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International Conference on Fracture
Mechanics Technology Applied to Mate-
rial Evaluation and Structure Design,
Melbourne, Australia, August 10-13 1982

CRITERION FOR FRACTURE OF PLAIN CONCRETE

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The crack behaviour of concrete in simple compression has two stage until failure. The first stage is about 30% of the ultimate strength, which corresponds to the bond crack formation of coarse aggregate and below this stage the two components of concrete work together and concrete is an elastic body. The second stage is about 80% of the ultimate strength which corresponds to the onset of continuous cracking due to the extension of the bond cracks of coarse and fine aggregate in matrix. The behaviour of concrete is quasi-elastic. The fracture mechanism of concrete is closely connected with the formation of the bond cracks. On the base of the behaviour the criterion for the failure of concrete is analyzed by the model structure of concrete. The criterion is analogous to the octahedral stress theory, which enables us to consider the quasi-elastic behaviour due to the physical properties of aggregate and matrix and this criterion is also available for the application of the ultimate strength envelope of plain concrete under compression and shear stresses.

The 8th Congress of The International
Ergonomics Association (IEA '82), Aug.
23-27, 1982, Tokyo

Required Clearance for Passages in Dwelling Houses

Yoshiro KOH and Kiyoko KAMADA

An experimental study on the required clearance for passages in dwelling houses was carried out by changing the height and width of doorways, and the width of passages between walls and furniture. The behaviour of the research subjects were recorded by TV-recorder and analysed based on a psychological assessment of five or three ratings of satisfaction. The necessary height and width of passages, such as doorways, entrance hall and passages between walls and furniture, were given by selecting a limit of confidence for a suitable percentile of normal distribution, based on standing height or the maximum breadth of the human body in a natural posture, according to the rating of satisfaction.

Considering the recent increase of body height in the young people in Japan, the door height in dwelling houses should be designed 20-30cm higher than the standard size. The minimum width of passages shown in literature as the data for designs, which were too small to walk or work with any degree of comfort should be enlarged.

The Fourth International Symposium on
the Use of Computers for Environmental
Engineering Related to Building, March
30-April 2, 1983 Tokyo, Japan

A SUCCESSIVE INTEGRATION METHOD FOR THE ANALYSIS OF THE THERMAL ENVIRONMENT OF BUILDING

Part 2 Simplification and Applications for the Non-Linear Problems

Noboru ARATANI and Yasuhiko KONNO
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This calculating method for the analysis of thermal environment of building (part 1) was reported at the first symposium in Washington. Main features of this method are :

Successive calculation method of integral equation of heat flow using the convenient nature of exponential function

Easiness of changing Δt in the way of calculation

Heat balance equation using integrated value of heat flow in each Δt step

In this report some improvements have been made using designated series of exponents for the approximation of indicial response of heat flow and they make it possible to minimize the memories, to simplify the program and to use a personal computer for the calculation of multi-room problems.

Third Pacific Chemical Engineering
Congress (PACHEC '83), Seoul, Korea,
May 8-11, 1983

HIGH CAPACITY DEPTH FILTER

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Sapporo 060, JAPAN

To increase the capacity of a depth filter with high performance, the authors proposed a dual-floor depth filter with an upper Rasching ring bed and a lower sand bed. Effectiveness of the high capacity filter was proved by pilot plant studies. Mechanism of the upper Rasching ring bed was analysed and a kinetic model of the removal process was proposed.

The 8th Congress of the International
Ergonomics Association, August 23-27,
1982, Tokyo, Japan

SEVERAL ASPECTS OF THE PREDICTION METHOD OF LOCAL MUSCLE ENERGY METABOLIC RATE

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**Department of Hygiene, School of Medicine, Showa University, Tokyo, Japan

A prediction method of local muscle energy metabolic rate was presented and has been developed by the authors. Local muscle energy metabolic rate is a valuable piece of information for establishing a thermoregulation model, because it affords a main source of heat production in the body.

By using it, the quantitative evaluation can be decided not only among different exercise items in a muscle group but also among different muscle groups during an exercise item.

The method also gives information concerning the maximum energy metabolic rate in a muscle group and quasi-basal metabolic rate of internal organs excluding those of muscle groups.

In this paper several aspects of the prediction method and their utilization examples were described.

23rd us Rock Mechanics Symposium,
Texas (USA) 1983

Fracture Initiation and Breakdown Pressure — Are They Similar?

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Associate Professor Department of Resources and Development Engineering

J. C. ROEGIERS

Senior Researcher Dowell Division of Dow Chemical USA

There exists abundant evidence suggesting that under the microhydraulic fracturing the crack is propagating, well before the maximum borehole pressure is reached. This paper investigates the initiation phenomenon using a newly developed numerical approach, the Body Force Method. It enables evaluation of the stress intensity factor under quasi-static conditions. The coupling with the seepage phenomenon around the borehole and through the fracture faces is accomplished using a Line Source Flow Distribution Method allowing incorporation of transient pore pressure effects. The approach is extremely efficient and requires a minimum of computing time.

The proposed numerical method, limited to two-dimensional analyses so far, has been used to investigate the sensitivity of the fracture initiation process to (1) the preexisting crack size, (2) the pumping rate, (3) the in-situ stress, (4) the formation permeability and (5) the fluid viscosity.

5th Congress of the International Society
for Rock Mechanics, Melbourne
(Australia) 1983

Strata Control in Deep Coal Mine in Hokkaido
— **In-situ Monitoring and Interpretation**
of Stress Changes in Coal Seams

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Dept. of Resources Development Engineering, Hokkaido Univ.

It has been shown from case studies that the occurrences of coal bumps in deep coal mines in Hokkaido, Japan, was restricted to the zone around coal pillars which had a sandstone roof measuring more than four times the thickness of the coal seam. In order to clarify the mechanism of coal bumps, laboratory tests, field measurements and numerical analyses on the coal seam behaviour were performed. As a result, it was ascertained that, when coal fails, a roof composed of sandstone supplies a large amount of energy to the coal seam compared to that composed of shale.

Furthermore, both the mode of energy release and the patterns of stress changes are remarkably disturbed when an irregular distribution of mechanical strength of coal appears in the coal seam. There two conditions are the most important factors causing coal bumps, so that the measurement of stress changes in coal seam is effective to predict coal bumps.

1st International SME-AIME Fall Meet-
ing, Honolulu, Hawaii, September 4-9,
1982

A Review of Coal Preparation in Japan
— **Its Process and Operation**

Takakatsu TAKAMORI
Faculty of Engineering, Hokkaido University.

This paper reviews the present status of coal preparation in Japan, mainly on the process in coal cleaning and waste water treatment. This paper also discusses recent topics in maintenance, automatic control, raw coal management, etc.

14th International Mineral Processing
Congress, Toronto, Canada, October 17-
23, 1982

**Separation of Calcite from fluorite Ore by
the Adsorption-Washing-Flotation Method**

Takakatsu TAKAMORI and Masami TSUNEKAWA
Faculty of Engineering, Hokkaido University.

In the fluorite-calcite water system, the property of aqueous phase is remarkably affected by calcite, even if the ratio of calcite to fluorite is very low. Dissolution of chemical species from both minerals and adsorption of these chemical species on mineral surface suitable for adsorption occur simultaneously in the system, and the concentrations of the chemical species are regulated by the solubility product.

Effects of calcite on the flotation behavior of fluorite through an aqueous solution in the system were investigated using an apparatus developed in our laboratory. It was confirmed that the floatability of fluorite with SDS was markedly depressed by carbonate species.

Regarding adsorption of SDS on fluorite, both the chemical adsorption forming surface calcium dodecyl sulfate and electrostatic adsorption should be considered. On the other hand, adsorption of SDS on calcite would be mainly electrostatic.

On the basis of these results, the Adsorption-Washing-Flotation Method was developed. When the final flotation pH was below 7, fluorite was preferentially floated by this method.

International Symposium on Biohydro-
metallurgy, Cagliari, Italy, May 1-4, 1983

**Some Properties of *Thiobacillus Ferrooxidans* and
Applications of the Properties to Improvement of
the Rate of Sulfide Mineral Leaching**

Takakatsu TAKAMORI, Hideo KAKUTA and Masato SUMIYA
Faculty of Engineering, Hokkaido University.

By the chemotaxonomy, the microorganism used in the present study was identified as *Thiobacillus ferrooxidans* and verified to have no contamination. In the nitrogen source free medium, *T. ferrooxidans* showed sufficient ability to oxidize ferrous iron and leach chalcopyrite for at least 24 hours without growth.

It was found in the batch and continuous chemostat culture that the enrichment of carbon dioxide in air remarkably improved the specific growth rate and growth yield. A small amount of copper sulfate addition to 9 K medium contributed to the increase of the specific growth rate and growth yield. It was found that copper leaching rate was improved depending upon the cell concentration at cell number above 10^8 cell/ml.

U. S.-JAPAN Cooperative Science Program on Solidification Processing, Dedham, Massachusetts, U. S. A., June 26-29, 1983

SPECIFIC PERMEABILITY OF AN Al-Si ALLOY BY SEEPAGE METHOD

Tadayoshi TAKAHASHI

Department of Metallurgical Engineering, Hokkaido University, Sapporo

The interdendritic liquid was allowed to flow into a cylindrical hole formed in the solid-liquid coexisting zone due to metallostatic pressure because of the height of the zone (i. e. seepage method), and the fluidity of the liquid in the zone of Al-2.4mass%Si alloy was investigated.

From the relationship among effective permeability coefficients, namely the volume fraction of solid and cooling rate at the initiation of solidification, an empirical equation related to the effective permeability coefficient was established. The density and viscosity of the liquid during solidification were calculated and specific permeability was determined by the effective permeability coefficient and those values.

Furthermore, the minimum specific permeability above which the interdendritic liquid could not flow under solidification contraction was determined. The pressure drop due to the contraction was calculated by using its value and this coincided with the density change in an unidirectionally solidified ingot.

The 4th International Conference on Boundary Element Methods in Engineering, Southampton, England, September 21-23, 1982.

Application of Indirect Boundary Integral Method to Three-Dimensional Problems in the Linear, Couple-Stress Theory

Michiya KISHIDA and Kazuaki SASAKI

Department of Mechanical Engineering

This paper describes a numerical approach by the indirect boundary integral method to three-dimensional boundary value problems in the linear, couple-stress theory. In this approach, we introduce appropriate potentials, corresponding to those for a concentrated force and a couple in an infinite medium, and reduce the problem to solving the simultaneous Fredholm type integral equations of the first kind.

As concrete examples, the stress concentration problems are treated for a circular cylinder with arc-shaped annular groove under uniaxial tension and simple torsion, respectively.

From the results, the influences of parameters, such as Poisson's ratio, characteristic length and the ratio of bending-twisting moduli, newly defined in the couple-stress theory, on stress concentration factor are made clear.

SIXTH INTERNATIONAL CONFERENCE ON WIND ENGINEERING
Gold Coast, Australia, 21-25 March, 1983

Free-Stream Turbulence Effects on a Separation Bubble

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Hokkaido University, Sapporo, 060, Japan

Effects of free-stream turbulence on a separation bubble formed along a side of a blunt flat plate with right-angled corners were studied. The free-stream turbulence was generated by a thin circular rod which was placed at a certain distance upstream of the plate along its stagnation streamline. Various characteristic parameters of the separation bubble such as the bubble length, the integral length scale of large-scale vortices, the maximum pressure difference in the bubble, a few characteristic lengths of the time-mean pressure profile etc. were fairly well correlated with the turbulence intensity just outside the separated shear layer near the edge. The turbulence length scale there was a fairly unique function of the turbulence intensity. Statistical properties of the fluctuating surface pressure were also studied and used to demonstrate the large-scale vortices in the reattaching zone. The maximum r. m. s. surface pressure was found to be estimated in terms of the longitudinal gradient of the time-mean pressure and the length scale of vortices in the reattaching zone.

7th International Heat Transfer Conference, Munich, Germany, September 6-10, 1982

CONVECTION IN A HORIZONTAL FLUID LAYER HAVING A SHEAR-FREE UPPER SURFACE AND UNIFORM VOLUMETRIC ENERGY SOURCES

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**Department of Mechanical Engineering, Hokkaido University

Measurements were made on the temperature distribution and heat transfer in a horizontal layer of dilute electrolyte which has a uniformly-distributed volumetric energy source provided by an alternating current passing horizontally through the layer. The fluid is on a rigid bounding plate maintained at constant temperature. The upper surface of the liquid is in contact with a thin layer of helium to maintain an almost shear-free boundary. The temperature of this liquid-gas interface is maintained equal to the temperature of the lower solid-liquid interface. Horizontally-averaged temperature profiles of the electrolyte are determined with an interferometer. A comparison of the results obtained with rigid upper and lower boundaries under similar conditions indicates that with the free upper surface there is a higher heat transfer coefficient at the upper surface, a somewhat smaller coefficient at the lower surface, and a decrease in the maximum temperature of the layer.

18th Intersociety Energy Conversion
Engineering Conference (IECEC)
Orlando, Florida, U. S. A., August 21-26,
1983

ENERGY-CONSERVING HEAT PUMP-BOILER SYSTEMS FOR DISTRICT HEATING

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The energy saving potential of a proposed heat pump-boiler system for district heating is analyzed. Fuel is supplied to a boiler which generates steam to drive a turbine. The turbine output is used to power a heat pump which takes energy from the environment. Introduction of a screw type expander in place of the throttling valve in the heat pump cycle is planned to increase the system performance. District heating is provided by hot water which is heated as it flows through the condensers in the heat pump and turbine cycles. Both series and parallel connected condenser arrangements are considered. Results show that the heat supplied to the water for district heating can be as high as 200 percent of the heating that would be provided by use of the fuel supplied to a conventional boiler system with a thermal efficiency of 90 percent.

Society of Automotive Engineers Inter-
national Off-Highway Meeting
Milwaukee, Wisconsin, U.S.A.,
September 13-16, 1982

A Method to Improve the Solubility and Combustion Characteristics of Alcohol-Diesel Fuel Blends

Tadashi MURAYAMA, Noboru MIYAMOTO, Takanobu YAMADA, Jun-ichi KAWASHIMA
Dept. of Mechanical Engineering, Hokkaido Univ.

and Koichiro ITOW
Komatsu Ltd.

This paper reports the results of two parallel investigations: An investigation on the solubility of alcohols in diesel fuels, and the diesel engine performance with the blended fuels.

The investigation proposes an empirical formula for the solubility of alcohols in diesel fuels, as a function of temperature, water content, additive concentration and specific gravity of the diesel fuel.

The engine performance when using the blended fuels was also investigated. Compared with conventional diesel fuels, the blended fuels show promise of better thermal efficiency, smoke free operation, and reduction of HC, NO_x, and CO emissions.

Society of Automotive Engineers International Congress & Exposition, Detroit, Michigan, USA, February 28-March 4, 1983

Elimination of Combustion Difficulties in a Glow Plug-Assisted Diesel Engine Operated with Pure Ethanol and Water-Ethanol Mixtures

Tadashi MURAYAMA, Noboru MIYAMOTO, Takemi CHIKAHISA, and Hideyuki OGAWA
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Forced ignition with glow plugs has a great potential for the utilization of alcohol fuels in diesel engines. However, the installation of glow plugs may cause misfiring or knocking in parts of the operating range. This paper presents an analysis of the factors influencing the ignition characteristics of ethanol in a glow plug-assisted diesel engine ; these factors may be classified into two categories : the factors related to the temperature history of the droplets before contact with the glow plug, and those related to the probability of contact. By optimizing these factors, the combustion difficulties were successfully eliminated over the whole operating range, and engine performance comparable with conventional diesel operation was achieved.

1983 ASME-JSME Thermal Engineering Conference, March 20-24, 1983
Honolulu, U.S.A.

Effects of Nitric Oxide on Formaldehyde Formation in Exhaust Gases from a Methanol Fueled S. I. Engine

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Department of Mechanical Engineering, Hokkaido University, Sapporo, Japan

This paper describes an approach to a simplified chemical kinetic model (S-model) for predicting formaldehyde formation in an engine exhaust system and theoretical and experimental details of the effects of nitric oxide on the methanol and formaldehyde reaction behaviors.

The developed S-model consisted of 45 elementary reactions involving 18 chemical species. The computer time was reduced to approximately one tenth of that required by the previous detailed model (D-model).

Methanol oxidation was faster with an increase of nitric oxide and oxygen in exhaust gases. The presence of oxygen had favorable effects on formaldehyde formation, while the presence of nitric oxide had favorable effects on formaldehyde destruction.

The results of chemical kinetic analysis agreed well with the experimental results. It was theoretically and experimentally determined that the conversion reaction of nitric oxide to nitrogen dioxide played a significant role for methanol oxidation and formaldehyde formation.

All Division 5 Conference of International Union of Forestry Research Organizations, Madison, Wisconsin, U.S.A., June 27-July 5, 1983

The Influence of Tooth Tip Radius and Chip Pocket Volume on Cutting Forces

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Hokkaido University

In the cutting action of saw teeth the tooth sharpness and the chip pocket space have a great influence on the power demand and energy consumption.

Experiments on these factors were carried out.

Relationships of the tooth tip radius and the chip pocket volume to the cutting force and the ploughing force are obtained.

The cutting force and the ploughing force which has been noted in metal cutting increase by increasing the tooth tip radius, and a marked tendency is observed in the range of common radii.

Grinding and blunting of teeth are of importance as well as the other factors in the cutting conditions.

When the sawdust is packed full in the chip pocket and the volume ratio of the packed sawdust to the chip pocket becomes high, the cutting force increases by about ten times that of the low volume ratio.

A strong correlation is observed between the cutting efficiency and the volume ratio.

Ninth Canadian Congress of Applied Mechanics, Saskatchewan, Canada, May 30-June 3, 1983

Free Vibration of a Circular-Segment-Shaped Plate of Rectangular Orthotropy

Toshihiro IRIE, Gen YAMADA and Yukinori KOBAYASHI
Department of Mechanical Engineering II, Faculty of Engineering
Hokkaido University, Sapporo, 060 Japan

An analysis is presented for the free vibration of a circular-segment-shaped plates of rectangular orthotropy. A clamped circular-segment-shaped plate is formed on a simply supported rectangular plate by clamping an edge of the extended plate and the circular segment inside it. With the reaction force and the bending moment acting along the segments regarded as unknown harmonic loads, the stationary response of the extended plate to these loads is expressed by use of Green's function. The unknown force and moment distributions along the segments are expanded into Fourier sine series with unknown coefficients, and the linear homogeneous equations for the coefficients are derived by restraint conditions at the segments. The natural frequencies and the mode shapes of vibration of the actual plates are determined by calculating the eigenvalues and the eigenvectors of the equations.

UTAM Symposium on Turbulence and
Chaotic Phenomena in Fluids,
September 5-10, 1983 Kyoto, Japan

**The wall pressure fluctuation and the turbulent structure
of a boundary layer**

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*Hokkaido Institute of Technology

**Nihon University

***Hokkaido University

The wall pressure fluctuations of a turbulent boundary layer are characterized by two groups, one which decays slowly and moves at nearly the same speed of the outer flow and one which decays ten times faster and moves at the half speed of the outer. The correlation measurements between wall pressure and velocity fluctuations show that the slowly decaying pressure corresponds to the large scale vortex-like motion which governs the outer layer of the the boundary layer, while the rapidly decaying one is closely related to the burst which appears in the inner layer close to the wall. The burst appears at the fixed phase of the large scale motion (bulge) suggesting that the bulge causes the burst to occur.

International Workshop on the Role of
Chaos in Biological Information Proces-
sing, Stuttgart, W-Germany, Feb. 17-18,
1983

**Decaying Property of Correlation Function in
a Chaotic Dynamical System**

Tomomasa NAGASHIMA

With the intent of understanding the characteristics of the statistical properties in chaotic dynamical systems, an attempt was made to classify the modulation property of correlation function in a certain family of chaotic dynamical systems. It was reported that a chaotic property in the orbit structure can be hereditary to the temporal development of correlation function in chaotic dynamical systems.

The Institute of Electrical Engineering
of Japan INTERNATIONAL POWER
ELECTRONICS CONFERENCE, March
27-31, 1983 Tokyo, Japan

A Microprocessor-Controlled Speed Regulator for Commutatorless Motor Drives

Shoji FUKUDA, Yuzo ITOH and Akio NII
Department of Electrical Engineering

The presented study describes a new control method in which a microprocessor is used to implement all monitoring and control functions of a speed regulator system for a commutatorless motor. According to the increase of a load, a decrease of an effective leading angle and an increase of an overlapping angle of an inverter reduces a commutation margin angle, and the inverter will fail in commutation. To prevent the failure, the presented strategy contains a speed, current, a margin angle and an air gap flux control loops which are executed with the microprocessor. A distinct feature of this control scheme is that a power factor and an efficiency are optimized, and the overload capacity is extended.

A speed regulator using a microprocessor was tested and the efficiencies of the presented control scheme and the conventional one were measured. The presented strategy gives a higher efficiency all over the operating range.

12th European Microwave Conference,
13-17 Sept., 1982, Finlandia Hall, Helsinki,
Finland

New Polarization Switching Technique of Microstrip Antenna

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In electromagnetic remote sensing and inverse scattering technique, polarization information is important since an electromagnetic scatterer acts in a manner similar to a polarization transformer. Therefore, polarization switching of an antenna is needed in the above-mentioned applications as well as communications. This paper deals with a new technique of polarization switching using a rectangular microstrip patch antenna which is coupled in close proximity to a microstrip feed line. The polarization of the patch antenna is switched by ON-OFF of the end of the feed line. ON-OFF switching of the feed line is performed at a high speed by a simple electronic ON-OFF device such as a PIN diode mounted on the feed line.

First, the operation principle is described. Then, the paper gives some experimental results in order to prove the validity of the principle.

Fourth International Conference on Integrated Optics and Optical Fiber Communication, June 27-30, 1983, Tokyo, Japan

Finite-Element Analysis of Anisotropic Optical Waveguides

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Several methods for the analysis of a three-dimensional optical waveguide have been proposed, and the vectorial finite-element method in axial-components formulation is widely used. However, the vectorial finite-element solutions have been known to include nonphysical spurious solutions. If one desires to compute a set of eigenmodes, it is difficult and very cumbersome to distinguish between the spurious and physical modes of the waveguide. Recently, Mabaya, Lagasse and Vandembulcke presented an approximate scalar finite-element formulation for the analysis of the isotropic waveguides. In this approach, the nonphysical spurious solutions do not appear.

In this paper, this approximate scalar finite-element method is extended to the anisotropic waveguides having a permittivity tensor with nonzero off-diagonal elements. For a two-dimensional waveguide, the matrix equations derived by this approach are reduced to the exact expressions for two-dimensional guided modes.

Second International Symposium on Spouted Beds, Vancouver, Canada, October, 1982

Particle Segregation in a Spouted Bed of Binary Mixtures of Particles

O. UEMAKI, R. YAMADA and M. KUGO

Faculty of Engineering, Hokkaido University

Minimum spouting velocity and segregation behaviour of binary mixtures of particles differing in size were studied. The experiments were carried out in a bed of 20cm diameter at superficial gas velocities up to 1.3 Ums by use of silica sand of four different particle sizes from 0.655 to 2.23mm.

An empirical equation was proposed for minimum spouting velocity of binary mixtures. The effects of the particle size difference and the superficial gas velocity on segregation were investigated. Results showed that considerable radial segregation as well as axial segregation occurred even for high gas velocity under the condition of large particle size difference.

International Symposium on Carbon
1982, Toyohashi, Nov. 1982

Thermal Decomposition Reaction of Pitches

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Benzene soluble fraction of coal tar pitch and of Minus vacuum residual oil were heat treated under vacuum (in a closed system) or under 3 or 10 MPa of nitrogen atmosphere and the products were fractionated by solvents.

Both the lighter and heavier fractions increased with time. This is interpreted by the fact that initially thermal cracking produced free radicals which abstract hydrogen from other molecules to stabilize small lighter fractions and the other part of radicals or the molecules from which hydrogen was abstracted polycondensate to produce the heavier fraction. Under higher nitrogen pressure, the lighter fraction produced remained in the liquid phase and took part in the reaction to reduce the production of lighter and heavier fractions.

Pan-Pacific Synfuels Conference,
Tokyo, Nov. 1982

Liquefaction of coal with metal-water system, (1) Zinc and water system

F. MONDRAGON, H. ITOH and K. OUCHI

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Zinc and water react at a higher temperature than 400°C, producing nascent hydrogen which can effectively hydrogenate coals. Solvents like naphthalene or wash oil are very effective to obtain high conversions. The lower rank coals show a higher conversion and the higher rank coals give lower conversion. The highest benzene conversion of 94% of Taiheiyo coal was obtained in the reaction at 445°C for 1h using wash oil as solvent.

Fourth International Conference on
Organic Synthesis (IUPAC) August 22-
27, 1982 Tokyo. JAPAN

Haloboration and its Application to Organic Synthesis

Shoji HARA, Hidetaka DOJO, Yoshitaka SATOH, Satoru TAKINAMI, Hiroko ISHIGURO,
and Akira SUZUKI

Department of Applied Chemistry, Faculty of Engineering

Recently, stereodefined alkenyl halides have been used as important intermediates for the stereoselective synthesis of olefinic compounds. We have found that B-Br- or B-I-9-BBN reacts with

1-alkynes to give (Z)-2-bromo- or (Z)-2-iodo-1-alkenylboranes. These reactions are completely regio-, stereo- and chemoselective. The boron-halogen bond adds to the triple bond through a cis-manner and the boron atom attaches to the terminal carbon of 1-alkynes. On the other hand, alkenyl, halo and ester groups can be tolerable to B-Br-9-BBN. The protonolysis of the alkenylboranes is easily achieved by the addition of acetic acid to give the corresponding 2-halo-1-alkenes.

Although alkenylboranes are not stable under basic conditions, they react with lithium acetylide and iodine at low temperature without significant decomposition to give (Z)-1-(1-alkynyl)-2-halo-1-alkenes in good yields.

1983 International Symposium on Wood
and Pulping Chemistry, Tsukuba
Science City, Japan, May 23-27, 1983

An Aspect on the Irreversibility between Cellulose I and II Families

J. HAYASHI and M. TAKAI

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Hokkaido University

We have found that there is an irreversibility of transformations between cellulose I family and cellulose II family as in Cellulose I and II.

It was proposed in the previous paper that the skeletal chain conformations for cellulose I family and II family are different from each other and the fact results in the irreversibility.

Blackweell and Sarko have proposed that the crystal structures in cellulose I family and II family are based on parallel chain and antiparallel chain respectively, and the irreversibility results from the difference of chain polarity. IR and ^{13}C NMR spectra on solid samples of cellulose modifications were analysed to prove our conclusion.

In both spectra, the bands or signals related to chain conformation split into two peaks in cellulose II family (II, III_{II}, and IV_{II}) and did not split in cellulose I family (I, III_I, and IV_I). These results showed different states around glucosidic bond in two families and supported our conclusion.

Blackwell proposed a mechanism of reverse of chain direction during the transformation from Cellulose I to II in fibrous state. By the mechanism, sheet plane of chains must be change from (1 $\bar{1}$ 0) in Cellulose I to (0 2 0) in Cellulose II. The possibility of that was examined and a few other observations were made.

3rd International Symposium on "Preparation of Catalysts", Louvain-la-Neuve, Belgium, September 6-9, 1982

Preparation of Copper Supported on Metal Oxides and Methanol Steam Reforming Reaction

H. KOBAYASHI, N. TAKEZAWA, M. SHIMOKAWABE and K. TAKAHASHI
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The effects of the catalyst preparation on the title reaction were studied over various copper-containing catalysts. It was concluded that highly dispersed CuO clusters formed preferentially on metal oxides at high copper loading or high calcination temperature. These precursors were readily reduced to metallic copper under reaction conditions and provided highly active and selective catalysts.

The support effect on the reaction emerged when the metallic copper surface area exceeded 100 m²/g Cu. The precursors on Al₂O₃, ZrO₂ and MnO₂ were highly susceptible to the reduction and fine particles of metallic copper were formed on these oxides.

Invited Lecture IUPAC 28th Macromolecular Symposium, Amherst, Mass., USA, July 12-16, 1982

Gamma Radiation Induced Cross-Links Studied by CP-MAS NMR

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Chemical shift of ¹³C-NMR observed by the CP-MAS NMR spectrometry provides us interesting information on specification of cross-links induced by gamma irradiation.

A new peak appears weakly but clearly after the irradiation and the relative intensities in the spectra were changed before and after the irradiation. The relative concentrations of the cross-linking points, chemical species of which were assigned from the observed chemical shifts, were evaluated from their peak heights. The concentrations were compared with the concentration of the cross-links estimated by the Charlesby-Pinner Plot derived from the measurement of gel fractions. The concentrations estimated from the CP-MAS spectra were larger than those from the gel fraction measurements and origin of the difference was reasonably discussed.

IUPAC 28th International Symposium
on Macromolecules, Amherst,
Massachusetts, U. S. A., July 12-16, 1982.

**Piezo-and Ferro-Electric
Properties of Polyvinylidene Fluoride**

Akira ODAJIMA

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In this paper we deal with piezoelectric properties of β -phase polyvinylidene fluoride (PVDF) in relation to its ferroelectric domain morphology. Up to the present two kinds of models have been proposed for the domain morphology ; one is a lamellar type of domain¹⁾ and the other is a spherical domain²⁾. In order to clarify which model is more appropriate for the domain morphology of PVDF, we have measured both the piezoelectric constant e_{33} and the residual polarization P_r . From a comparison of the experimental ratio, e_{33}/P_r , with the theoretical ones of the lamellar and spherical domains, it is concluded that both the domain models are not appropriate. Thus a new type of the domain morphology has been proposed to explain the temperature change in the observed ratio, e_{33}/P_r .

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- 1) M. G. Broadhurst et al : J. Appl. Phys. **49** 4992 (1978).
 - 2) Y. Wada and R. Hayakawa : Ferroelectrics **32** 115 (1981).

Hydrological Aspects of Alpine and
High Mountain Areas, IAHS Exeter
Symposium, 19-30 July 1982

**A simplified model for estimating glacier
ablation under a debris layer**

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In order to predict the ablation of glacier ice under a debris layer, a simple model is proposed. Required data for the prediction are global radiation, air temperature, the degree-day factor around the area to be investigated, albedo of debris-free ice, and critical thermal resistance of the debris cover. It is shown that this latter variable can be estimated from the four former variables, which are comparatively easy to measure or estimate. Regarding the physical properties of the debris layer *per se*, its albedo and thermal resistance need to be given. The predictions of this model compare favourably with field observations.

VI International Symposium on the
Physics and Chemistry of Ice, Rolla,
Missouri, U.S.A., Aug. 2-6, 1982

Effect of the Hydrostatic Pressure on the Rate of Grain Growth in Antarctic Polycrystalline Ice

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Abstract : In view of the importance of the growth of recrystallized grains for clarifying the mechanism of the depth variation of the ice fabric near the bottom of the ice sheet, experiments of the normal grain growth of ice were carried out under a hydrostatic pressure up to 50 MPa. Specimens prepared from both the Antarctic deep ice core and artificially grown polycrystalline ice were observed in a high pressure tank.

Growth processes were analysed from intermittently taken photographs and they obeyed the relationship $D^2 - D_0^2 = Kt$ between the average grain diameter D and the time t . The activation energy of the grain growth was obtained from the temperature dependence of the rate constant K and it was 0.7~0.8 eV at a low temperature range. The pressure dependence of the K gives the activation volume of the grain. It is $-6.7 \times 10^{-5} \text{m}^3/\text{mole}$ for the Antarctic ice and $-0.8 \times 10^{-5} \text{m}^3/\text{mole}$ for the artificial ice. Implications of large values of the activation energy and the activation volume in the grain growth compared to those for the self-diffusion in bulk ice are discussed.

VI International Symposium on the
Physics and Chemistry of Ice, Rolla,
Missouri, U.S.A., Aug. 2-6, 1982

Generation and Absorption of Dislocations at Large-Angle Grain Boundaries in Deformed Ice Crystals

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X-ray topographic observations were carried out on the large-angle grain boundary (GB) in ice bicrystals during deformation. Misorientations of the bicrystals were made as close as possible to the $\langle 10\bar{1}0 \rangle / 34.1^\circ$ CSL. The section topographs of the GB in equilibrium after a long term annealing revealed no long-range stress field associated with the GB but there appear in the topographs strong stress fields at intersections of GB facets when the GB was subjected to shear stress.

Traverse topographs were taken during compressive deformation of bicrystal specimens to observe generation and absorption of lattice dislocations at the GB and their behavior under stress. It was found that dislocations are generated from the GB when it was subjected to shear stress. When the GB was subjected to only compressive stress, absorption of dislocations occurred at the GB. Generation and absorption mechanisms of lattice dislocations are proposed on a basis of the

structure of the faceted GB composed of stable CSL lattice plane and intrinsic grain boundary dislocations (GBD_s).

VI International Symposium on the
Physics and Chemistry of Ice, Rolla,
Missouri, U.S.A., Aug. 2-6, 1982

Formation and Annihilation of Stacking Faults in Pure Ice

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Both the formation and annihilation processes of stacking faults in fresh ice crystals grown from high purity water were studied by X-ray topography. Stacking faults with the fault vectors $\mathbf{R} = \frac{1}{6} \langle 20\bar{2}3 \rangle$ and $\mathbf{R} = \frac{1}{3} \langle 10\bar{1}0 \rangle$ were observed in dislocation loops of interstitial type formed during crystal growth.

Formation mechanism of such stacking faults is discussed in terms of segregation process of excess interstitial water-molecules which are introduced at the interface and are generated by cooling of the grown crystals.

Shrinkage rates of both faulted and unfaulted dislocation loops were measured. A variety of the shrinkage rates for the faulted loops can be attributed to large Burgers vector of the loops confirmed by their strong contrast in X-ray topographs. The stacking fault energy for the fault of which $\mathbf{R} = \frac{1}{6} \langle 20\bar{2}3 \rangle$ was determined to be 0.31mJ/m² by the use of shrinkage rates both for faulted- and unfaulted dislocation loops.

Symposium on Ice and Climate
Modelling, Evanston, Ill., U.S.A., June
26-July 1, 1983

Mechanical Properties of Greenland Dye-3 Deep Ice Cores

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Abstract : Uniaxial compression tests were carried out with specimens cut from several deep ice cores obtained at Dye-3, Greenland in 1980 and 81. The power law relation of $\dot{\epsilon} = A \sigma^n$ was obtained between the uniaxial strain rate $\dot{\epsilon}$ and the uniaxial stress σ . In a range of strain rate between 10^{-8} s^{-1} and 10^{-7} s^{-1} , number of power n was approximately 4, clearly larger than 3 which has been generally accepted from various experiments using artificial polycrystalline ice. Work-hardening effect was found for the samples from 1900m depth core which had smaller grain size than others. Recrystallization occurred when the temperature of a specimen was raised during the test and this ultimately caused the formation of the so-called diamond pattern of ice fabric.

Symposium on Ice and Climate
Modelling, Evanston, Ill., U.S.A., June
26-July 1, 1983

Computer Simulation of the Ice Sheet in the Shirase Basin, Antarctica

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Abstract : A three-dimensional numerical model is developed to simulate the time variation of the form of the ice sheet in the Shirase Basin, Antarctica. The model is composed of two-dimensional grids on which the mass flux of ice is computed so as to satisfy the equation of continuity. Local conditions of the flow of ice, particularly the effect of the depth profile of temperature, are considered. Adopting a simple method for calculating the mass flux developed by the same authors (Nagao et al. : Mem. Nat 1 Inst. Polar Res., Spec. Issue, 24, 192, 1982) procedures of numerical calculations are simplified. Areal grids of 50km distances covering the basin are used, paying special attentions to the boundary conditions at its margin and glacier tongue.

Results of the calculations show that a nearly stable form of the ice sheet could be obtained after approximately 10^4 years when started from 1000m ice thickness all over the basin. The obtained stable surface topography shows its sensitive dependence on the bedrock topography. There appeared a tendency that the bottom temperature of the downstream of the Glacier is higher than the melting point, which may conform to the suggested instability of the ice sheet near the central stream line of the Shirase Glacier (Mea, S. : J. Glaciol., 24, 53, 1979).

U. S. -Japan Seminar on Polymer Liquid
Crystals, June 14-20, 1983 (Kyoto Univer-
sity)

"Electron Spin Resonance of Spin-Labeled Poly (γ -benzyl L-glutamate) in Organic Solvents."

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To study the molecular dynamics of α -helical polypeptides in the solution, the electron spin resonance (ESR) spectra for poly (γ -benzyl L-glutamate) (PBLG) to which a nitroxide radical is labeled at the polymer end were measured in several helix forming organic solvents such as dimethylformamide, dichloromethane etc. The spectra in these solvents were characteristic of the nitroxide undergoing an axial motion, and the hyperfine splittings considerably depended on both the solvent and the concentration. It was found that the order parameter S increases with a decrease of the solvent polarity and an increase of the polymer concentration. These results were interpreted in terms of the molecular association of the rod-like polymers in the solution and the structure of the liquid crystal. A detailed discussion about the structure and the dynamical behaviors at the polymer end was also made by means of the computer simulation of the spectra.

International Conference on Martensitic Transformations (ICOMAT-82), Leuven (Belgium) August 8-12, 1982

The Bainite Formation Under Stress in Cu-Zn-Al Alloys

K. TAKEZAWA, H. IMAMURA, K. TANIZAKI and S. SATO

β_1 single crystals of Cu-29.8at% Zn-6.1at%Al alloy with different orientations were heated at temperatures higher than 420°K under tensile stress. Two variant crystals of bainite having different habit planes, for example $(12\ 11\ 2)$ and $(12\ \bar{2}\ \bar{1}\bar{1})$ for $[10\ \bar{6}\ \bar{5}]$ tensile direction, from that of stress-induced martensite, $(12\ \bar{2}\ 11)$, were observed after a certain period of incubation. The variant crystal having a maximum Schmid factor with respect to the shear on the habit plane was preferably formed in the martensite case. The close-packed plane of martensite is to be transformed from (110) plane of matrix crystal by the shear of $(1\ \bar{1}\ 2)$ $[1\ \bar{1}\ \bar{1}]$. On the other hand, it is found that the variant crystal of bainite having $(12\ \bar{2}\ \bar{1}\bar{1})$ habit plane has a larger Schmid factor with respect to the shear of $(1\bar{1}\bar{2})$ $[1\bar{1}1]$, which would also transform (110) matrix plane to a close-packed plane. The other variant having $(12\ 11\ 2)$ habit plane has an orientation which allows for a fcc structure to be formed by crossing with the first one in pair. The crossed region of these two bainite crystals was observed as a pink-colored area. Electron diffraction patterns indicate that the bainite has a disordered 9R structure. It is assumed that the intrinsic transformation shear producing the close-packed structure operates preferably in the bainite case, whereas the minimization of strain energy in the transformation is dominant in the martensite case. A mechanism of the shape change associated with the stress-induced bainitic transformation is also discussed.

International Conference on Martensitic Transformations (ICOMAT-82), Leuven (Belgium) August 8-12, 1982

Coherency of the Transformation Strain at the Grain Boundary and Fracture in Cu-Zn-Al Alloy

K. TAKEZAWA, T. IZUMI, H. CHIBA and S. SATO

Two types of Cu-Zn-Al bicrystal specimens having $e/a \approx 1.4$, one with the boundary parallel to and the other perpendicular to the tensile direction, were extended at room temperature.

The morphological change near the boundary was continuously observed by a microscope with a VTR during the extension. For a particular orientation with respect to the tensile direction, the same variant crystal of β'_1 martensite as that which would be stress-induced in an individual component crystal is produced with transformation strains which occasionally continue across the boundary. However, a different variant crystal from that in the individual component crystal is usually formed near the boundary depending on the orientation of the other grain. This variant has an orientation which allows for the strain fields to coincide with those produced by a variant which is transformed simultaneously in the other grain near the boundary. Since the possible number of shear systems producing the intimate β'_1 variants in both grains is limited, the fracture takes place

often at the grain boundary. However, when α'_1 martensites are formed at the boundary, as frequently observed, the fracture is suppressed to a great extent, because of increasing numbers of independent shear systems. Consequently, the degree of ease or difficulty in producing α'_1 martensites is closely associated with the character of fracture in Cu-Zn-Al alloy.

An extended work on fracture using polycrystalline specimens of different c/a values also supported the above results.

10 th International Congress on Electron
Microscopy, Hamburg, August 17-24,
1982

Image Characteristics of Defects in Internal Boundaries and Their Use for Identification

Kenzaburo MARUKAWA

Electron microscope images of defects lying in a boundary have been computed theoretically in various cases. Compared with the corresponding images in the bulk case, these images have such a feature that signs associated with defects are more clearly recognizable. In the case of dislocations, the sign of $g \cdot b$ or $g \cdot (b \times u)$ can be easily determined from the asymmetry or tint of images, where g is the reflection vector, b the Burgers vector, and u the dislocation line vector. These signs are used for the identification of the Burgers vector.

It is also shown that the nature of point defect clusters can be easily determined by examining their image feature. These results are illustrated by the observation of defects in twin boundaries in an iron alloy.

International Collaboration on Advanced
Neutron Sources (ICANS-VI), Algonne
National Laboratory, U. S. A., June 27
—July 2, 1982.

GROOVED COLD MODERATOR TESTS

K. INOUE, Y. KIYANAGI, H. IWASA, N. WATANABE,
S. IKEDA, J. M. CARPENTER and Y. ISHIKAWA

We performed some grooved cold moderator experiments for methane at 20 K by using the Hokkaido University linac to obtain information to be used in the planning of the KENS-I project. Cold neutron gains, spatial distribution of emitted beams and time distribution of the neutrons in the grooved cold moderator were measured. Furthermore, we assessed the effects of the grooved cold moderator on the performances of the spectrometers presently installed at the KENS-I cold source. We concluded that the grooved cold moderator benefited appreciably the performances of the spectrometers.

YAMADA CONFERENCE VI ON
NEUTRON SCATTERING OF CON-
DENSED MATTER, Hakone, Japan
September 1-4, 1982

NEUTRON QUASIELASTIC SCATTERING STUDIES OF MOLECULAR LIQUIDS AND POLYMERS BY PULSED COLD NEUTRON SOURCE

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We have developed a neutron quasielastic scattering spectrometer with conventional energy resolution. The spectrometer operates at the spallation cold neutron source in the KENS spallation source at the National Laboratory for High Energy Physics. High efficiency was achieved by adopting large analyser mirrors and the spallation cold neutron source. The spectrometer has demonstrated considerable usefulness in spectral profile analysis studies of molecular liquids and polymers.

5th "Tihany" Symposium on Radiation
Chemistry, Siófok, Hungary, 19-24
September 1982.

Stroboscopic picosecond pulse radiolysis of carbon tetrachloride

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and M. KATAYAMA

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Transient species in the radiolysis of CCl_4 has been investigated by stroboscopic picosecond pulse radiolysis using 11 ns pulses of 45 MeV electrons. The optical absorption spectrum showed two principal bands at 325 and 480 nm. Kinetic traces were obtained in the time region of 35-350 ps from the beginning of the pulse. The narrow band centered at 325 nm, which formed during each fine structure electron pulse and decayed after pulse $t_{1/2} = 6.5$ ns, was attributed to CCl_4^+ cation. The broad band centered at 480 nm and built up after the electron pulse was assigned to a charge transfer complex $[\text{CCl}_4^+ \cdot \text{Cl}^-]$. Since cations do not show any fast decay during the formation of charge transfer complexes, the presence of excited cations is strongly suggested. Effects of various cation scavengers on these bands were also studied, and the primary processes in CCl_4 were discussed together with the results of studies on some other halomethanes.

7th International Heat Transfer Conference, München, Fed. Rep. Germany, September 6-10, 1982

HEAT TRANSFER TO LIQUID SODIUM THROUGH CONCENTRIC AND ECCENTRIC ANNULAR CHANNELS

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Sapporo 060, Japan

An experimental study was performed on heat transfer of liquid sodium flowing through concentric and eccentric annular channels with an inner to outer radius of 0.576. Measurements were performed over a range of Reynolds number from 18,000 to 113,000 for a Prandtl number of 0.007 under the condition of the heated inner wall and the adiabatic outer wall. A sheathed thermocouple probe was set in the test section for measuring temperature distributions transverse to the sodium stream. In the case of the concentric condition, turbulent thermal diffusivities, a_t , and mean Nusselt numbers were compared with the results of some works previously reported. The eccentric condition measurements were made for the reduction in mean Nusselt number, and the results were compared with the predictions of Yu and Dwyer.

JAPAN-U. S. Seminar on Thorium Fuel Reactor, Nara, Japan, October 18-23, 1982

BASIC STUDIES FOR MOLTEN-SALT REACTOR ENGINEERING IN JAPAN

Ryoji ISHIGURO, Kenichiro SUGIYAMA and Hiroto SAKASHITA
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Sapporo 060, Japan

A research project of nuclear engineering for the molten-salt reactor which is conducted under the support of a Grant-In-Aid for Scientific Research of the Ministry of Education is reported. Some related studies which are carried out in Japan are also mentioned. Discussions on the following four subjects are summarized in this report : a) Vapor explosion when high temperature molten-salts are brought into direct contact with water. b) Measurements of exact thermophysical properties of molten-salt. c) Free convection heat transfer with uniform internal heat generation and a constant heating rate from the bottom. d) Stability of frozen salt film on the container surface.

1983 ASME-JSME THERMAL ENGINEERING CONFERENCE, Honolulu, U. S. A., March 20-24, 1983

AN ANALYTICAL STUDY OF HEAT TRANSFER TO LIQUID METALS FLOWING UNIFORMLY ACROSS A SINGLE CYLINDER

Kenichiro SUGIYAMA and Ryoji ISHIGURO
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Sapporo 060, Japan

A numerical analysis is performed to clarify the local heat transfer characteristics of liquid metal flowing uniformly across a single cylinder. An inviscid flow model taking into account the separation region which was ignored in the previous analyses is applied in the present study.

It is found that the present local Nusselt numbers have almost constant values behind the cylinder as observed in the experiments, while those of the previous analyses decrease monotonously. The present local Nusselt number is in fairly good agreement with the numerical solution of the viscous flow at a Reynolds number of 1300. Comparisons between the present numerical results and the experimental values previously reported by the authors are made at Reynolds numbers of 1200, 2200, 7400 and 17000 ($Pr=0.0066$). The agreement between them is considerably good except the rear region of the cylinder in the case of Reynolds number 17000.

The 5th Japan-USSR Seminar on Electrochemistry, Hokkaido University, Sapporo, Japan, September 16-18, 1982

Stability of Passivation Film on Titanium under Cathodic Reduction in Acidic and Neutral Solutions

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and Norio SATO
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Sapporo, 060, Japan

In deaerated sulphuric acid, the anodic oxide film on titanium dissolves slightly under open-circuit conditions and its rapid thinning occurs when cathodic current is applied. The cathodic reduction of the film takes place producing titanium ion in the solution ; $TiO_2 (ox) + 4H^+ (aq) + e \rightarrow Ti^{3+} (aq) + 2H_2O (aq)$.

In neutral phosphate solution no change is observed to occur with the film under open-circuit conditions, but application of cathodic current causes an appreciable change of the optical constant of the film. This optical change is reversibly restored under open circuit or anodic polarization conditions. This reversible change is attributed to the following oxidation-reduction of the film, $TiO_2(ox) + H^+(aq) + e \rightleftharpoons TiOOH(ox)$, which corresponds to the hydrogen adsorption and desorption in the film.

The 3rd Japan-USSR Seminar on Corrosion, "Improvement of Corrosion Stability of Structural Metallic Materials in Aggressive Media", Karpov Institute of Physical Chemistry, Moscow USSR, October 18-12, 1982

Role of Corrosion Precipitates in Passivation and Localized Corrosion of Metals

Norio SATO and Masao SAKASHITA

This paper deals with the ionic transport through corrosion precipitate membranes and its role in the propagation of corrosion of underlying metals. Corrosion precipitates are either anion- or cation-selective to ion permeation. Anion-selective precipitates accelerate the corrosion, since aggressive anions such as chloride ion migrate from the bulk solution into the precipitates, leading to acidification of solution adjoining the metal. Multivalent anions such as PO_4^{3-} and MoO_4^{2-} , if adsorbed, change the ion-selectivity of the precipitates from the anion-selective to the cation-selective, and prevent aggressive anions from entering the precipitates. The most favourable precipitate for corrosion protection is of bipolar type with an anion-selective layer on the metal side and a cation selective layer on the solution side.

The 3rd International Symposium "High Temperature Corrosion of Metals and Alloys" Fuji Kyoiku Kenshujō, Susono, Japan, November 17-20, 1982

Selective Surface Oxidation of Fe-30Ni Alloy

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Fe-30Ni alloy specimens were oxidized for 10min-240min at 433K-473K under a pure oxygen pressure of $1.33 \times 10^4 \text{Pa}$. The thickness of oxide films was measured by a multiple-angle-of-incidence ellipsometer. The kinetics of film growth was found to obey a parabolic rate law. The depth-profiling of oxidized surfaces, performed with simultaneous use of Auger electron spectroscopy (AES) and argon ion sputter-etching technique, reveals that iron component is selectively oxidized producing an iron depletion zone in the underlying alloy. The thickness of the iron depletion zone increases with increasing oxidation time or oxidation temperature.

During surface oxidation of the alloy, the transport rate of iron component in the film is almost equal to the interdiffusion rate in the underlying alloy, indicating the establishment of a steady state. The values of interdiffusion coefficient, D , of the underlying alloy estimated from the depth-composition profiles are more than ten orders of magnitude as large as the values extrapolated from the lattice-diffusion data of the corresponding alloy obtained at high temperature.

10th International Congress on Electron
Microscopy, held in Hamburg, West
Germany, August 17-24, 1982.

STUDY OF RADIATION-INDUCED MICRO-SEGREGATION IN ALLOYS BY MEANS OF HVEM AND EDX

T. TAKEYAMA, S. OHNUKI and H. TAKAHASHI

The micro-segregation near internal defect sinks, was investigated by means of HVEM, STEM and EDX. The irradiation was performed by HVEM at 650-1000kv with electron flux of $3 \times 10^{23} \text{e}/\text{m}^2 \cdot \text{s}$. The concentration profiles of solute atoms analyzed by EDX were measured by taking the ratio of $K\alpha$ or $L\alpha$ intensities for solute and solvent. Two types of segregation were observed near G. B. In Cu-2 at % Si alloy which has positive volume size factor, a double peak of Si concentration was observed near irradiated G. B. The concentration on the G. B. was lower than in the surrounding area. In contrast, a single peak of Ni concentration was observed on G. B. in Cu-2 at % Ni alloy which has negative size factor. Once the concentration decreased with distance from the G. B., and then slightly increased. In Ni-8 at% Si alloy which is a solid solution at equilibrium condition, which has negative size factor, precipitation of Ni_3Si were detected on the loops and G. B. By prolonged irradiation, Ti-modified 316 stainless steel of voids were produced at higher temperature. The segregation profiles were also determined near voids, Cr depleted strongly, but Ni, Si and Ti enriched at void surfaces. The segregation occurring at internal defect sinks in electron-irradiated binary and commercial alloys is classified into the enrichment and depletion types depending on the difference of the mode of interaction between radiation-induced point defects and solute atoms.

10th International Congress on Electron
Microscopy, held in Hamburg, West
Germany, August 17-24, 1982.

A STUDY OF EFFECT OF SOLUTE ATOMS ON DEFECT CLUSTER FORMATION IN Fe-Ti ALLOYS BY HIGH VOLTAGE ELECTRON MICROSCOPY

H. TAKAHASHI, S. OHNUKI and T. TAKEYAMA

The effect of the interaction between solute atoms and point defects on defect cluster formation such as dislocation loops and voids in an iron (Fe) and iron-based Ti alloys containing 0.1-0.2 at% Ti was studied by using high voltage electron microscope.

The specimens were irradiated at the temperature range between 525K and 725K. The loops formed at the early stage of irradiation continued to grow and subsequently new loops were separately nucleated.

These small loops often shrunk after gliding of the grown interstitial loops during further irradiation and disappeared, which seem to be vacancy type clusters. In the Fe-Ti alloys the higher dose was needed for the occurrence of voids, and no voids could be nucleated with increasing of Ti concentration.

The activation energy for vacancy migration obtained by the measurements of the growth speed of interstitial loop were 0.58, 0.82 and 1.21 eV for the Fe-0.1 at% Ti, Fe- 0.2 at% Ti and the Fe, respectively.

If the impurities are trapped by the Ti, the value would approach to the actual vacancy migration energy and vacancy clusters would be preferentially formed. In contrast, void formation will be retarded in the Fe-Ti alloys.

Third JIM International Symposium on
High Temperature Corrosion of Metals
and Alloys, November 17-20, 1982, Lake
Yamanaka, Japan

HIGH TEMPERATURE SULFIDATION OF IRON AND ITS ALLOYS IN LOW SULFUR PRESSURES

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High temperature sulfidation of iron and its alloys are discussed with results obtained in low sulfur pressures, and compared with the corrosion kinetics and scale morphologies of commercial alloys.

Using Wagner's parabolic rate theory, the corrosion behavior of pure iron is compared with experiments in low sulfur pressures. Based on a similar kinetic theory developed recently for binary alloys with scales following parabolic growth and the difference between the dissociation pressures of ferrous sulfide and other sulfides of the alloying elements, the corrosion behavior is discussed and compared with the expected behavior of the alloy corrosion kinetics.

Commercial alloys such as SUS 316L and SKD 11 were sulfidized in low sulfur pressures and the similar or different results observed with these alloys is evaluated.

Third JIM International Symposium on
High Temperature Corrosion of Metals
and Alloys, November 17-20, 1982, Lake
Yamanaka, Japan

GRAIN BOUNDARY SULFIDATION OF Fe-Cr ALLOYS IN H₂S-H₂ ATMOSPHERES AT HIGH-TEMPERATURES

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Hokkaido University, Sapporo 060, Japan

The sulfidation properties of Fe-Cr alloys were investigated at 1073 and 1173 K in H₂S-H₂ gas mixtures with low sulfur pressures by TGM, X-RAY DIFFRACTION, EPMA, and SEM-EDAX. Significant grain boundary sulfidation emerged from below the surface scale formed on Fe-medium Cr alloys at 1073K under sulfur partial pressures below the dissociation pressure of ferrous sulfide.

At a temperature of 1173K, a surface scale and copious internal and grain boundary sulfidation was formed, also for the Fe-high Cr alloys at 1073K. Both the sulfides formed on the alloy surface and at the grain boundary consisted of (Cr,Fe)S_x phase containing small amounts of iron.

It appears that for the initiation of the grain boundary sulfidation there are at least two features : the diffusion flux of the sulfide is much greater than that of alloys, and a selective sulfidation of chromium

Further, sulfidation propagates rapidly by reaction with sulfur and carbides precipitated along the grain boundaries.

Third JIM International Symposium on
High Temperature Corrosion of Metals
and Alloys, November 17-20, 1982, Lake
Yamanaka, Japan

SULFIDE FORMATION AND CONTROL IN HIGH TEMPERATURE CORROSION OF IRON AND ITS ALLOYS IN SO₂ ATMOSPHERES

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The scaling kinetics and scale morphology of iron at high temperatures in argon-diluted sulfur dioxide were investigated. The sulfide formation is caused by a local rise in the activity of the sulfur adsorbed on the scale surface, as a result of a rapid consumption of oxygen. It is suggested that the formation of a scale having the lowest possible cation diffusivity is necessary to control the sulfide formation.

24th Rocky Mountain Conference,
Denver, Colorado, USA, Aug. 1-5, 1982

Roles of Catalyst for Coal Liquefaction Reaction by Means of High Temperature ESR and NMR

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High temperature ESR and NMR were used in order to obtain in-situ information on the behavior of catalysts during coal pyrolysis. The free radical population in coal was observed in the presence of catalyst at the pyrolysis temperature. The spin concentration increased drastically for the coal/ZnCl₂ system while that of coal alone showed a small increase at 420°C. This increase in the spin concentration may result from the formation of complexes between zinc chloride and polyaromatic compounds and/or the cleavage of some heterolinkage. Marked broadening of the line width of ¹H NMR spectrum occurred correspondingly above 430°C in the presence of zinc chloride. Activity of MoO₃ over the various metal oxides was discussed relating to the concentration of Mo⁵⁺.

32nd Canadian Chemical Engineering
Conference, Vancouver, B. C., Canada,
Oct. 3-6, 1982

Liquid Fluidization of Dissimilar Solid Particles

Hiroshi MORITOMI and Tadatoshi CHIBA
Coal Research Institute, Hokkaido University, Sapporo 060, Japan

Solid segregation in liquid fluidized beds containing binary mixtures of spherical particles of different density and size was studied over a range of liquid velocities, bulk bed compositions and particle properties. When the bulk volume fraction of the lighter particles is high and the liquid velocity is relatively low, the bed forms two layers, i. e. the upper layer consisting almost entirely of the lighter and the lower mixed layer consisting of both components. A completely mixed bed is obtained at a certain velocity and then a further increase of the velocity causes "layer inversion". The layer inversion occurs for a given particle mixture when the liquid velocity passes through a value at which the volume fraction of the lighter in the lower layer becomes equal to the bulk bed composition or for a given velocity, when the bulk bed composition becomes equal to the fraction of the lighter component which exists in the lower layer.

International Symposium on Carbon,
Toyohashi, Japan, Nov. 1-4, 1982

Proton Relaxation Studies of Cokes from Petroleum Residues

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Proton relaxation studies were done in order to characterize cokes formed in carbonization and co-carbonization from petroleum residues.

A good correlation was found between slow relaxation component of spin-spin relaxation time (T_2) of cokes and the size of optical texture of resultant cokes.

Proton spin-lattice relaxation time (T_1) was proved to be a useful parameter to characterize cokes from co-carbonization of petroleum residues.

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Transferable Hydrogen in Coal and Pitch during Mesophase Formation

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In order to obtain detailed informations of hydrogen transfer which plays an important role during mesophase formation, the effects of carbonization condition, that is, catalysts, oxidation, and rate of heating upon transferable hydrogen of coals and pitches (including model compounds) were investigated by means of $^1\text{H-NMR}$ and high temp. ESR.

Pan Pacific Synfuels Conference,
Tokyo, Japan, Nov. 17-19, 1982

Dehydrogenation of Coal over Catalysts : An Evaluation of Coal and Catalysts for Liquefaction

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The amount of hydrogen evolved from a coal during pyrolysis at atmospheric pressure in the presence of coal hydroliquefaction catalysts can be used to compare the activity of different catalysts for the high pressure hydrogenation of coals. It has been found that a close correlation exists between the hydrogen evolution from coal-catalyst systems for various catalysts under atmospheric pressure and the catalytic activities of hydroliquefaction under high pressure. This technique has also now been used to compare the variation in the effectiveness of $\text{MoO}_3\text{-TiO}_2$, and $\text{Fe}_2\text{O}_3\text{-SO}_4^{2-}$, as hydroliquefaction catalysts with change in coal rank. It is indicated that coals with carbon contents near 82% are the most amenable to hydroliquefaction at high pressure. The measurement of hydrogen evolution is a rapid and convenient method of evaluating catalytic activities for coal liquefaction.

The 4th International Conference on
Fluidization, Kashikojima, Japan, 29th
May-3rd June, 1983

Defluidization of Large Particles due to Segregation

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Segregation during fluidization of dissimilar materials occurs near the bottom of the bed when the minimum fluidization velocity of the mixture is greater than the superficial gas velocity. Such

defluidization is disastrous in coal combustion and gasification and in granulation.

Mechanisms of defluidization and their relationship to the superficial gas velocity and the properties of the solid mixture have been examined, mainly with binary mixtures of equal density. By adding the smaller particles into defluidized layer the change in the defluidized layer height has been observed in a column with a perforated plate or standpipe distributor.

The experiments showed that a bed of smaller particles can mix and fluidize a certain amount of the larger defluidized ones even though the superficial gas velocity is less than the U_{mf} of the larger. A mathematical model for estimating the defluidized layer height has been developed which compared well with the observed data.

First International Symposium on Mol-
ten Salt Chemistry and Technology.
April 20-22, 1983 Kyoto, Japan

NMR Study of Thallium (I) Ions in Molten Binary Mixtures of Nitrates and Chlorides

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The chemical shifts of ^{205}Tl NMR in molten binary mixtures of nitrates and those of chlorides have been measured as a function of composition and temperature. The shifts increase in the diamagnetic direction with decreasing the size of foreign cations and increase in the paramagnetic direction with increasing temperature. These results are interpreted by changes in the "overlap" of orbitals of the Tl^+ ion and the anion, which depend upon composition and temperature.