes into a metastable distribution, in which a substantial number of nitrogen atoms are bound to chromium atoms. The binding energy of a Cr-N pair has been estimated independently from two experiments: 20 ± 2 kJ/mol from the kinetics of the redistribution and 15 kJ/mol from the characteristics of the Snoek relaxation profile, which are in reasonable agreement with each other.

Microstructure

A comparative study of observed and predicted austenite microstructures during hot rolling of a medium carbon steel

J.J. CRUZ-RIVERA *et al.*

Two-step deformation sequences, applied by laboratory rolling to a medium carbon steel, were used to assess the applicability of the most important models of microstructural evo-

lution to describe the actual behavior of the austenite in this material. The model developed by the IRSID group gave the best overall agreement with the metallographic observations, mainly due to a better prediction of the rates of grain growth in the temperature range of 1 173 to 1 473 K, as compared to the models proposed by the researchers at Sheffield University and Nippon Steel Corp., which predictions resulted grossly inaccurate at the higher temperatures. Nevertheless, all three models contain the essential features to lead to reasonable values of the austenite grain size, especially as the number of deformation steps increases concurrently with a progressive decrease in the temperatures of deformation.

Lattice constants of γ and γ'' phases and γ''/γ lattice mismatches in a Ni-15Cr-8Fe-6Nb alloy

K. KUSABIRAKI *et al.*

The lattice parameters of γ and γ'' phases and the γ''/γ lattice mismatches in a nickel-base superalloy, a modified NCF 3 type alloy, are investigated by X-ray diffraction. The measurement on the γ'' phase is carried out using γ'' precipitates extracted from the alloy. The relationship between the morphology of the γ'' phase and the lattice parameters of the γ and γ'' phases and γ''/γ lattice mismatches is discussed in detail.

With increasing aging time, the lattice parameter of the γ phase decreases and those of the γ'' phase increase. The former after a certain aging time reaches at a constant value at each aging temperature. The changes in the lattice parameters in various aging conditions suggest a change in the composition of the γ and γ'' phases. The γ''/γ lattice mismatch in the direction normal to the habit plane of the γ'' plate, *i.e.* parallel to the c axis is nearly twice greater than that in the plane. The dependence of the morphology of the γ'' phase on the lattice parameters, the γ''/γ lattice mismatches and the axis ratio c_0/a_0 for the γ'' phase is not clear. The γ'' phase precipitated at 1 073 K in a square plate-like morphology is incoherent with the matrix in the direction of the c axis, while that precipitated at up to 1 033 K in a near disc-shaped morphology is coherent.

Crystallography of intermetallic interface layers in hot-dip galvanizing steel sheets

H. OHTSUBO *et al.*

The crystallography of the intermetallic phases forming at the zinc-film/substrate interfaces in hot-dip galvanizing steel sheets has been investigated mainly by means of scanning electron and cross sectional transmission electron microscopies. Zinc atoms penetrating into a ferrite lattice precipitate within the ferrite as monoclinic ξ -phase rods with parallelogram cross sections initially in contact with the interface. The growth direction of the ξ -phase rod is probably the $[001]_{\xi}$ which lies parallel

to a $\langle 111 \rangle_{\alpha}$. The orientation relationship between ξ and the substrate ferrite, $[001]_{\xi} // [\bar{1}11]_{\alpha}$, $(100)_{\xi} // (101)_{\alpha}$, $(010)_{\xi} // (12\bar{1})_{\alpha}$, can be expected by considering both the results of scanning microscopy and the atomic configurations of ξ phase and those of ferrite. The additional heat cycle up to 723 K to produce thin foils for cross sectional transmission electron microscopy after the hot-dip galvanizing process induces the formation of δ_1 -phase at the ferrite/ ξ interphase boundary. No specific orientation relationship between the δ_1 -phase and the substrate ferrite was obtained.

Isothermal transformation products in a Cu-bearing high strength low alloy steel

D. P. DUNNE *et al.*

A time-temperature-transformation (TTT) diagram has been determined for a copper-bearing steel, which is a low alloy TMCP variant of the ASTM A710 type of structural steel. Quantitative measurements have been supplemented by optical microscopy and transmission electron microscopy to investigate the isothermal transformation behaviour as well as the associated precipitate morphologies. It is shown that the kinetics and product phases of the polymorphic transformation and precipitation reactions are sensitive to both temperature and time. The TTT-diagram shows a prominent transformation region for bainitic structures, at temperatures intermediate between those of polygonal ferrite and martensite. In the intermediate region, the microstructures were characterised by a ferritic matrix with a lath and/or plate shaped grains containing a high dislocation density, together with a minor dispersed "island" phase. For a short holding time (5 sec) at intermediate temperatures (530–440°C), the island phase was identified as untempered twinned and lath martensite, autotempered twinned and lath martensite, and martensite/austenite constituent, depending on the level of carbon partitioning in the remaining austenite before quenching in water. For a longer holding time, the carbon enriched austenite regions decomposed to carbide and ferrite by coupled growth. Polygonal and quasi-polygonal ferrite were formed at relatively high transformation temperatures and these microstructures contained a low dislocation density and were associated with interphase ϵ -copper precipitates.

Physical and Mechanical Properties

Influence of microhardness and inclusion on stress oriented hydrogen induced cracking of line pipe steels

A. TAKAHASHI *et al.*

The influence of microstructure and inclusion on the susceptibility to stress oriented hydrogen induced cracking, SOHIC, or type II sulfide stress cracking, SSC, was investigated for two kinds of line pipe steels which have the

different susceptibility to SOHIC. The steel plates, produced by accelerated cooling after controlled rolling, were heated to three temperatures of 1 100, 900 and 750°C to vary their microstructures. For the four microstructures including the as cooled one, the susceptibility to SOHIC was evaluated by the SSC test specified in NACE TM0177-90 method A in addition to an SSC test using a large scale plate.

The cracking susceptibility depends on the minimum microhardness rather than maximum hardness in the specimens with yield strength of API X65 or less. The local region with minimum microhardness, having lower yield strength, is deduced to become a preferential site for the hydrogen induced blister cracking, HIBC, and also to provide plastic zones through which the existing blisters are prone to link to each other, leading to the cracking through thickness. As a consequence, the decrease in the local hardness is concluded to enhance cracking susceptibility. Non-metallic inclusions were identified as the crack initiation site. The critical dispersion distance of inclusions, within which a second blister can be induced in the plastic zone formed around the first blister, is estimated based upon calculation of the stress around the blister. The observed results of crack propagation and arrest were both well interpreted in terms of the estimated critical distance.

Improvement of yield strength-transition temperature balance by microstructural refinement

A. TAKAHASHI *et al.*

Grain-refinement simultaneously increases yield strength and toughness while the strengthening without the grain-refinement still deteriorates the toughness. It is worth assessing how the strengthening relates to the toughness to strengthen steels most appropriately. In this study, different mechanisms for strengthening are compared with respect to the relationship between yield strength and ductile-brittle fracture transition temperature. Considered are the grain-refinement by solute copper or thermo-mechanical control process (TMCP), the precipitation-hardening by copper, and the bainite-transformation hardening with alloying elements such as boron, molybdenum, and nickel. The transition temperature rises with strengthening by the precipitation-hardening and the transformation-hardening. In this case, the relationship is approximately linear with the gradient of 0.46°C/MPa. In contrast, the transition temperature lowers in the case of the strengthening by the grain-refinement achieved by the solute copper and the TMCP. This study aims at relating the yield strength to the transition temperature on a dislocation crack model of brittle fracture. Considering the energy balance for brittle crack initiation and the temperature dependence of the friction stress for the Hall-Petch relationship gives the linear lowering of transition temperature with increasing

yield strength by grain-refinement, of which gradient is estimated at approximately 0.80 °C/MPa. The comparison of experimental results with this relationship indicates that the strengthening by solute copper effectively utilizes the grain-refinement.

The effect of austenitizing process on the hardening behaviour of Cr-Mo-Mn-C air-hardening cast tool steel

C. WU et al.

The hardening behaviour of Cr-Mo-Mn-C air-hardening cast tool steel was investigated by using an electron microprobe analyser (EPMA), a transmission electron microscope (TEM), an energy dispersive X-ray spectrometer (EDS), as well as a scanning electron micro-

scope (SEM). The results obtained in the present investigation show a significantly different behaviour in the Cr-Mo-Mn-C air-hardening cast tool steel. It is concluded that the performance of (Fe,Cr)₃C dissolution and (Cr,Fe)₇C₃ reprecipitation during austenitizing is a key factor controlling the hardening behaviour as well as ultimate hardness obtained in air cooling.

Prediction of low cycle fatigue lives of low alloy steels

T. GOSWAMI

An assessment of low cycle fatigue data was conducted in this paper with two new life prediction methods. A phenomenological me-

thod was developed that accounts for the flow characteristics as a result of loading in terms of dynamic viscosity. The other method is a statistical method known as Diercks equation that has been used as a "first approximation" of low cycle fatigue behavior of steels in the literature. The Diercks equation has been modified in this paper and life prediction analyses were conducted and compared against the other method.

The phenomenological method predicted low cycle fatigue lives conservatively under different temperatures and strain rates whereas, modified Diercks equation had several limitations that were identified in this paper. Both the methods were found to correlate the low cycle fatigue data within the acceptable band of $\pm \times 2$.