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Eighth World Conference on
Earthquake Engineering, July
21-28, 1984, San Francisco, U. S. A.

Dynamic Behavior of Concrete Stave Silos

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Concrete stave silos, which have been utilized as industrial and agricultural storage facilities, are assembled from more than a thousand precast concrete blocks and held together by exterior adjustable steel hoops.

A series of shaking table tests was performed using a 1/6-scale silo model filled with and without brown rice or wet sawdust to clarify the dynamic behavior of stave silos with such discontinuous walls and the effects of stored materials. The experimental results indicated that the stored materials lowered the resonant frequencies of the silo model, and that the response ratio of the model filled with brown rice decreased gradually as the peak table acceleration was increased.

A static loading test, earthquake simulation test and nonlinear response analysis were also carried out concerning another small-scale silo wall model. It was found that the discontinuous wall structures of stave silos showed remarkable hysteretic restoring force characteristics in which the skeleton curve was a soft-spring type.

7th International Symposium on
Ice Problems Hamburg, F. R.
Germany, August 27-31, 1984

Estimation of Internal Pressure due to a Growth of Ice Thickness of a Caisson

Hiroshi SAEKI

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Recently, caisson type structures have been used for offshore structures, such as artificial islands and oil drilling rigs, in very cold regions. As the liquid filling the caisson freezes, the internal pressure rises. This pressure rise, due to an increase of volume caused by ice growth, can be estimated. In this paper the theoretical analyses and experiments required to develop a method to estimate this internal pressure.

The following conclusions were obtained from this investigation ;

- 1) With the use of saline water as the filling material in the test tank, the internal pressure increased only

slightly with the growth of ice thickness and the surface of the ice sheet became wet due to the permeability of saline-water ice. Radial cracks did not occur on the surface of the ice sheet.

2) If an air gap exists between the caisson cover and filling material, the rise in internal pressure due to the growth of ice thickness would be lessened because of the permeability of sea ice to water.

3) In the case of fresh water as the filling material in the test tank, internal pressure increased markedly with the growth of ice thickness, also radial cracks occurred on the surface of the ice. The occurrence of these cracks did not have an effect on internal pressure.

Roc. of 7th International Symposium on Ice Problems Hamburg, F. R. Germany, August 27-31, 1984

Determination of Compressive Strength of Sea Ice by Using an Ultrasonic Pulse

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It would be very useful for the advancement of ice engineering technology if there existed a quick, inexpensive way to accurately determine the strength of sea ice. The strength of sea ice is determined by its composition and temperature.

This paper reports on experiments which determined both the composition and the uniaxial compressive strength of sea ice using an ultrasonic pulse. We also aim to describe the testing methods by means of the ultrasonic pulse for sea ice.

19th International Conference on
Coastal Engineering Houston,
Texas, U. S. A. September 3-7,
1984

Effects of Opposing Current on Wave Transformation on a Sloping Sea Bed

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Hiroshi SAEKI

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The transformation and the breaking of waves affected by opposing currents on a sloping sea bed were discussed. It was found that the characteristics of wave transformation before the breaking point can be determined by the deep water wave steepness and the dimensionless unit width discharge. Regarding wave decay due to breaking, the characteristics of the change in the wave height depended only on the slope of the sea bed.

Fourth order solutions of Stokes wave on a uniform current were calculated based on the first and second definition of the wave celerity, respectively. The theoretical solutions for wave transformation by the energy flux method were presented. Comparisons between the theoretical solutions and experimental results gave the criteria of the applicability of our solutions corresponding to the dimensionless unit width discharge. In the calculation of energy flux, it was pointed out that the change in the mean level of the free surface should be taken into account. It was clarified that the change in the mean level of the free surface can be evaluated by Bernoulli's equation, and the energy flux in which the change in the mean level of the free surface was taken into account was proposed.

The criteria of breaking corresponding to the dimensionless unit width discharge were clarified, experimentally.

International Symposium on
Snow and Ice Processes on the
Earth's Surface Sapporo, Japan,
2-7 September, 1984

Experimental Study on Direct Shear Strength of Sea Ice

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Niu en ZONG

Ministry of Communication, The People's Republic of China

Naoki NAKAZAWA

Pacific Consultants Co., Ltd., Japan

Investigations were made on the establishment of testing methods for direct shear strength as well as on the mechanical properties of shear strength of sea ice. Experiments were carried out systematically for

four years by using cylindrical specimens which were made from real sea ice on the Okhotsk Sea coast.

First, the authors clarified the effects of stress rate, strain rate, normal stress and size of specimen on the direct shear strength. Based on the results mentioned above, the most suitable testing methods for direct shear strength were proposed.

Second, the difference in direct shear strength due to load directions (parallel and normal to growth direction of sea ice), the relation among ice temperature, normal stress and direct shear strength and the relation between direct shear strength and uniaxial compressive strength were clarified.

Finally, the application limit of Coulomb's formula and the relation among cohesion, angle of internal friction and ice temperature were decided for sea ice.

17th Annual Offshore Technology
Conference Houston, U. S. A.
7-9 May, 1985

An Experimental Study on Ice-Structure Interaction

Masahiko TUCHIYA, Shunji KANIE and Akira YOSHIDA

Taisei Corporation

Hiroshi SAEKI

Hokkaido University

To date, despite a number of studies regarding dynamic ice-structure interaction problems, it seems that a practical and effective solution for the problems has not yet been established. It is considered that, at present, more experimental data is required needed for the establishment of a solution for the problem.

In our experimental study, intensive model ice loading tests were conducted at the Saroma Lagoon in Hokkaido, Japan. Three model pile structures having different natural frequencies were laterally loaded with ice sheets at wide-ranging strain rates of ice. Significant relationships were observed between the strain rate of ice and both the ice force and the response of the model structure.

The test results were analyzed using a rather simple analysis model which took the strain rate dependent ice force into consideration. The analysis model reasonably predicted the dynamic response of the model structure due to ice loading.

10th Congress of the International Association for Accident and Traffic Medicine, May 27th-31th, 1985 Tokyo, Japan

Traffic Accidents in Winter related to Tires

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In Hokkaido, road surfaces are covered with snow and ice in winter. The skidding resistance on snow and ice covered road surface is much less than that on dry or wet road surface, so that traffic accidents are related to skidding in many cases. But, there are only a few reports showing such a relation. It is necessary to investigate skidding accidents on snow and ice covered road surface. In this report, we discuss the relation between traffic accidents in winter and the skidding resistance.

The findings from the analysis are as follows : 1) Slipperiness of road surface such as ice and snow covered has a strong intimate relation with skidding accidents in winter. 2) It is important to check tires frequently carry out thorough maintenance of tires. 3) It is necessary that the driving technique be improved to cope with slick road surfaces.

10th Congress of the International Association for Accident and Traffic Medicine, Tokyo, Japan, May 27th-31th, 1985

The Skidding Properties of Studded Tires on a Wet and a Dry Surface Road

T. KAKU, M. TODA, Y. ONODERA, T. NAKATSUJI
Department of Civil Engineering, Faculty of Engineering

Major European countries, considering the negative effects accompanied by studded tires, have restricted the usage of them. In Japan, they have commonly been used to improve frictional capability on a icy road since the early 1970's. Complex conditions such as the fact that Japan has a considerable snowfall precipitation compared with European countries require more extensive investigations before considering the regulation thereof.

Experimental field tests were carried out to obtain frictional properties of studded tires on wet and dry surface roads. Contents of field tests performed here are

- (1) stopping distance test
- (2) locked-wheel test
- (3) cornering slip test

On a wet surface, studded tires have longer stopping distance than normal tires. Especially at speed of 60km/h, the stopping distance of them are similar to that of smooth tires.

Studded tires showed considerably lower frictional capabilities than normal tires in both locked-wheel

tests and cornering slip tests.

10th congress of the International Association for Accident and Traffic Medicine, Tokyo, Japan, 27th-31st May, 1985

Some Considerations on Traffic Accidents at Intersections

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Intersection accidents that occurred in Hokkaido were studied. Three arterial highways were selected and accident data were collected from collision diagrams for two years.

Some aspects of intersection accidents were recognized and accident rates were calculated in certain ways, but these rates should be investigated more precisely because of the unreliability of the used data.

U. S. -N. Z.- JAPAN Seminar,
May 29-30, 1985, Tokyo, Japan

Behavior of Three-Dimensional R/C Beam-Column Subassemblages with Slabs

T. SHIBATA and O. JOH

Faculty of Engineering, Hokkaido University, Japan

The purpose of the test was to study the shear resistance of interior beam-column joints in two-way reinforced concrete frames in comparison with the strength equations proposed in Japan and the requirements of ACI Code and New Zealand Standard. Four half-scale three-dimensional interior beam-column subassemblages with slab were used as the specimens on which lateral cyclic forces were applied in two directions perpendicular to each other by changing the direction alternately at every cycle. The variations in the specimens were made with the lateral reinforcement in the joints and with the beam-column depth ratio.

It is concluded from the test results that shear cracking stress in the beam-column joint could be assessed by the principal stress concept assuming the normalized interaction curve of an arc of circle for the biaxial shear, though some modification should be necessary for the oblong joints. The enhancement of joint shear strength by the transversed beams should not be expected either for cracking stress or for the ultimate strength.

U. S.- N. Z.- JAPAN Seminar, 30
July-1 August 1984, Monterey,
California, U. S. A.

Behavior of R/C Interior Beam-Column Joints with Various Details under Cyclic Lateral Loads

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Faculty of Engineering, Hokkaido University, Japan

The constitution of beam-column joints in reinforced concrete building frames has a large variance in detailing. The tests reported in this paper were carried out to investigate the mechanical behavior of interior beam-column joints in one-way frames mainly with regard to the geometrical configurations of beams and columns such as, for example, the ratio of beam width and column width or eccentricity between beam and column longitudinal axes. Six beam-column subassemblages in a plane frame of about a half scale without slabs and transverse beams were subjected to cyclically reversed lateral forces and a constant column-axial load.

Even if the ultimate strength of the frame is regulated by the flexural strength of beams, shear failure in beam-column joints may result eventually from severely repeated reversals of lateral loading after the occurrence of yielding in the beams. When beams and columns are connected eccentrically to each other, the influence of eccentricity should primarily appear in the ductility of the frame rather than in the strength.

The Eighth World Conference on
Earthquake Engineering, July 21-
28, 1984, San Francisco, Califor-
nia, U. S. A.

Shear Failure of Reinforced Concrete Columns Due to Biaxial Lateral Forces

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This investigation deals with the shear strength and the deformability of reinforced concrete columns under biaxial lateral forces. As the factors of influence on these properties, the variations in the column axial load ratio, in the shape of column cross-section and in the angle of lateral force for a principal axis of the column section were considered for thirteen specimens of a half scale. Nine specimens with a square cross-section and four specimens with a rectangular cross-section were subjected to lateral displacement reversals with increasing amplitude in the direction perpendicular or oblique to an axis of the cross section.

It is concluded from the test results that the normalized interaction curves relating to the shear components in the directions of both the principal axes of cross-section may converge on a circle at ultimate shear strength as well as at initial shear cracking. It was clarified by using a statistic method that the equations to estimate the initial shear cracking stress and the ultimate shear stress, proposed by one of

authors previously, was in better agreement than the equations used in the design code of American Concrete Institute.

Third international conference
on the durability of building
materials and components,
ESPOO, FINLAND, 1984

**Durability of Concrete in Cold Districts, Especially
on the Effect of Curing in Early Ages and of Freezing
Conditions**

Yoshiro KOH, Masayuki TABATA and Eiji KAMADA
Architectural Department, Faculty of Engineering, Hokkaido University

Several experimental studies were carried out to clarify the effect of various factors, such as drying in early ages, concrete temperature directly after mixing, and freezing and thawing conditions, on the deterioration of concrete. A special freezing and thawing test was applied for this experiments by introducing several kinds of models of temperature variations during winter in several cities combined with short drying period in summer considering previous test results.

Third international conference on
the durability of building materi-
als and components, ESPOO,
FINLAND, 1984

Frost Deterioration of Cellular Concrete

Eiji KAMADA, Yoshiro KOH, and Masayuki TABATA
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In the case of cellular concrete, generally the surface of specimens scale off slowly by freezing and thawing tests. However, destruction around the perimeter of outer frozen zone was observed. A new test method for cellular concrete was proposed taking into account Taber-Collins' theory.

Third international conference on
the durability of building materi-
als and components ESPOO,
FINLAND, 1984

Influence of External Weathering Factors on the Frost Damage of Concrete and the Calculation of the Degree of Frost Damage of Concrete

Toshio HASEGAWA and Yoshiro KOH
Architectural Department, Faculty of Engineering, Hokkaido University

External factors due to weathering action are one of the main factors which cause the frost damage of concrete, and causes the acceleration of frost damage of concrete. Several experiments were performed regarding the temperature conditions and the degree of saturation of concrete related to the weathering action. On the basis of the experimental results related to the external factors, the value of risk of frost damage (V_f) was calculated and defined by using weather data in a typical winter in Japan. The degree of risk of frost damage (D_f) was determined by classifying the value of V_f into 6 grades.

Proceedings of the IV Internatio-
nal Drying Symposium, July 9-12,
1984, Kyoto, Japan

On the Vapour Transfer Rate in Porous Media under a Temperature Gradient

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This paper deals with the problem of the vapour transfer rate in porous media such as a granular bed under a temperature gradient. The microscopic temperature and temperature gradient distribution in porous media were calculated by the numerical relaxation method for two regular packed models. These results revealed that the value of which is defined as the ratio of the temperature gradient in air filled void to that of the overall porous media was 1.5-2.0 for the usual bed.

The mass transfer experiments under a constant temperature gradient were achieved. In this experiment, granular beds of glass sphere particles containing sublimate substance, for example, naphthalene were used as the sample and the quantities of this substance transferred in a vapour state in a designated period were measured by quantitative analysis of gas chromatography. As a result, it was found that the experimentally obtained vapour transfer rate was generally larger than that of the calculated value by a past simple mass transfer model and these gave support to our theory.

The 7th International Acoustic
Emission Symposium Zao, Japan
October 23-26, 1984

Application of Acoustic Emission Methods to Investigation of the Cold Shortness of Steel Material in Mine

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Masayuki TAKEUCHI
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The problem of cold shortness of mine tub couplers in winter arose at a coal mine in Hokkaido, Japan. Regarding this problem, the acoustic emission method was applied to the tensile test of the stud link and the fracture toughness test of the same material at a low temperature. The link which failed due to the cold shortness broke at the source position of acoustic emission which generated before failure. The acoustic emission increased as the temperature of environment declined and the transition from ductile to brittle was estimated as the result. The fracture toughness and COD were evaluated by the sudden increase of the acoustic emission as fatigue crack extended.

The 7th International Acoustic
Emission Symposium Zao, Japan
October 23-26, 1984

Evaluation of Working Face Stability in Coal Mine with the Portable Acoustic Emission Counter

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Sapporo, Japan
Koichi NISHIMURA and Satoshi FUKAI
The Coal Mining Research Center Tokyo, Japan

A portable acoustic emission counter has been developed. This original counter is intrinsically safe and its weight is 0.35 kg with the dimensions of 200×100×40mm. In the device presented here acoustic emission events are indicated as the total numbers of acoustic emission ring down counts in digits on the front panel with LED display.

The acoustic emission activity occurred in the coal seam during mining or boring was observed with this equipment. The former presents the results to indicate the possibility of detection of the impending outburst hazard and the latter provides useful information for locating the range of the high stressed zone or the poor strength coal area in the coal seam.

The 7th International Acoustic
Emission Symposium Zao, Japan
October 23-26, 1984

Acoustic Emission Monitoring of Rock Burst in Excavation of the Kan'etsu Tunnel

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In the excavation of the Kan'etsu Tunnel, the rock bursts occurred many times expectedly and became a serious problem in safety. In order to cope with this problem, the fracturing activity was monitored over the rock burst-prone region by acoustic emission methods.

As the results of this monitoring, the rock fractures such as small rock bursts, spallings and rock noises occurred locally at the tunneling face with remarkable increase in acoustic emission activity. The exponent m-value of the amplitude-frequency distribution of acoustic emissions decreased with increase in the fracturing activities. These observational results indicated the possibility of an early detection of the state of rock burst hazard and provided a new interpretation for the processes of rock burst occurrence.

Third international mine ventila-
tion congress, Harrogate, Eng-
land, June 13-19, 1984

Macroscopic Characteristics of a Complicated mine Ventilation Network

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Faculty of Engineering, Hokkaido University, Sapporo, Japan

To gain the very important understanding of the macroscopic characteristics of a complicated mine ventilation network used for ventilation control a large number of simulations were performed with varying aerodynamic resistance, R , of the branches in mine ventilation networks.

The principal results of the study are reported.

(1) An H-Q (pressure loss-quantity) diagram enables energy consumption, RQ^3 , in a network branch to be evaluated.

(2) Maintenance of the same condition in a branch with a large energy consumption is important to the stable working of the main fans : when the aerodynamic resistance of a branch k in a network is increased the airflow rate at the main fans decreases, and the difference in airflow rate at the main fans between the first and second conditions is proportional to the energy consumption in branch k under the first condition.

(3) The relationship between aerodynamic resistance, R_q , of a branch q and airflow rate, Q_p , through branch p can be represented by the empirical formula

$$Q_p = A(R_q/B + R_0/B)^{-n} + Q_0$$

where A , R_0 and Q_0 are the constant coefficients with dimensions m^3/s , $kg \cdot s^2/m^8$ and m^3/s , respectively,

n is a dimensionless coefficient and B is given a value of 1μ (μ , murg) to render the values in the brackets dimensionless.

4th International Bureau of Mining Thermophysics, Burton-on-Trent, England, May 13-17, 1985

The Effects of Air Flow Path and Aerodynamic Resistance on Temperature Distribution in the Underground Roadway Network

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Faculty of Eng.

Hokkaido University, Sapporo, Japan
Shiruu LIN

Engineer, Fushun Institute of Coal Science, Fushun, China

Kiyoshi HIGUCHI
Dr. and Professor, Faculty of Eng.
Hokkaido University, Sapporo, Japan

An algorithm for simulation of underground climate has been explained, and some factors relating to air temperature at the headentry T-junction have been discussed.

Main results in this study are as follows,

1) When geothermal gradient can be considered a constant, relationship between pressure potential and air temperature at nodes in a network is approximately linear and its inclination changes at a point of the maximum air temperature.

2) When a new airway is excavated for increase of airflow rate in coal face, air temperature at the headentry T-junction is not always lower than one at the original network.

3) When aerodynamic resistance of airway is become less by blowed mortar on the wall of the airway, air temperature at the headentry T-junction decreases and air temperature at this node has a linear relation to airflow rate passing through the coal face.

The First SINO-JAPANESE
Physical Metallurgy Symposium
From 8 to 11 Nov. 1984, Peking

The Strengths of Directionally Solidified γ' (Ni_3Al)— δ (Ni_3Nb) Eutectic Alloy

Kastuya WATANABE and Masaaki FUKUCHI
Department of Metallurgy

The directionally solidified eutectic alloy of γ' — δ has a high application potential as a surpassing heat resistive material. In this study the deformation behavior of both the entire specimen and the individual phases were examined.

The δ phase, which was previously considered brittle, showed a tolerable plasticity under certain stress orientations.

The effect of Nb dissolution on the positive temperature dependence of strength of the γ' was studied, and it became clear that the stress-temperature relation does not alter substantially by Nb dissolution.

In the directionally solidified eutectic alloy, two kinds of lamella structure were observed. The cause of brittleness of the eutectic alloy at lower temperatures can be ascribed to the deficiency in the number of slip systems to operate in the γ' phase. Electron microscopic observations showed at high temperatures that the deformation systems in the both constituent phases work in cooperation with each other and the stress is highly concentrated at the lamellae boundary.

Society of Automotive Engineers
International Congress & Exposition,
Detroit, Michigan, February
25-March 1, 1985

Description and Analysis of Diesel Engine Rate of Combustion and Performance Using Wiebe's Functions

Noboru MIYAMOTO, Takemi CHIKAHISA, Tadashi MURAYAMA
and Robert SAWYER

Two laboratory engines, one direct injection and one indirect injection, were operated for a range of speeds, loads, injection timings, fuels, and steady and transient conditions.

Rate of combustion data were derived and analyzed using a double Wiebe's function approximation. It is shown that three of the six function parameters are constant for a wide range of conditions and that the other three can be expressed as linear functions of the amount of fuel injected during ignition lag.

Engine noise, smoke, and thermal efficiency correlate with the parameters describing the amount of premixed combustion and diffusive combustion duration.

These characteristics may be optimized by reducing the quantity of premixed combustion while maintaining the duration of diffusive combustion to less than 60 °CA.

Society of Automotive Engineers
International Congress & Exposition,
Detroit, Michigan, February
25-March 1, 1985

The Effects of Flash Boiling Fuel Injection on Spray Characteristics, Combustion, and Engine Performance in DI and IDI Diesel Engines

Nobuyuki YAMAZAKI, Noboru MIYAMOTO and Tadashi MURAYAMA

This paper deals with the effects of flash boiling injection of various kinds of fuels on spray characteristics, combustion, and engine performance in DI and IDI diesel engines.

It is known that spray characteristics change dramatically at the boiling point of fuel. When the fuel temperature increases above the boiling point, the droplet size decreases apparently and the spray spreads more widely. At higher fuel temperatures, above the boiling point, the apparent effects are a lower smoke density and improved thermal efficiency at higher loads, resulting from the shorter combustion duration; it is thus possible to obtain a markedly improved engine performance in engines with a low air-utilization chamber.

Remarkable changes in heat release with the increase in fuel temperature are; an increase in premixed combustion quantity and shortening of the combustion duration. The changes in smoke emission and thermal efficiency for different engine types are also considered in this paper.

Society of Automotive Engineers
International Off-Highway &
Powerplant Congress & Exposition,
Milwaukee, Wisconsin, Sep-
tember 10-13, 1984

Low Carbon Flower Buildup, Low Smoke, and Efficient Diesel Operation with Vegetable Oils by Conversion to Mono-Esters and Blending with Diesel Oil or Alcohols

Tadashi MURAYAMA, Young-TAIG OH, Noboru MIYAMOTO,
Takemi CHIKAHISA, Nobukazu TAKAGI and Koichiro ITOW

The purpose of this investigation is to evaluate the feasibility of rapeseed oil and palm oil for a diesel fuel substitution in a naturally aspirated D. I. diesel engine, and also to find means to reduce the carbon deposit buildup in vegetable oil combustion.

In the experiments, the engine performance, exhaust gas emissions, and carbon deposits were measured for a number of fuels: rapeseed oil, palm oil, methylester of rapeseed oil, and these fuels blended with ethanol or diesel fuel with different fuel temperatures.

It was found that both of the vegetable oil fuels generated an acceptable engine performance and exhaust gas emission levels for short term operation, but they caused carbon deposit buildups and sticking

of piston rings after an extended operation. Practical solutions to overcome the problems were : increasing the fuel temperature to over 200°C, blending 25 vol% diesel fuel in the vegetable oil, blending 20 vol% ethanol in the fuel, or converting the vegetable oils into metylesters.

20th Symposium (International)
on Combustion, Ann Arbor, Mich-
igan, August 12-17, 1984

The Effects of NO₂ on Catalytic Oxidation of Unburned Species from a Methanol Fueled Spark Ignition Engine

Kenichi ITO and Osamu FUJITA
Department of Mechanical Engineering,
Hokkaido University

Catalytic oxidation processes of unburned species in methanol combustion products are examined with an emphasis upon the role of NO₂. Two kinds of experiments were carried out. One was on the catalytic oxidation of the engine exhaust gases and the other was on prepared gases which simulated the engine exhaust gases. In an automotive catalyst bed, unburned methanol, formaldehyde, NO and NO₂ concentrations were determined at air-fuel equivalence ratios and gas temperatures, ranging 0.8-1.5 and 400-540 K, respectively.

When the exhaust gas entered the catalyst bed, NO₂ decreased and NO increased rapidly. Methanol was also oxidized and its oxidation ratio had a maximum value at an equivalence ratio of 1.2 and was related to the NO₂ emission characteristics. The conversion of NO₂ to NO was improved by the presence of methanol and the oxidation of methanol was promoted by NO₂. Because of the higher NO₂ concentration in the methanol engine exhaust gas, the effect of NO₂ in the catalytic oxidation appeared more strongly than in the exhaust gases from engine using other fuels.

China-Japan Vibration Joint Con-
ference 1984, Shanghai, China,
Oct. 23-26, 1984

Free Vibration of a Point-Supported Spherical Shell

Toshihiro IRIE, Gen YAMADA and Yasushi MURAMOTO
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Hokkaido Univeristy, Sapporo, 060 Japan

An analysis is presented for the free vibration of an elastically or a rigidly point-supported spherical shell. For this purpose, the deflection displacements of the shell are written in a series of the products of the associated Legendre functions and the trigonometric functions. The maximum kinetic and strain

energies of the shell are evaluated analytically, and the frequency equation is derived by the conditions for a stationary value of the Lagrange functional. For a rigidly point-supported shell, the Lagrange multiplier method is conveniently employed. The method is applied to a closed spherical shell supported at equispaced four points located along a parallel of latitude; the natural frequencies (the eigenvalues of vibration) and the mode shapes are calculated numerically, and the effects of the point supports on the vibration are studied.

XVIth International Congress of
Theoretical and Applied Mechan-
ics, Lyngby, DENMARK, August
19-25, 1984

Plastic Flow Rule for Cyclic Loading

H. ISHIKAWA

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H. LIPPMANN

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Technische Universität München

The stress analysis of structures subjected to cyclic loading requires stress-strain relations sufficiently simple to be usable for computer programming and yet adequate to describe the essential features of the plastic behaviour of the material. One such form was proposed by Drucker and Palgen. However, the procedure of the introduction of their model did not show clearly enough that it follows the theory for cyclic plasticity.

In this paper the constitutive equation for cyclic plasticity in consideration of the motion of the center of the loading surface is proposed. Using a modified plastic work, the dependency of the loading history of material is taken into account. The Ramberg-Osgood's law which is applied to each stress strain loop from the current center of the loading surface plays an important role in this theory. The theory is verified from the point of view of several kinds of experiments on type 304 austenitic stainless steel.

International Conference Series
on Advances in NUMERICAL
METHODS IN ENGINEERING :
THEORY AND APPLICA-
TIONS, Swansea, U. K., January
7-11, 1985

Stress-strain Relation of 304 Stainless Steel for Cyclic Loading

H. ISHIKAWA

Professor, Hokkaido University

H. LIPPMANN

Professor, Technical University Munich

The stress analysis of structures subjected to cyclic loading requires stress-strain relations sufficiently simple to be usable for computer programming and yet adequate to describe the essential features of the plastic behaviour of the material.

In this paper the constitutive equation for cyclic plasticity incorporating the motion of the center of the loading surface is proposed. Using a modified plastic work, the dependency of the loading history of materials is taken into account. The Ramberg-Osgood law which is applied to each stress strain loop from the current center of the loading surface plays an important role in this theory. The theory is verified from the point of view of several kinds of experiments on type 304 austenitic stainless steel.

The 5th International Conference
on Production Engineering,
Tokyo, July 9-11, 1984

Mechanism of Internal Stress Generation in a Tensile State in High Speed Electroformed Nickel brought about Caused by Hydrogen

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Sapporo 060, Japan

Electroformed nickel from Watt electrolyte of normal has a toughness but has a disadvantage of high internal stress in a tensile state as compared with nickel from sulfamate nickel electrolyte. Actually low internal stresses are observed in nickel electroformed by certain high current densities and superimposed currents from Watt of normal. Whether the hydrogen contained in the high speed electroformed nickel contributes or not to the origin of the internal stress in a tensile state was determined by means of quantitative analysis of hydrogen in the nickel.

The hydrogen discharge from the electroformed nickel contracts the lattice of the nickel crystal, and as a result internal stress in tensile state occurs.

The 5th International Conference
on Production Engineering,
Tokyo, July 9-11, 1984

Electrochemical Machining with Bipolar Electrode Connection

Eiji MAKINO, Kenichi MARUYAMA and Toshikazu SATO
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The basic features of bipolar ECM were studied theoretically and experimentally using a nickel-base superalloy, Hastelloy X, as the workpiece, 3mol/dm^3 NaNO_3 electrolyte at 30°C , and an inert platinum anode. The current and the machining gap were measured against geometrical variables of an electrode configuration. The results show that an equilibrium condition was established during bipolar ECM under conditions of constant cathode feed rate and cell voltage. The equilibrium gap decreased with the increasing feed rate and with the increasing gap between the anode and the workpiece. And it becomes zero at a maximum feed rate which depends on a maximum current. By arranging the anode in proximity to the cathode, stray machining was reduced. Thus, undesired pitting, intergranular attack, and deformation of a workpiece shape at cutting edges can be prevented.

IFAC 9th World Congress,
Budapest, Hungary, July 2-6, 1984

Variational System Theory (II)

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Observing the behavior of a "functional" $J(t) = h(x(t))$ along the system trajectory $x(t)$ and making use of the variational formula for ΔJ corresponding to input perturbation $\delta u(t)$, we can study the stability, optimization and design procedures of the system. This is the basic manner of our thinking including the scope of study of our "Variational System Theory". We decompose various concepts of system theory into combinations of four properties: complete invariance, output controllability, stability and functional independence. Then, the suitable combinations of necessary and/or sufficient conditions of these properties yields the design procedures. To illustrate our methods, we present a design procedure of state feedback law, with which the system is non-interacting and the disturbances are decoupled. Moreover, we present a definition of the model following control system with a combination of complete invariance and stability, which is a natural one from a view point of variational approach. A sufficient condition is given and the obtained design procedure is successfully applied to an example of bilinear system: DC-motor. Some of our results seems to be equivalent to the "local" version of the studies via differential geometric approach. But, our methods are easy to understand and readily applicable to practical problems.

Proceedings of the 5th International Conference on Production Engineering, JSPE, Tokyo 1984

Automatic Dimensioning on 3-D Solid Geometry

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In traditional CAD systems, dimensioning is time consuming work—designers should specify dimensions with the aid of a light pen, cross cursor and so on. In any ideal CAD system, this work should be automated. This study is concerned with how to solve this problem—the problem of realizing automatic dimensioning. An approach is made from the point of view of knowledge engineering—because, dimensioning rules applied by designers are closely related to human knowledge and human experience. The approach we propose was applied to 3D solid geometry models and satisfactory solutions were obtained.

Proceedings of Computer Integrated Manufacturing and Robotics, The Winter Annual Meetings of the ASME, New Orleans, 1984

A Theoretical Approach to Generalized Robot Control Problem

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In this paper, a new problem of a generalized control problem of a robot is proposed and discussed theoretically. The proposed problem includes both geometrical and physical problems and a method of automatic trajectory planning is also proposed as one of the applications of optimal control.

In order to avoid the complexity arising from the primal problem, all the constraints and the performance indexes are transcribed to the corresponding penalty functionals and they are combined linearly to handle different kinds of entities.

Also to make the system accessible, the decoupling via state feedback is introduced.

The automatic trajectory planning for a revolute-type robot of seven degrees of freedom is experimented with a performance index of minimum-time and the constraints of the permissible angles and angular velocities of the joints.

Proceedings of the International
Symposium on Design and Synthesis,
JSPE/IFTOMM, Tokyo, 1984

Software Tools for Machine Design and Their Applications

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Mechanical engineering has a number of essential problems which must be solved immediately, such as shortening design-process time and establishing a technique for estimating design results. A trial-and-error method is generally employed in performing the design process on a problem which cannot be solved by an analytical method. It is important to grasp the influence of modifying specification. Moreover, there are a number of design variables closely related to each other. This paper represents on the designing method based on the boundary factor method and the software tools to perform the design process. The software tools are classified into two groups. One is the utilities to perform the design procedure independent of the given design object and the other is the units constructing the design object. The design base is proposed for the storage of the units and an example is demonstrated for the application.

Proceedings of the International
Symposium on Design and Synthesis,
JSPE/IFTOMM, Tokyo, 1984

Shell Geometric Modelling with the Euler Topology Characteristics

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The paper represents the shell geometric modelling for sheet metal products and shell structured products. A modelling method of shell geometry objects adopts the Euler topology based model first developed by Baumgart for the solid modelling. The Euler characteristic equation derived here is $v-e+f-r=s-h$ and Euler-like operations for the shell geometric modelling are introduced by the use of the derived equation. For designing the shell geometric modeller, the global topological properties to the surface such as orientability, closedness and genus and the data base structure, and the modelling verification are represented as well.

Proceedings of the 16th CIRP International Seminar on Manufacturing Systems, Tokyo, 1984

Pattern Recognition Approaches to GT Code Generation on CSG

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From the viewpoint of constructing an ideal engineering data-base system, the problems of how to model and store a part geometry with some attributes and how to recognize and retrieve the modeled geometry, play important roles. There are fine R&D on geometric modeling such as CSG, however, R&D on the recognition and retrieval of the modeled geometry are limited. Conventional GT code approach must be one of the keys to solve this latter problem. From this new angle of the problem, the pattern recognition approaches to GT code generation on CSG are proposed here. Regarding the axial geometry part, the generated GT code here is composed of of three kinds of code systems, i. e., dimensional code, macro geometry code and semantic code systems. By using this proposed GT code as atom data, some application problems, auto-conversion to other GT code, generative process planning, pattern recognition problems, e. g. are briefly shown with some results.

Proceedings of the 6th International Conference on Software for Discrete Manufacturing, PROLAMAT 1985, IFIP/IFAC PARIS, 1985

Development of SMDS : SONY Mold Design System

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To reduce the cost and period of designing an injection mold is one of the most important requirements facing many industries. A CAD system for designing an injection mold must be quite powerful to solve these requirements, but there are many difficult problems in developing the system. As one of these systems, SMDS (SONY Mold Design System) is developed using CSG solid modeler TIPS-1. In this paper, methodologies and algorithms to realize SMDS and some experiments using SMDS are presented.

Proceedings of the 6th international Conference on Software for Discrete Manufacturing, PROLAMAT 1985, IFIP/IFAC, PARIS, 1985

Development of Full Automatic Dimensioning System Based on 3D Solid Geometry

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The automatic dimensioning problem is one of the difficult problems in CAD. This paper describes an approach to the problem. First, CSG is given as geometry data, the geometric pattern is recognized and dimensions are extracted depending on the types of Primitives. Second, based on graph theory, the extracted dimensions are connected to each other under the constraint that related dimensions (such as size of Primitives) must not be missed. Third, to verify the consistency of the obtained dimensions, graph theory is introduced again. Experiments are conducted by using TIPS-1 Solid Modeler and practical mechanical parts (piston, valve etc.) are automatically dimensioned.

International Conference on Dislocations in Solids, Tokyo 1984

Shrinkage of Dislocation Loops by Emission and Absorption of Point Defects and Identification of Self-Diffusion Carriers in Metals and Semiconductors

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A general classification of thermally activated shrinkage mechanism of dislocation loops of vacancy and interstitial type is given. Characteristic difference between the shrinkage by emission and by absorption of point defects is demonstrated experimentally in a representative fcc metal. The analysis of the observed shrinkage behaviour in semiconductor germanium leads to the conclusion of the vacancies to be the defects responsible as the carrier in self-diffusion. Self-diffusion energies in these materials are estimated from the temperature dependence of the shrinkage speed of interstitial type dislocation loops. A discussion is given on the diffusion carrier in silicon.

International Conference on Dislocations in Solids, Tokyo 1984

Characterization of Dislocation and Stacking Fault in Electron Irradiated Germanium

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Two types of (113) faulted dislocation loops, interstitial type and vacancy type, can be introduced by 2MeV electron irradiation. By using lattice imaging technique together with diffraction contrast technique in an electron microscope, the atomic displacement situation around these faulted loops and the origin of electron microscope image contrast from the dislocation surrounding the stacking fault were investigated in detail.

13th International Conference on Defects in Semiconductors, Colorado 1984

Formation of Point Defect Clusters and the Nature of Point Defects in Electron Irradiated Ge and III-V Compound Semiconductors

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The nature of point defects in III-V compounds, deduced from the growth behavior of dislocation loops under electron irradiation, is presented. In Fe, lattice distortion around two types of radiation induced (113) faulted dislocation loops, of interstitial and vacancy type were carefully investigated by the electron microscopic technique called lattice imaging.

ICFRM-I First International Conference on Fusion Reactor Materials, Tokyo 1984

Defect Structure Evolution from Radiation Damage with Cascades

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Irradiation of a variety of metals in a rotating target D-T fusion neutron source RTNS-II and observation of defect structures were performed to obtain a unified understanding of defect processes. These were involved in damage structure evolution from irradiations that generate large cascades. The maximum separation of interstitial atoms from the vacancy rich zone is measured. Vacancy type defect clusters form groups reflecting the damage with sub-cascades, and the three-dimensional configuration of sub-cascades is disclosed by high resolution stereo-electron microscopy. The effective collision cross-sections to produce defect clusters is estimated, and the damage efficiency is obtained to be unexpectedly small. The variation of point defect processes is discussed based on the variation of the stability of small clusters in different materials irradiated at different temperatures. The roles of free interstitials released from cascade zones, the elimination of vacancy clustered defects and the formation of their own clusters, are clearly understood from a comparison of the results of irradiation of bulk specimen with those of thin foils. The necessity of dynamical effects from collisions on the defect cluster formation is concluded and its mechanism is discussed.

ICFRM-I First International Conference on Fusion Reactor Materials, Tokyo 1984

Development of Defect Structures from Displacement Cascade Damage in D-T Neutron Irradiated Gold

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Pure gold is adopted as a suitable material for the exposure of important damage and defect processes induced by fusion neutron irradiation. Vacancy type unit defects, typically the stacking fault tetrahedra of 1-2 nm, form groups of a size up to 20 nm containing up to 20 unit defects, reflecting the damage process in the presence of sub-cascades. The three dimensional configuration of sub-cascade damage is measured and illustrated. From the density of the sub-cascade groups, the neutron collision cross-section to create the observable defects is estimated to be 1-8 barns. The observed formation of vacancy clusters at low temperatures implies the existence of dynamical effect of collisions in the point defect reactions. Mechanism of defect structure evolution at higher temperatures, one defect cluster from one neutron

collision, is discussed and the importance of point defect cluster stability and the role of free interstitials are emphasized.

ICFRM-I First International
Conference on Fusion Reactor
Materials, Tokyo 1985

Formation of Secondary Defects in Copper by 14MeV Neutron Irradiation and Their Effects on Microstructure Evolution

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Pure Copper specimens were irradiated at 25, 200 and 400°C by 14MeV neutrons using RTNS-II to the dose of $3.6 \times 10^{22} \text{ n/m}^2$ and their damage structure was examined by means of transmission electron microscopy.

At 25 and 200°C, stacking fault tetrahedra (SFT), partially dissociated Frank loops, aggregates of vacancies, and interstitial loops are nucleated by cascade collapse. They have a tendency to be formed as a group up to about 10. Because SFT are very stable under irradiation, excess interstitials corresponding to the vacancies retained in SFT are accumulated in the matrix and form their clusters. Interstitial loops nucleated near a dislocation grow preferentially by absorbing the interstitials migrating towards the dislocation.

Voids were observed at 400°C. They will play a very important role in void swelling at a high doses.

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Conference on Fusion Reactor
Materials, Tokyo 1985

Roles of Free Interstitials and Temperature Dependence of Cascade Stability in D-T Fusion Neutron Irradiated Nickel

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An observation of defects formed in nickel by D-T neutron irradiation is made. The differences in number density and size of small dotted vacancy type clustered defects between thin foil and bulk specimens are fully accounted for by the reaction of free interstitials in the bulk, in contrast to their negligible effect in thin foils by their prompt escape to the specimen surfaces. Interstitial clusters observed

only in the bulk specimens are also the result of higher concentration of free interstitials during irradiation in the bulk than in the thin foils. A much less number of vacancy clustered defects is observed at room temperature in specimens irradiated at low temperature than in specimens irradiated at room temperature itself. Considering that the room temperature is still far lower than the free migration temperature of vacancies in this material, the temperature dependence of dynamical effects on the process of cascade collapse is discussed.

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Conference on Fusion Reactor
Materials, Tokyo 1984

Metastable Defects in D-T Neutron Irradiated Germanium

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The observed transmission electron microscope images of structural changes in germanium introduced by D-T neutron irradiation at room temperature are not those expected from a strain field of any crystalline defects nor those from phase shifts of crystal periodicity. A high resolution lattice image observation was performed, and the observed distorted images which are not accompanied with the surrounding lattice distortion are considered to support the occurrence of three dimensional volume of amorphous regions. These amorphous regions diminish in their size and the majority of them disappear during observation under an electron microscope whose electron acceleration voltage is far below the damage threshold, by the athermal transfer of energy to atoms from incident electrons, this is the so-called radiation induced type of diffusion. A discussion will be given on the relation of the observed structural changes with the degradation of radiation detectors and functional devices.

D-T Neutron Irradiation of JPCA and its Model Alloys

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Japanese Primary Candidate Alloy, TPCA and its pure based model alloys named A1 (Fe-15Cr-16Ni), A2 (Fe-15Cr-16Ni-0.25Ti), A3 (Fe-15Cr-16Ni-0.25Ti-0.06C) and A7 (Fe-15Cr-16Ni-0.5Si) were irradiated at 25, 200 and 400°C by 14 MeV D-T neutrons using RTNS-II. Radiation induced defect clusters were very small compared to those of pure metals such as Ni and Cu, and most of them are just above the limit of resolution (0.5nm). The formation of stacking fault tetrahedra, which were typical vacancy clusters in fcc metals, was confirmed from their image characters. The density of observable clusters at 25°C reached as high as $2 \times 10^{17}/\text{cm}^3$ by the irradiation of $5 \times 10^{17} \text{ n/cm}^2$, and did not depend strongly on the component. Cluster density at 200°C depended on minor elements. That of JPCA irradiated $7.5 \times 10^{17} \text{ n/cm}^2$ was $3.4 \times 10^{16}/\text{cm}^3$ which was more than one order smaller in A1 and A2. The mobility of vacancies at 200°C in A1 and A3 is high enough to form their clusters by thermal process but not enough in JPCA, and thermal rearrangement of vacancies in a cascade is important for microstructure evolution.

Low Temperature D-T Neutron Irradiation and Cryotransfer Observation of Cascade Defects of Metals

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Electron microscope specimens of Au, Ag, Cu, Al and Fe were irradiated below 20K by D-T neutrons with RTNS-II to the fluence of 1×10^{16} to $1.5 \times 10^{17} \text{ n/cm}^2$. After irradiation, foils were dismantled from the cryostat and mounted on a TEM cold holder in liquid nitrogen, and transferred to an electron microscope without warming them. The results presented here describe preliminary observations on only a few foils. Cascade defects composed of small defects in a group were observed in Au, Ag and Cu. The relaxation

of cascade defects occurring during the isochronal annealing was examined. No irradiation-induced defect was detected in cryotransferred aluminum at the instant of starting the observation, while observable cascade defects appeared during a subsequent illumination. This process is due to athermal collapsing of cascades induced by the electron illumination. In iron, no defects could be observed at low temperature or after annealing. Generally cascades collapse at low temperature to non-compact metastable structures and change to stable ones during the stage III annealing.

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Materials, Tokyo 1984

Cascade Defects in Very Low Dose D-T Neutron Irradiated Metals

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Very low dose of D-T neutron irradiation (10^{15} n/cm²) of thin metal specimens is made in order to observe an isolated cascade defect. Cascade defects were observed in all irradiated specimens of Au, Ag, Cu, Ni and Mo. As a typical example, the number density of cascade defects in gold was 1×10^{13} and 1.5×10^{14} /cm³ for 40 and 400 counts on a neutron detector, respectively. These values of the number density correspond to the cascade formation rate, 0.03 (cascade defect/cm³)/(n/cm²). The number seems to have a good agreement with those obtained from pure gold irradiated with higher doses. However, a remarkable difference of cascade defect structure was found in specimens irradiated between a very low dose and a higher dose. Only an isolated cascade defect was observed in very low dose specimens, while cascade defects have structures composed of several point defect clusters in the specimen of higher dose irradiation. The result suggests that cascade damages before their collapsing interact strongly with cascade defects already collapsed.

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**Electron Microscopic Studies of Damage Evolution
in Fission Neutron Irradiated Metals**

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The same kinds of specimens of both thin foil and bulk of metals as those irradiated previously with D-T neutrons at RTNS-II were irradiated with fission neutrons at KUR. The irradiations were performed at 20 and 350 K to the fluences of 5×10^{16} and 1×10^{19} f. n./cm², respectively. The results of pure Au were described to exemplify the typical value of cascade defects. A cascade in Au consists from 2.2 of the small defects on average. This is compared with 6.7 for the fusion neutron irradiation. The cross section for the formation of observed cascade defects was 0.51 barn which is twelfth of fusion neutron irradiation. The average size of small defects in fission neutron irradiated Au was much smaller than that of fusion neutron irradiation. The cryotransfer of thin foil to EM were carried out to observe the cascade defects at low temperatures. The damage evolution in bulk was also examined.

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**A Study of Point Defect Processes in Candidate
and Model Alloys for Fusion Reactor First Wall by HVEM**

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High energy electron irradiation on a modified 316 austenitic stainless steel and its model alloys, Fe-Cr-Ni, Fe-Cr-Ni-Ti and Fe-Cr-Ni-Si, and a ferritic steel was performed with a high voltage electron microscope. Interstitial type dislocation loops are formed in all alloys. A comparison of nucleation and growth speeds of the interstitial loops among four austenitic steels suggests a strong interaction between titanium atoms and vacancies and between silicon atoms and interstitials. The effect of preheat treatment above 600°C on loop nucleation during lower temperature irradiation was found, especially near the specimen

surface. In ferritic steel, the nucleation of interstitial loops occurs continuously during steady irradiation, where the dynamically balanced state of point defects is established. This difference from general behavior of pure metals is attributed to the smaller difference of mobilities between vacancies and interstitials in the steel than those of pure metals.

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Conference on Fusion Reactor
Materials, Tokyo 1984

Ion-Irradiation Experiment for the Fundamental Studies of Damage Evolution of Fusion Materials

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Cascade defects produced by ion irradiations were studied by the weak-beam method of electron microscopy for fcc metals (Au, Ag, Cu, Ni, Al) and bcc metals (Fe, Mo, V) as well as practical alloys (SUS316, ferritic steel). The thin foil specimens are irradiated with 40 to 70 KeV Ar⁺ and metal ions at the temperatures between -130 and 600°C, changing the ion flux and the irradiation fluence. The defect yields are greatly different for the materials and the defect densities increase proportionally only in the region of low fluence. The numbers of the observed defects show a tendency to saturate in the region of high fluence. The repeated irradiations show that the individual defects change their size discontinuously and disappear suddenly in Au. A typical feature of unstable defect evolution is seen in Al, where the observed defects appear only in the high flux ion irradiations and at relatively low temperatures.

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Conference on Fusion Reactor
Materials, Tokyo 1985

Correlation Among a Variety of Miniaturized Mechanical Tests and Their Application to D-T Neutron-Irradiated Metals

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The tensile, disk bulge and microhardness tests with miniaturized specimens were performed on a variety of metals and alloys. It was shown that both the bulge fracture load and the microhardness strongly correlate with tensile strength. Specimens as thin as 0.1 mm generally fulfil the requirements for obtaining bulk properties from miniaturized tensile tests, for materials where grain size can be properly controlled.

The tensile and bulge tests were carried out on Ni, Au, Cu and austenitic stainless steel irradiated with D-T fusion neutrons from RTNS-II. Au and Cu showed a large increase in yield strength up to factors of 7 and a reduction of ductility down to 50% by the irradiation of 1.7×10^{22} n/m². The increase of the yield strength for Ni and stainless steel is about half that for Cu and Au, and the decrease of ductility is small for stainless steel. The variation of deformation mode with increasing neutron fluence was detected by optical and electron microscopy.

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Conference on Fusion Reactor
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Low Fluence Neutron Irradiation Responses to Ferritic Stainless Steels

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Five kinds of ferritic stainless steels were irradiated at 300 K, 473 K and 673 K with 14 MeV neutrons to a fluence of 1.3×10^{18} n/cm² and were irradiated with fission neutrons at about 600 K to a fluence of 5×10^{18} n/cm². Micro-bulge test, micro-Vickers hardness test and electron microscopy were used to characterize the radiation damage. Many radiation induced defect clusters were observed and the temperature dependence and the fluence dependence of those defect clusters suggest them to be vacancy type clusters. Corresponding to the microstructural changes irradiation softening were observed.

The 10th International Cryogenic
Engineering Conference, Helsinki,
Finland, July 31-August 3, 1984

Strategic Usage of SMES for Utility in the Year 2000

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The method and the benefit of the future usage of 1GW 5 GWh SMES in the Japanese power system proposed for use in the year 2000 was studied. The size is estimated as appropriate for the Japanese electric power demand in the year, 2000. A theoretical study to obtain the optimal operation scheme considering the economical benefit revealed that SMES has its characteristic features which are slightly different from the conventional storage method such as the pumped hydro power station. The exclusive daily cycle is favored for SMES while the pumped hydro power station is used not only for daily but also for weekly cycles. The macroscopic strategy using SMES was extensively discussed for various situations of the power system.

The 8th Power Systems Compu-
taion Conference, Helsinki, Fin-
land, August 19-24, 1984

On the Optimal Allocation of the Measurements in Power System State Estimation Considering Loss-of-Measurements Conditions

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On the optimal allocation of measurements in power system static state estimation, this paper presents a new idea ; i. e., deciding such a measurement set with minimum redundancy where the states of a power system can be estimated with better accuracy than at a prespecified desired level even in the loss-of -measurements conditions as well as in normal condition. The proposed solution process consists of three fundamental tasks ; i. e., Task I, Task II and Task III.

Task I makes the power system be observable even in the loss-of-measurements conditions by adding new measurements. A new index defined in this paper and named as appropriateness is used in order to select the appropriate additional measurments from the probable candidate measurements. Task II evaluates expected accuracies of estimator for the normal and all loss-of measurements conditions. Task III decides the optimal measurements to be added in order to improve the expected accuracy of the estimator against the normal condition or the worst loss-of- measurements condition.

1984 International Conference on
Plasma Physics, Lausanne, Switz-
erland, June 27-July 3, 1984

An Analysis of Plasma Surface Waves Based on Special Functions and a Boundary Element Method

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In the cylindrical coordinates, plasma surface waves for cold plasma with a noncircular cross section are studied using a boundary element method (BEM). From the formulation based on the BEM, we have the dispersion relation of the plasma surface waves. For the plasma with a circular cross section, we can conclude from the numerical results based on the BEM that the relative error is less than 0.1 % for using the boundary element with one tenth of plasma radius and the characteristic of convergence has nearly a second-order accuracy. On the other hand, if the plasma is surrounded by the dielectric constant, the dispersion relation of the plasma surface waves agrees with the dispersion equations which can be verified numerically through an analysis based on the special functions in the toroidal coordinates. Furthermore, for the toroidal plasma with a metal wall, a dispersion relation of plasma surface waves is studied using the toroidal functions.

1984 International Conference on
Plasma Physics, Lausanne, Switz-
erland, June 27-July 3, 1984

Three-Dimensional Analysis of Convective-Diffuse Plasma Flow Using a Boundary Element Method

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For a three-dimensional steady-state resistive plasma flow, we have the convective-diffuse equation with a source term. In order to solve the equation, the boundary element method (BEM) is proposed for not only obtaining stable solutions but also to save data preparation. From the boundary element solutions, we can conclude the following : (1) the solution is unconditionally stable in space, i. e., the stability of solution is independent of the type of boundary element used in the analysis. (2) the relative error almost never depends on the value of Peclet number, Pe . (3) the characteristic of convergence has an approximate second-order accuracy. (4) the mixed BEM solutions in comparison with the constant BEM have a rather high accuracy with the increase of the value of Pe . Therefore, it is found that the mixed

BEM is more useful and effective in studying resistive plasma flow for complex geometry.

6th International Conference on
Boundary Element Methods in
Engineering, on board the liner,
the Queen Elizabeth II, South-
ampton to New York, July 3-8,
1984

Three-Dimensional Boundary Element Solutions for a Steady-State Convective Diffusion Problem Using 0-1 Order Boundary Elements

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Convective diffusion (CD) problems have been already analyzed in fluid dynamics. Such a problem has become of some interest lately in electrical and electronic engineering; for example travelling magnetic fields. However, it is extremely difficult to solve a CD equation when the convection term dominates because most of the domain-type methods give solutions which oscillate in space.

In order to eliminate such a numerical instability, the authors apply an effective and efficient boundary element method which is based on a mixed discretization using both constant and linear elements (mixed BEM) to a steady-state CD equation in three dimensions.

Spatial potential distributions, relative errors and convergence are investigated as compared with those of BEM with only constant elements (constant BEM) and exact solutions for a simple test model. As a result, we can conclude as follows :

- (1) The solution is stable in space up to nearly the Peclet number $Pe=600$ in the present code.
- (2) The relative error almost never depends on the value of Pe .
- (3) The characteristic of convergence has a second-order accuracy, similar to that of constant BEM.

1984 International Symposium on
Electromagnetic Compatibility,
Tokyo, Japan, October 16-18, 1984

An Analysis of TE_{10} Cutoff in TEM Cells Using the Boundary Element Method

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A transverse electromagnetic transmission (TEM) cell developed at the National Bureau of Standards

is analyzed by using the boundary element method (BEM). In this paper, we study the cutoff problem of TE_{10} mode in the TEM cells. The cutoff problem of TE_{10} mode is governed by the scalar Helmholtz field, so that the problem is reduced to an analysis of the scalar Helmholtz field. In the analysis, in order to reduce the data required to solve the problem, we use the BEM which requires only discretization of the external boundary. Cutoff frequency is obtained numerically from the secular equation with respect to the wavenumber. Furthermore, in order to reduce the input data, we consider the symmetrical condition under which all higher modes are divided into three pairs of modes. As a result, we can obtain cutoff frequency with high accuracy. It is found that the BEM is available for the analysis of cutoff problem in TEM cells.

1984 International Symposium on
Electromagnetic Compatibility,
Tokyo Japan, Oct. 16-18, 1984.

A Three-Dimensional Analysis of a TEM Cell Model by a Mixed Boundary Element Method

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Transverse electromagnetic (TEM) transmission cells are analyzed three-dimensionally using a boundary element method based on a mixed discretization using both constant and linear elements. In this work, the distributed characteristic impedance is evaluated from the boundary values solved. This is one of the most important characteristic quantities for structure design.

As a result, in this paper we show the following :

- (i) The present numerical method is very powerful in the three-dimensional characteristic impedance analysis of TEM transmission lines.
- (ii) A non-uniform division such as the one based on zeros of Tchebycheff polynomials is effective for three-dimensional problems, and it shows a better convergence with fewer boundary elements.
- (iii) On the basis of these conclusions, we will analyze a more realistic TEM cell model which is tapered and also the main line sections are taken into account.

4th International Conference on
Applied Numerical Modeling,
Tainan, Taiwan, December 28-31,
1984

Boundary Element Method Applied to MHD Equilibria of Toroidal Plasmas

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MHD equilibria of toroidal plasmas described by the Grad-Shafranov equation are numerically analyzed by using a boundary element method (BEM). Since the BEM has the advantage of the reduction in the effective dimensionality by one, this means considerable saving of data preparation and computational cost in three dimensional problems. In the present work, the BEM is applied to treat three cases of compact toroidal plasmas with force-free fields, with both toroidal and poloidal fields, and with a poloidal field alone. Boundary element solutions are compared with both analytical and finite element solutions. As a result, it is found that the BEM is available in solving the MHD equilibrium problem of the compact toroidal plasma. As numerical examples, we analyze MHD equilibria of the qun-generated compact toroidal plasma with a center conductor and of the spheromak configuration.

Sino-Japanese Joint Meeting on
Fiber Science and Electro-
magnetic Theory, Beijing, China,
May 15-19, 1985

Analysis of Three-Dimensional Steady-State Convective Diffusion Equation Using Regular Boundary Element Methods

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Regular boundary element method (R-BEM) is applied to analyze convective diffusion equation. We consider a three dimensional cubic model with Neumann's boundary condition as a simple example in order to study the stability and accuracy of regular boundary element (R-BE) solutions. It is found that R-BE solutions are unconditionally stable and their relative errors almost never depend on the location of source points. We can also show that R-BE solutions have a second-order accuracy as well as conventional BE solutions. In addition, numerical precision is studied through the condition number of the system matrices used in the analysis for a few parameters. As a result, it is shown that the R-BEM is available for the analysis of three-dimensional steady-state convective diffusion equations.

Sino-Japanese Joint Meeting on
Optical Fiber Science and Electro-
magnetic Theory, Beijing, China,
May 16-19, 1985

A Boundary Element Approach to Laplace-Poissonian Field Problems

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A new boundary integral formulation is presented in order to solve a general Laplace-Poisson's equation, which is one of the basic equations of semiconductor devices. As this formulation is based on Green's second identity or Gauss' divergence theorem, no conventional volume integral is needed, regardless of the arbitrary distributions of space charges. The potentials and electric field intensities at interface nodes placed between a Laplace and a Poisson domain are analytically calculated, because the interface nodes are treated in the same way as internal points. It is an effective and powerful tool for device analysis such a junction-gate field effect transistor with interfaces movable according to the operation bias conditions. On the basis of simple numerical experiments, the present method is applied to simplified device models. It is shown that device analysis can be easily obtained for a more small discretized model. In consequence, numerical results also demonstrate the effectiveness of this approach.

1985 IEEE International Confer-
ence on Plasma Science, Pitts-
burgh, U. S. A., June 3-5, 1985

Mixed Boundary Element Methods Applied to Analysis of TEM Waveguides for ICRF Plasma Heating

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Transverse electromagnetic (TEM) waveguides with two inner conductors used for ion cyclotron radio frequencies (ICRF) plasma heating are analyzed by using the mixed boundary element method (mixed BEM). As design criteria, available transmission powers and characteristic impedances are calculated for several geometrical parameters of TEM waveguides. From the numerical results, in order to increase available transmission powers, it is necessary to increase the separation of two inner conductors with a larger radius of curvature. On the other hand, when taking into account the matching impedance, it is necessary to decrease the characteristic impedance because the impedance of plasma is as sufficiently small as copper. As a result, there exists the trade-off problem that the increases of the available transmission powers result in an increase of a separation while matching characteristic impedances results in a decrease of the separation.

5th COMPUMAG Conference on
the Computation of Electro-
magnetic Fields, Fort Collins, U.
S. A., June 3-6, 1985

Regular Boundary Element Steady-State Solutions of the Traveling Magnetic Field Problem

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Regular boundary element method (R-BEM) is applied to analyze the steady-state traveling magnetic field problems for which convective diffusion equation is considered as the governing equation. We deal with a three-dimensional rectangular prism as a simple example in order to study the stability and accuracy of regular boundary element (R-BE) solutions. It is found that R-BE solutions are unconditionally stable for a rectangular prism to be extended along a traveling direction. Furthermore, we can show that R-BE solutions have a second-order accuracy as well as the conventional BE solutions. Finally, numerical precision is studied through the condition number of the system matrices used in the analysis for a few parameters. It is shown that the R-BEM is available for the analysis of three-dimensional steady-state convective diffusion equations.

The Electrochemical Society
167th Meeting State-of-the-Art
Program on Compound Semicon-
ductors, Toronto, Ontario, Cana-
da, May 12-27, 1984

On the Role of Surface in the Performance of Compound Semiconductor Devices

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A large number of surface states which appear on the GaAs surface imposes various restrictions on the performance of GaAs devices. One of the restrictions is the surface breakdown and side-gating. Evidences are given indicating the side-gating due to bulk states being initiated by the surface breakdown, which is, in turn, caused by the surface states. The way to reduce side-gating as well as the possibility of alternative materials such as InP will be discussed.

European Workshop on Active
Microwave Semiconductor De-
vices, October 10-12, 1984, Veld-
hoven, Netherlands

Recent Japanese Developments of High-Speed Devices by Non-MESFET Approaches

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Although the self-aligned version of the "classical" MESFET has been extremely successful for integration, achieving LSI complexities, intensive efforts are also being made towards highspeed devices by non-MESFET approaches in order to exploit the potential speed capability in the compound semiconductor materials to its fullest extent. This paper reviews the current status of such efforts made in Japan, including HEMTs, MIS-like FETs, HBTs and HETs and InP MISFETs.

HEMTs have now achieved LSI complexity, resulting in a subnanosecond 1kb SRAM with 4kb version being pursued. Process control and photo-induced instability of V_{th} , compatibility with implantation for self-alignment are the major issues. Use of AlAs/GaAs SL reduces DX centers and improves the stability. The MIS-like FET using undoped AlGaAs semi-insulator is an attractive alternative to the HEMT structure for 77k operation.

For realization of high-speeds in LSI complexity, large driving capability of devices is an essential requirement. Thus, the HBT gate is a natural extension of Si ECLs. A discrete device with f_t of 25 GHz has recently been fabricated and its integration is now being pursued. Tunneling HET is expected to achieve subpicosecond operation.

Since the FET current is determined by velocity x sheet carrier concentration, InP and InGaAs MISFETs can potentially offer advantages of large drivability, less tight control of V_{th} and large N. M. for room temperature operation. Reduced drain current instability with a high channel mobility has recently been obtained.

European Workshop on Active
Microwave Semiconductor De-
vices, October 10-12, 1984, Veld-
hoven, Netherlands

Integrated Photoconductive Detector/FET GaAs Receivers with Simple Optical Coupling to Optical Fibers for Interconnections in Very High Speed IC's

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In order to meet the need for high speed, cross-talk free interconnections in high speed IC's, integration of highspeed GaAs photoconductive detectors and MESFETs with simple coupling to optical fibers is investigated.

Two photoconductive detectors and one FET were integrated on a 0.2 μm thick n-type MOCVD GaAs layer doped to $2 \times 10^{17} \text{ cm}^{-3}$ grown on a semi-insulating GaAs substrate. Photoconductors had a width of 200 μm and a length of 10 μm . Gate length of the MESFET was 10 μm and width 500 μm .

For simple coupling of optical fibers to the integrated receiver, a single mode fiber was laid directly on one of the detectors and coated with epoxy resin. To enhance the efficiency of distributed coupling between fiber and detector, optical fiber was etched to 5 μm to remove the coating and cladding. The resulting connection loss between fiber and detector was about -1.4 dB with 5 ns overall response time, which is excellent considering the very simple method and structure employed here. The response time is limited by the surface state effect of the detector and can be improved by employing window layers such as GaAlAs.

Monolithic integration of photoconductive detectors with GaAs MESFETs together with a simple and low-loss coupling of optical fibers to the integrated high speed receivers are thus shown to be very promising for optical interconnection in very high speed IC's and systems.

1984 Int. Symp. GaAs and Related
Compounds, Biarritz, France,
Sept. 26-28, 1984

Mechanism and Suppression of Drain Current Drift in InP MISFETs

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Drain current drifts in n-channel $\text{Al}_2\text{O}_3/\text{InP}$ and $\text{Al}_2\text{O}_3/\text{native oxide}/\text{InP}$ MISFETs prepared by anodization in AGW electrolyte or in oxygen plasma were studied. The interfaces were characterized by Auger profiling, ellipsometry, I-V, C-V, DLTS and PCTS methods.

To explain the temperature dependence of drift, previous models assumed interfacial defect states lying above E_c . However, this temperature dependence is a measurement artifact. The new model involving interface states below E_c , explains the drift quantitatively.

Incorporation of a thin native oxide interfacial layer by anodization reduces N_{ss} , and suppresses drift. This is explained by the present model for interface states.

SSI circuits exhibited excellent low-frequency stability with a high channel mobility of 2000-3000 $\text{cm}^2/\text{V} \cdot \text{sec}$, showing promise for LSI/VLSIs.

1984 Int. Symp. GaAs and Related
Compounds, Biarritz, France,
Sept. 26-28, 1984

A New Side-Gating Model for GaAs MESFETs Based on Surface Avalanche Breakdown

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Side-gating of MESFETs limits the packing density of GaAs LSI/VLSIs. This paper studies the side-gating of OMVPE MESFETs on undoped LEC substrates and with PCVD SiO₂ or Si₃N₄ passivation.

Side-gating took place in the presence of negative side-gate potential with its threshold V_T equal to breakdown voltage between the side gate and source.

Experimental results include light emission from the source outer edge at the onset of side-gating, long-range nature of side-gating, linear dependence of V_T on side gate-to-source distance and extreme sensitivity of V_T to passivation conditions. These cannot be explained by the previous space charge limited current model. A new model is proposed which includes field concentration by surface state filling and the resulting avalanche injection.

The 13th Congress of the Interna-
tional Commission for Optics,
August 20-24, 1984, Sapporo

Monolithic Integration of GaAs Photoconductive Detectors and MESFETs for Optoelectronic IC's

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In order to meet the need for high speed optoelectronic IC's for interconnections in very high speed IC's and systems, integrated detector/FET GaAs receivers with simple coupling to optical fibers are developed.

Two photoconductive detectors and one FET were integrated on a 0.2 μm thick MOCVD GaAs layer doped to $2 \times 10^{17} \text{ cm}^{-3}$ grown on a semi-insulating GaAs substrate. Two alloyed Au/Ge : Ni ohmic contacts with 10 μm separation and 200 μm width were formed for a photoconductive detector. The Gate length of the MESFET was 10 μm and width 500 μm , showing 13 mS of transconductance. Two photoconductive detectors were connected in a series and the gate of the MESFET was connected to the electrode between two detectors. The unwanted epitaxial layer was etched away for device isolation.

The surface of one detector was illuminated perpendicularly with 0.85 μm light from a GaAlAs/GaAs laser diode. The rise time of the integrated receiver was 5 ns for photon flux density of $1 \times 10^{20} \text{ cm}^{-2}\text{s}^{-1}$.

The change in output current was 1.4 mA when FET was loaded with a 50 ohm resistor.

For simple coupling of optical fibers to the integrated receiver, a single mode fiber was laid directly on one of the detectors and coated with epoxy with refractive index of 1.44. To enhance the efficiency of distributed coupling between the fiber and the detector, the optical fiber was etched to 5 μm . The resulting connection loss between the fiber and the detector was about -1.4 dB with a 5ns overall rise time, which is excellent considering the very simple method and structure employed here.

The 16th (1984 International) Conference on SOLID STATE DEVICES AND MATERIALS, KOBE, 1984

Panel Discussion GaAs LSI/VLSIs : Advantages and Applications

Organizer/Moderator :

H. HASEGAWA, Hokkaido University

Panel Members :

M. Abe, Fujitsu Laboratories Ltd.

P. M. ASBECK, Rockwell International Corporation

A. HIGASHIZAKA, NEC Corporation

Y. KATO, Sony Corporation

M. OHMORI, Nippon Telegraph & Telephone
Public Corporation

Over the past several years, we have witnessed a rapid increase of integration size and functional Complexity in GaAs digital integrated circuits, now reaching LSI/VLSI levels. Until recently, large scale integration was limited only to D- and E/D MESFETs, but we recently have heard about successful LSI level integration of HEMT devices and heterojunction bipolar transistors (HBT), resulting in a subnanosecond HEMT 1kb SRAM and an HBT 1k gate array. In a smaller scale of integration, various GaAs-based devices, including MESFETs, JFETs, HEMTs and heterojunction bipolar devices, have consistently shown impressive speed-power performances significantly outperforming various commercially available Si devices.

Therefore, apparently the time has now come for one to ask on a more realistic basis what roles we should expect for this fascinating technology of GaAs ICs to play in this world of silicon.

The present panel is intended to discuss the advantages of various technologies of GaAs ICs over Si technology. More specifically, D- and E/D-MESFET, JFET, HEMT and HBT technologies are compared with Si technologies in terms of performance, functional complexity and ease of fabrication. Pertinent application areas in near term or far term are to be identified for each technology.

The panel includes GaAs technologists who are actively involved in the IC development.

43rd Annual Device Research Conference, June 17-19, 1985, University of Colorado, Boulder, Colorado

Surface Electrical Breakdown and Leakage Current Between Ohmic Electrodes on Semi-insulating InP Substrates

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One of the factors limiting the achievable packing density in the compound semiconductor LSI/VLSIs is the failure of the device isolation due to surface electrical breakdown and/or to sidegating. Although small scale integration as well as excellent performance of InP power MISFETs are reported, there has been no report on the surface electrical breakdown and the side-gating in InP. This paper reports the surface electrical breakdown characteristics in semi-insulating InP and shows that the surface breakdown voltage in InP is at least one order of magnitude higher than GaAs.

Surface I-V characteristics between two Au/Ge ohmic electrodes formed on Fe doped LEC semi-insulating InP wafers were measured. The activation energy of the leakage current was also measured. The surface was either bare or covered with passivating films such as plasma CVD SiO₂, Si₃N₄ and anodic native oxide.

The leakage current of InP shows ohmic behavior up to 10-30 kV/cm after which InP permanently breaks down. This behavior is in contrast to the GaAs where a threshold voltage V_T is observed above which a steep rise in current takes place. V_T of GaAs corresponds roughly to 1 kV/cm. Leakage current in InP is sensitive to the passivation processes and a thin anodic native oxide gives the lowest leakage. The measured activation energy of the ohmic current is also extremely sensitive to the processing condition.

High breakdown voltage in InP is believed to be due to the reduced number of surface states compared to GaAs. This is backed not only by the fact that InP has less surface states than GaAs but also by the fact that the Fe doping level in InP is comparable to the EL2 density or Cr doping level in GaAs suggesting that the breakdown is not controlled by bulk.

A new model for surface breakdown and sidegating in GaAs which involves surface states was reported recently [1]. According to this model, high energy carriers generated by microplasma due to surface breakdown above V_T trigger the sidegating. Since InP shows no such V_T , it is expected that InP is immune to sidegating.

The present observation indicates that InP is a material superior to GaAs from the point of view of device isolation in highly integrated compound semiconductor LSI/VLSIs.

[1] Hasegawa, H., Kitagawa, T., Sawada, T., and Ohno, H. : Int. Symp. Gallium Arsenide and Related Compounds, 1984.

1985 ELECTRONIC MATERIALS CONFERENCE, University of Colorado, Boulder, CO, June 19-21, 1985

**(GaAs)_i/(InAs)₁ Superlattice Semiconductor
Grown by Molecular Beam Epitaxy**

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This paper deals with the growth of (GaAs)_i/(InAs)₁ superlattice by molecular beam epitaxy, which belongs to a new class of semiconductor, the superlattice semiconductor. The name superlattice semiconductor is used for semiconducting monolayer layer to several monolayer superlattice since the electronic structure of this semiconducting material cannot be treated as a structure formed by two semiconductors described by effective mass theory. A variety of new properties is expected to be realized by this superlattice semiconductor. One of the possibilities is the elimination of alloy scattering in Ga_{0.47}In_{0.53}As alloy by replacing the alloy by (GaAs)_i/(InAs)₁ superlattice semiconductor which has almost the same average composition.

(GaAs)_i/(InAs)₁ superlattice semiconductor was grown on InP substrates with and without AlInAs buffer layer and on GaAs substrates with GaAs buffer layer. Growth temperature was in the range of 450 C to 500 C with the growth rate of 0.5 μm/h. Shutters were operated with pneumatic valves and 2 sec time interval was inserted between closing one shutter of group III material and opening another one. Arsenic beam was kept constant during growth. RHEED oscillation was observed and used to calibrate the growth rate. Streaky RHEED pattern was observed throughout the run. The total number of periods was 360 to 720.

The grown layer was characterized by X-ray diffraction, optical absorption, and Hall measurements. X-ray diffraction revealed that superlattice was formed : no significant interdiffusion was observed. Band gap determined by the optical absorption measurement was 0.83 eV at room temperature. Theoretically calculated band gap using tight binding approximation is 0.935 eV at 0 K which corresponds to 0.87 eV at room temperature and agrees well with the experimentally observed value. The purity of the layer was not enough to observe the elimination or reduction of the alloy scattering. Typical carrier concentration was about $2 \times 10^{17} \text{ cm}^{-3}$.

Although there is 7% lattice mismatch between GaAs and InAs, the present study indicates that it possible to synthesize a new superlattice semiconductor out of GaAs and InAs. Superlattice semiconductors with other combinations will provide various different advantages over the conventional compound and alloy semiconductors.

1985 ELECTRONIC MATERIALS CONFERENCE, University of Colorado, Boulder, CO, June 19-21, 1985

Carrier Depletion and Deep Level Formation at Re-growth Interface in MOVPE GaAs

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Advanced high speed devices such as Static Induction Transistors and Pemeable Base Transistors require a re-growth of the epitaxial layer after processing gate structures. It is essential for such devices that the re-growth interface being high quality : without any unintentionally introduced impurities nor deep levels. This paper describes what happens when such a re-growth process is carried out on GaAs using metalorganic vapor phase epitaxy (MOVPE).

All growths were performed in a vertical reactor with a load-lock apparatus under atmospheric pressure using TMG and arsine. GaAs layers were grown at 760°C on n⁺ GaAs substrates at a rate of 500 Å/min with V/III=20. No dopants were used in this study. Typical carrier concentration under this growth condition was 1 to 2 x 10¹⁶ cm⁻³ with a room temperature mobility of 4800 cm² V⁻¹ s⁻¹.

The Growth was terminated by stopping TMG flow and then the arsine flow was stopped when the substrate temperature became 400°C. The surface of the epitaxially grown GaAs was exposed to air and treated by various solvents as well as etchants before the second growth. Then the sample was loaded in the reactor and the second layer of GaAs was grown directly on the treated surface. Al Schottky diodes were formed on the surface and In-Sn ohmic contacts on the backside for electrical measurements.

Carrier concentration profiling by C-V measurements revealed that a significant carrier depletion of about 2.0 x 10¹¹ cm⁻² was present at the re-growth interface when the surface was exposed to air for 3 hours prior to re-growth. The carrier depletion extended for 2000 to 3000 Å and the lowest part showed n=5 x 10¹⁵ cm⁻³. Treating the surface by organic solvents or by HCl prior to the second growth did not reduce nor enhance the carrier depletion. Deep level transient spectroscopy was used to detect deep levels. A broad electron trap signal was observed at the regrowth interface. No electron traps except EL2 were observed in the layer away from the interface.

The depletion of carriers may be understood as carbon contamination during exposure to air. The effect of other treatments as well as the way to eliminate such carrier depletion will be discussed.

1985 ELECTRONIC MATERIALS CONFERENCE, University of Colorado, Boulder, CO, June 19-21, 1985

On the Role of Interface States in a-Si : H MIS Structures

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It has been believed that the gap state plays a major role in a-Si : H MIS characteristics and the effect of interface states has been usually ignored. This paper presents evidences that the electrical characteristics of a-Si : H MIS structures are controlled by the interface states.

a-Si : films were prepared by glow-discharge decomposition of SiH_4/H_2 onto n^+ crystalline silicon substrates. Two different insulators were formed on a-Si : H films for MIS structures : anodically oxidized Al_2O_3 /native SiO_2 double insulator and plasma CVD Si_3N_4 . Vacuum evaporated Al dots on insulators were used as electrodes.

C-V measurements were done using 5 Hz to 1 MHz measurement frequencies at 70°C. Both insulators showed similar low frequency (5 Hz) MIS C-V characteristics with two structures : a dip in capacitance on the positive bias side and a steep decrease on the negative side. At high positive bias (around 20 V) the capacitance becomes insulator capacitance. Between the dip and the steep decrease, the capacitance approaches insulator capacitance.

The onset of steep decrease shows a strong frequency dependence, indicating the structure being due to the long time constant of the states well under the conduction band. The structure on the positive bias side is due to the movement of surface potential. As the voltage goes from negative to positive, high frequency capacitance starts to increase at the voltage where low frequency capacitance shows a dip, supporting this interpretation. Effective gapstate density including both the gapstates and the interface states were calculated from the dip in 5Hz C-V curve. The effective gapstate density was too high to be interpreted by the gapstates in a-Si : H but is in the reasonable range of 10^{13} to $10^{14} \text{ cm}^{-2} \text{ eV}^{-1}$ as interface states. The density of effective gapstates is reduced dramatically by annealing at low temperature below the substrate temperature during deposition of a-Si : H. Both facts indicate that the dominating states are the interface states.

Further evidence of the interface states controlling the MIS interface is obtained by isothermal capacitance transient spectroscopy (ICTS), where a comparison is made between the ICTS signal from MIS diodes and Schottky diodes. ICTS signal from Schottky diodes shows a prolonged tail in capacitance transient whereas signals from MIS diodes show only relatively fast transient. This difference in time constant is another strong evidence of responding states being different in the two structures : a-Si : H gapstates in Schottky and interface states in MIS.

GaAs IC SYMPOSIUM,
BOSTON, MASSACHUSETTS,
OCTOBER 23-25, 1984

A Microwave Photoconductance Technique for Non-Destructive Characterization of Semi-insulating GaAs Substrates

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Electrical uniformity of the semi-insulating substrate is the key factor that governs the fabrication yield of the GaAs integrated circuit chips. The purpose of this paper is to describe a simple method for non-destructive characterization of semi-insulating substrates, using a microwave photoconductance technique. The technique was first proposed by the present authors [1] on an empirical basis. This paper clarifies the photoconductance mechanism on the basis of detailed experimental data on both Cr-doped HB and undoped LEC materials. It is shown that this method is capable of measuring the distribution of Cr acceptors in HB wafers and that of carbon and other residual acceptors in undoped LEC wafers in a non-destructive way.

A microwave coplanar stripline probe is used to detect the surface conductance change due to illumination by Ar or He-Ne laser through non-destructive capacitive coupling. The probe is connected to a microcomputer controlled automatic data acquisition system, and is scanned over the substrate.

The microwave photoconductance mechanism is analyzed by solving the rate equation for free carriers near the surface, taking account of photogeneration, Shockley-Read recombination, direct recombination, surface recombination and diffusion of carriers. The main results are the following: (1) when the illumination intensity I is low, the photoconductance G is proportional to I and is related to the effective carrier life time. For a high surface recombination velocity S ($=10^6 - 10^8$ cm/s) as is the case for GaAs, G is given by $G = K \cdot I / \sqrt{D}$ (K : constant, α : absorption constant and D : diffusion constant) and is independent of the exact value of S . (2) $G \propto \sqrt{I}$ for large I due to domination by direct recombination. (3) In both Cr-doped HB and undoped LEC substrates, G is dominated by photogenerated electrons. (4) In the case of Cr-doped HB substrates, $1/G$ is proportional to ((Cr concentration) - (residual donor concentration)). (5) In the case of undoped LEC substrates, $1/G$ is proportional to the residual acceptor concentration since it is equal to the concentration of ionized EL 2 donors under low photoexcitation.

Detailed photoconductance and photo-Hall experiments were performed together with (dark) sheet resistance and EPD measurements, using HB wafers with known Cr concentrations and undoped LEC wafers with known carbon concentrations (as determined by the FTIR technique). The results were in excellent agreement with the above (1)-(5). Undoped LEC substrates showed either W-shaped or M-shaped variation of G across the wafer with more rapid fluctuations being superposed on it. The observed variations of sheet resistance, the reported variations of cathode luminescence intensity and of activation for ion implantation are all consistent with the interpretation that the observed G variation is due to carbon and not to EL 2, indicating accumulation of carbon at dislocations by the Cottrell effect which lead to the reported FET threshold voltage dependence on distance from the dislocation [2].

In contrast to the IR absorption technique which measures the distribution of EL 2 donors in the bulk [3], the present method measures the distribution of acceptors near the surface which obviously has a more direct influence on the threshold voltage variation of n-channel FET devices.

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International Conference on Molecular Beam Epitaxy, August 1-3, 1984, San Francisco, California

Deep Level Characterization of AlGaAs and Selectively Doped N-AlGaAs/GaAs Heterojunctions

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High quality AlGaAs and AlGaAs/GaAs interfaces grown by MBE made it possible to realize selectively doped heterojunctions as well as superlattice structures, which are currently used in devices such as High Electron Mobility Transistors (HEMT). To fully understand the operation of these devices, characterization of AlGaAs layers and AlGaAs/GaAs interface properties are of extreme importance. Indeed McAfee et al [1] found deep levels localized near the AlGaAs/GaAs interface, which could influence the mobility of electron gas in selectively doped heterojunctions.

In this paper we report the results of deep level characterization of Si doped AlGaAs layers and the interface of selectively doped N-AlGaAs/GaAs heterojunctions grown by MBE. The selectively doped N-AlGaAs layers were grown on n^+ as well as on semi-insulating GaAs substrates at 680°C. GaAs buffer layer of 0.5 μm was grown onto substrates, and then 23 nm undoped $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ followed by 140 nm Si doped $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ was grown. The resulting room temperature mobility was $\mu = 4900 \text{ cm}^2/\text{Vs}$ with a sheet carrier concentration of $n_s = 1.0 \times 10^{12} \text{ cm}^{-2}$, and $\mu = 170,000 \text{ cm}^2/\text{Vs}$ with $n_s = 4.0 \times 10^{11} \text{ cm}^{-2}$. Aluminum dots were deposited on the Si doped AlGaAs layers as well as on selectively doped structures to form Schottky barriers for DLTS measurements.

Three electron traps have been detected by DLTS in AlGaAs layers. Two of them (activation energy $E_a = 0.48 \text{ eV}$ with capture cross-section $S = 1.8 \times 10^{-12} \text{ cm}^2$ and $E_a = 0.45 \text{ eV}$ with $S = 9.4 \times 10^{-12} \text{ cm}^2$) were the main dominant levels in the bulk of Si doped AlGaAs, which correspond to DX centers reported by Lang et al [2]. The remaining one (0.46 eV with $2.0 \times 10^{-13} \text{ cm}^2$) was detected in the bulk AlGaAs as a prolonged tail of the DLTS signal from DX centers. Curve fitting was used to obtain accurate values of activation energies and capture crosssections. The last level ($E_a = 0.4 \text{ eV}$) showed a peak in concentration near the AlGaAs side of the heterojunction interface in the selectively doped heterostructures. All three traps were found in the AlGaAs layer in the selectively doped N-AlGaAs/GaAs heterostructures. The location of the deep levels near the interface and its effect on the mobility of electron gas at the selectively doped heterojunction will be discussed.

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The 16th (1984 International)
Conference on SOLID STATE
DEVICES AND MATERIALS,
KOBE, 1984

Al₂O₃/Native Oxide Double-Layer Mis Structure for InP MISFET ICs

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This paper describes novel InP MISFETs with high quality double-layer gate insulators of Al₂O₃ and thin native oxide, showing high effective electron mobilities and marked reduction of drain current instability. Feasibility of the MISFET ICs is demonstrated by E/D inverters and ring oscillators.

N-channel enhancement-mode MISFETs were fabricated by a simple anodization process of evaporated Al on semi-insulating InP in electrolyte or in oxygen plasma. For electrolytic anodization, aqueous solution of tartaric acid mixed with propylene glycol (AGW) was used. Plasma anodization was performed using a newly developed oxidation system with a grounded mesh electrode between rf electrode and sample holder in order to avoid high rates of electron and ion bombardment caused by high energy plasma.

MIS capacitors were prepared on conducting substrates to assess the dielectric and interface properties [1]. Double-layer structures possess high resistivity and high breakdown strength with minimum N_{ss} of $9 \times 10^{10} \text{ cm}^{-2} \text{ eV}^{-1}$.

Enhancement and depletion-mode MISFETs prepared by both electrolytic and plasma anodization operated successfully with good saturation characteristics. Transconductance g_m of 10-12 mS/mm were obtained with 25 μm channel length at a gate bias of 3V, which corresponds to large g_m of 250-300 mS/mm when the gate length is reduced to 1 μm . For the double-layer MISFET prepared by the wet-anodized process, μ_{eff} were typically 1500-3000 cm^2/Vs with the best value of 3050 cm^2/Vs . For the double-layer MISFET prepared by the plasma-anodized process, μ_{eff} of 3200 cm^2/Vs was obtained for the best value.

The present double-layer MISFETs showed a drastically improved stability. This was explained on the basis of a new understanding of the drift mechanism obtained by drift characterization of double-layer MISFETs. Contrary to the previously proposed models[2][3], which assume involvement of states above the conduction band edge, it is clearly established that localized states below the conduction band edge at the interface are responsible for the decreasing-type drain current drift.

Enhancement-depletion (E/D) type InP MISFET inverters and 7-stage ring oscillators, were fabricated using the electrolytic anodization, in order to demonstrate the feasibility of the process employed. Inverter characteristics were found to be stable down to the measured lowest frequency of 10^{-2} Hz. This low-frequency stability of the inverter is greatly superior to GaAs MOSFET inverters. It is also consistent with the drastically reduced drain current instability in the present double-layer gate MISFETs. The ring oscillator with gate lengths of 5 μm for E-FET and 20 μm for D-FET, showed propagation delay per gate $t_{pd}=4$ ns with power-delay product $Pt_{pd}=7$ pJ at a supply voltage $V_{DD}=3\text{V}$ at 128 K. The propagation delay of 4 ns scales down to 160 ps for 1 μm gate length, which is limited by relatively large over laps between gate and source/drain electrodes.

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The 1st International Photo-voltaic Science and Engineering Conference, Nov. 13-16, 1984, KOBE

Selective Passivation of Material Defects by Anodic Oxidation for Fabrication of Large-Area Amorphous Silicon Solar Cells

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Hydrogenated amorphous silicon (a-Si : H) is one of the most suitable materials for low-cost and large-area solar cells. However, various material defects are present in a-Si : H films. The purpose of the present paper is to describe the selective passivation of the material defects in a-Si : H films by the anodic oxidation process, which enhances the fabrication yield and improves the photoelectric quality of a-Si : H solar cells.

An ethylene glycol solution of KNO_3 (0.04 mol/l) was used for the anodic oxidation of a-Si : H films. The anodic oxidation for passivation was done in a constant voltage mode in the dark. When thick oxide films were prepared, a large number of luminescent spots appeared on the a-Si : H films in the early stage of oxidation. As anodization proceeded, the luminescent spots merged together forming luminescent filaments, and then the filaments consolidated to form uniform luminescence over the whole wafer. This observation indicates the presence of nonuniformly distributed material defects acting as a current shunt path which is anodized out in the early stage of anodization.

To clarify the passivation effect by anodic oxidation, a-Si : H p-i-n solar cell arrays were fabricated. Each array consists of 36 p-i-n cells, having a n area of 5mm x 5mm, and the fabrication yield judged from I-V characteristics under illumination was compared before and after application of the passivation process. Without the passivation process, the typical yield of operating cells was about 70%. By removing metal electrode patterns and applying the dark anodization, all the cells worked. It was found that this process is also necessary for the high-yield fabrication of large area p-i-n solar cells (3cm x 3cm).

It should be noted that an optimum formation voltage (V_f) exists for the maximum passivation effect with best solar cell performance. The higher V_f is preferred for the passivation of the material defects acting as a shunt path, at the same time the oxide thickness at other parts should not be too thick so as not to deteriorate the solar cell performance. This optimum voltage was found to be 9V in the present experiment. Auger electron spectroscopy revealed that oxide thickness and composition of oxide films on a-Si : H films do not change up to this optimum 9V.

The 16th (1984 International) Conference on SOLID STATE DEVICES AND MATERIALS, KOBE, 1984

Amorphous Silicon MOSFETs by Anodic Oxidation

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Anodic oxidation allows formation of uniform thin and thick SiO_2 and $\text{Al}_2\text{O}_3/\text{SiO}_2$ insulating layers on hydrogenated amorphous silicon (a-Si : H) films at room temperature, as reported by the present authors [1]. The anodic oxidation process is shown to be stable, reproducible and electrically controllable as shown in [1]. The purpose of the present paper is to describe a-Si : H planar MOSFETs fabricated by such a process. Details of oxidation process and mechanism, MOS interface study, fabrication process and performance of MOSFETs are presented.

Al_2O_3 /native SiO_2 double insulating layer was grown by anodization of an Al/a-Si : H structure. Al was deposited in vacuum onto the grown a-Si : H film at substrate temperature range of 80–150°C. The electrolyte for anodization is AGW electrolyte [2], which is a mixture of 3% aqueous solution of tartaric acid and propylene glycol. The anodization was done in constant current mode. Since holes are required for anodization of a-Si : H, the anode was illuminated by W-lamp (70000 lx) during anodization. Resulting Al_2O_3 and SiO_2 thickness were 1200 Å and 60 Å.

MOS structures allow standard C-V and DLTS measurement for interface state and bulk deep trap measurements. G-V characteristics in MOSFET structures also provide useful information concerning the interface states.

a-Si : H films were deposited on the glass (Corning 7059). The n^+ -layer on the channel and device separation region was chemically etched off using an etchant consist of $\text{HF} : \text{HNO}_3 : \text{CH}_3\text{COOH} = 3 : 5 : 15$, which has an etching rate of 0.3 $\mu\text{m}/\text{min}$ for a-Si : H. $\text{Al}_2\text{O}_3/\text{SiO}_2$ layer was then formed. After a-Si : H MOS FET fabrication, annealing in H_2 gas in a temperature range of 160–230°C was done to improve the FET characteristics. The gate length and the gate width of the fabricated FET were 110 μm and 4.8 mm, respectively. The gate width was designed to be adequately wide in order to obtain a reasonable drain current level, which is typical for a-Si : H FETs due to the low conductivity of the material.

The annealing was done in H_2 gas at 230°C for 15 min. The effective mobility μ_{eff} , calculated from $\mu_{eff} = (L/W) (1/C_{ox}) (I_d/V_g) (1/V_{ds})$, was typically 0.1–0.2 cm^2/Vsec . The annealing process greatly enhanced the effective mobility by a factor of 2–10, which was typically 0.002–0.05 cm^2/Vsec prior to annealing. It is believed that this improvement is due to the reduction of interface state densities at insulator/a-Si : H interface by annealing. The effective mobility increased with temperature with an activation energy of 0.14 eV, indicating trap controlled non-dispersive transport. The mobility value at the high temperature limit was as high as 60 cm^2/Vs .

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The 13th Congress of the International Commission for Optics, Sapporo, Japan, August 20-24, 1984

Determination of Thickness Distribution in Non-Uniform Film Sample by Ellipso-Interferometry

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In this paper some consideration and experiment on ellipso-interferometry are presented from the view point of its application to mapping thickness of wedge-shaped thin films. The theoretical errors due to the use of a popular computer program which has been developed for analyzing parallel plane layer are calculated. The errors are small for most cases of thin film analysis excluding the regions around the multiples of film-thickness period. Therefore, the combination of ellipso-interferometry and computer programs of this kind are useful for the thickness determination of the film in which parallelism of both surfaces is poor. A demonstrative experiment with a slightly wedge-shaped thin film of SiO_2 is also shown.

Symposium on Structure and Reactivity of Coal March, 1985, London, England

Reaction Mechanism of Coal Hydrogenation (III) Effect of Coal Type

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Taiheiyo coal was liquefied in Naphthalene and tetralin under 10 MPa initial hydrogen pressure, using three catalysts; stabilized nickel, Co-Mo on alumina and iron dust with sulfur or without using catalyst. Naphthalene conversion was in the order of Ni Fe Co-Mo no catalyst and the hydrogen consumption showed also the same tendency. In tetralin the conversion and hydrogen consumption tendency were similar as in naphthalene, but the strong catalyst dominantly consumes the hydrogen from molecular hydrogen, in contrast to this in the weak or no catalyst cases the hydrogen was absorbed mainly from tetralin. This tendency confirms the previous conclusion, namely, under active catalyst the liquefaction proceeds by the absorption of gaseous hydrogen even in tetralin.

Symposium on Chemistry of coal
liquefaction and catalyst March,
1985, Sapporo, Japan

Coal Liquefaction by the Hydrogen In-situ Generated from Methanol Decomposition Studies on Catalyst

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Taiheiyo coal was liquefied with methanol. Several kinds of methanol decomposition catalysts and hydrogenation catalysts were examined. CuO containing catalysts showed an excellent high conversion. The liquid yield distilled up to 600°C achieved yielded more than 80% (daf base). The model reaction revealed that this catalyst has a methanol decomposition activity as well as splitting and hydrogenation activity.

The 1984 International Chemical
Congress of Pacific Basin Society
Dec. 1984, Hawaii, USA

Liquefaction of Coal by In-situ Generated Hydrogen from Methanol

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Methanol easily decomposes to hydrogen and carbon mono oxide in the presence of a suitable catalyst. The authors utilized this in-situ generated hydrogen for coal liquefaction. Taiheiyo coal, methanol, ZnO-Cr O (methanol decomposition catalyst) and stabilized Co (hydrogenation catalyst) were placed in an autoclave and heated at various temperatures for various times. The benzene conversion was measured in the products. The effect of time, temperature, solvent addition etc. was examined. The coal was liquefied effectively.

The 1984 International Chemical
Congress of Pacific Basin Society
Dec. 1984, Hawaii, USA

**Liquefaction of Coal by Treatment for a Long Time at Low Temperature
and for a Short Time at High Temperature under Relatively Low
Hydrogen Pressure**

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Akabira coal was hydrogenated at 420°C for 2 hours with iron dust with sulfur as the catalyst and wash oil as the solvent under 12-13 MPa of hydrogen pressure, then the temperature was rapidly raised to 500°C and the reaction was held for 0-20 minutes. After the reaction the distillate yield was measured by thermobalance. In comparison with the first step reaction product, the second step reaction product increased the distillate yield significantly. When the coal was liquefied in the same system at 500°C for 0-10 minutes directly without treating at 420°C, the distillate yield was lower, especially for the oil yield distilling distillation as a lower temperature range.

7th International Symposium on
Organosilicon Chemistry, Septem-
ber 9-14, 1984, Kyoto, Japan

**Highly Stereoselective Synthesis of Allylic and Dienyl Silanes from
Terminal Alkynes by the Stepwise Cross-Coupling Reaction of
(Z)-2-Bromo-1-alkenylboranes**

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Among a number of methods for the preparation of allylic and vinylic silanes whose increasing importance in organic synthesis has been amply verified. Only a few of them allow for a variety of structural modifications with complete regio- and stereochemical control. In the course of our work on haloboration reaction, we found that stereodefined trisubstituted olefins can be synthesized by the palladium-catalyzed, two-step cross-coupling reactions of (Z)-2-bromo-1-alkenylboranes, available in situ by the bromoboration of 1-alkynes. This reaction proceeds particularly well when silicon-substituted organometallics are used in the first coupling stage, thereby making the substituted, stereochemically homogeneous allylic and butadienyl silanes readily accessible. Moreover, our approach permits virtually unlimited structural variations.

The 1984 International Chemical
Congress of Pacific Basin Socie-
ties, Honolulu, Hawaii, U. S. A.
December 16-21, 1984.

Superacid Catalyzed Oxygenation of Single σ -Bonds in Functionalized Organic Compounds With Ozone

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The chemistry of the reactions of single σ -bonds with electrophiles generated in superacid media is an interesting and developing field. In this connection, electrophilic oxygenation of alkanes with protonated ozone (O_3H^+) genrated in superacid media is particularly noteworthy, in that it produces carboxonium ions, which can subsequently be hydrolyzed to alcohols and ketones. We extended our investigations of this novel reaction toward the functionalized organic compounds such as ethers and found the electrophilic insertion of protonated ozone into tertiary or secondary C-H bonds located at γ or further away from the oxygen atom in aliphatic ethers which lead to prouduce oxoalkyl ethers in $HF \cdot SbF_5$ at $-40^\circ C$. Ethers having $CH_3(CH_2)_n$ groups ($n > 2$) could react with protonated ozone almost exclusively to give (ω -1)-oxoalkyl ethers in good yields.

10th International Symposium on
the Reactivity of Solids. August
27-September 1, 1984, Dijon,
France

Emanation Thermal Analysis for the Initial Reactivity of Oxide Powders

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The phase-boundary reaction which takes place in the initial reaction stage plays an important role in the solid-state reactions. Emanation thermal analysis (ETA) was applied to the initial reactivity measurements of oxide powders for the Fe_2O_3 -ZnO and TiO_2 - $BaCO_3$ systems. The samples were labeled by a surface impregnation method using Ra-226 solution (3-4 $\mu Ci/mL$). New informations concerning the changes of the surface layer smaller than about 100 nm in thickness were obtained under working conditions from the ETA curves over the temperature range 25 to $1400^\circ C$.

10th International Symposium on
the Reactivity of Solids, Dijon,
France, August 27-September 1,
1984

Development of Duplex Structure of Magnetite Sintered in Nitrogen

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Powder compacts of several commercial Fe_3O_4 reagents were sintered in N_2 at 1150°C . Microscopic observations showed that only one reagent developed the duplex grain size distribution after the sintering. This duplex structure was considered to be due to the inhomogeneity of Fe^{2+} and Fe^{3+} ion distribution in Fe_3O_4 particles which are larger than $15\ \mu\text{m}$.

Plenary Lecture, 4th International
Symposium on Agglomeration
June 2-5, 1985, Toronto, Canada

Coordination Numbers and Porosity of Packing Calculated from Particle Size Distribution

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In the course of developing the kinetics of tumbling granulation of solid particles having wide range of sizes, it became necessary to have a knowledge of the number of contacts or coordination number per particle in a completely mixed packing. In relation to the contact numbers, theoretical porosity estimation is now going on in our laboratory by using the size distribution. The present lecture will outline the scope of our work and mention some possibilities for the application of our results.

Knowledge of the coordination numbers and the overall volume porosity is important for evaluating the cohesive strength of powder, so long as we know the cohesive force working at a contact point. It is also expected that the present theory can be applied to analysis of solid-solid chemical reaction when it proceeds through the contact points. Furthermore, by calculating the inter-molecular potential based on the nearest points we have been somewhat successful in predicting azeotropic liquid mixtures. Porosity estimation may be of vital importance for improving the strength of fine ceramics or for its processing, and another application can be made for obtaining very concentrated suspensions of solid in liquid, coal-water mixture for example.

Fourth International Symposium
on Organic Free Radicals, St.
Andrews, U. K., July 9-13, 1984

**Transformations of Steroidal Cyclic Alcohols and
Ketones into Cyclic Ethers Involving Alkoxy Radical
Rearrangement and Radical Cyclization**

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New methods for the transformations of hydroxysteroids and steroidal ketones into oxasteroids involving alkoxy radical rearrangement are presented.

The methods involve the formation of novel formates by β -scission of the alkoxy radicals, generated by the irradiation of the hypoiodites of hydroxysteroids or steroidal lactols derived from steroidal ketones and the cyclization of these formates with NaBH_4 or methyl lithium to cyclic ethers.

The experiments which used ^{18}O labelled mercury (II) oxide provided evidence that the transformation of hydroxysteroids into the formates involves a novel intramolecular combination of the carbonyl oxygen with a carbon-centered radical generated by the β -scission.

The 1984 International Chemical
Congress of Pacific Basin Soci-
eties, Honolulu, Hawaii, U. S. A.
December 16-21, 1984.

**Regioselectivity in the Electrochemical Introduction
of the Allylic Groups and Its Control**

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The allyl group of substituted allyl halides can be introduced to ketones, aldehydes, or α, β -unsaturated esters by means of electrochemical reductions. A detailed study of the regiochemistry of these reactions shows that when the carbonyl component is more readily reduced than the allyl component, the C-C bond formation takes place at the less substituted carbon of the allyl halides to give homoallyl alcohols which are not accessible by means of the organometallic reactions; but when the allyl halide is more readily reduced, the C-C bond formation takes place at the more substituted site of the allyl halides.

We also report how regioselectivity may be controlled by changing the electrolytic conditions. The application of the present electrochemical reaction to the synthesis of some terpenes is also reported.

2nd JAPAN-BELGIUM SEMI-
NAR ON POLYMER SCIENCE
BRUSSELS, BELGIUM 1-4 Octo-
ber, 1984

Solid State High Resolution NMR Studies on Reactions in Solid Polymers

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Recently, a high resolution ^{13}C -NMR spectrum is observable from a solid sample by the cross-polarization and magic angle spinning (CP-MAS) technique. These techniques were applied to cross-linked polymers and polyacetylene, which are insoluble into liquid solutions, in order to find out the chemical species produced either by the γ -irradiation or by the thermal degradation. In ethylene-propylene rubber, relative concentrations of the chemical groups were changed after the formation of cross-links by γ -irradiation. New chemical species were detected by the NMR from polyacetylene after thermal degradation in air. Formations of both methyl group and tertiary carbons were confirmed by the NMR. The presence of these carbons suggests the network formation in the thermally degraded polyacetylene. ^{13}C -NMR spectra were observed from γ -irradiated polyethylene having high crystallinity. Gauche fractions of polyethylene were quantitatively determined in the crystalline parts of γ -irradiated polyethylenes.

The 35th International Society of
Electrochemistry Meeting, Berke-
ley, California, U. S. A., August 5-
10, 1984

Passivity of Metallic Titanium (Invited speech)

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The anodic passivation of titanium in aqueous phosphate and sulphate solutions over a wide range of pH was studied by ellipsometry and Raman scattering spectroscopy. The thickness of the passive oxide film on titanium increases nearly linearly with potential in a potential range where the film appears to be amorphous. Above a critical potential (7.5V vs. RHE) the rate of film growth with potential becomes greater probably due to crystallization of the film. The Raman spectrum reveals that the film is primarily composed of anatase TiO_2 . The cathodic reduction of the film brings about the reductive dissolution of the film in acid solution and causes the transformation from TiO_2 to TiOOH in neutral solution.

The 13th Congress of the International Commission for Optics,
August 20-24, 1984, Sapporo

**Spectra of the Thin Surface Oxide Films
Anodically Formed on Iron and Titanium**

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and Norio SATO

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The spectrum of complex refractive index of anodic oxide films on iron and titanium was measured by the 3 parameter reflectometry in order to investigate the semiconductive property. The anodic oxide film on iron exhibits a resemblant absorption spectrum to that of α -Fe₂O₃ which includes two absorption edges for photo excitation of the band electron. The anodic oxide film on titanium has a absorption edge at $h\nu=3.2\text{eV}$, whose photon energy is slightly larger than the band gap (3.05eV) of bulk TiO₂. This discrepancy may be attributed to the hydration effect of the anodic oxide film.

The IXth International Conference
on Raman Spectroscopy, August
27-September 1, 1984, Tokyo

**Raman Spectroscopy Applied to the Oxide Films
Formed on Metals in Air and in Aqueous Solution**

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The oxide films thermally formed on SUS 304 stainless steel in air and anodically formed on titanium in aqueous solution were studied by laser Raman spectroscopy. The oxide film on the stainless steel was found to change in its composition at a temperature of 700°C. Below 700°C the main compounds of the film are α -Fe₂O₃ and Fe-Cr spinell and above 700°C however the main compounds turn to Fe-Cr spinel oxide and Cr₂O₃. For the anodic oxide film on titanium the anatase type of TiO₂ was identified in the potentiostatic oxidation at 10.6 V vs RHE in pH 6.9 phosphate solution.

The USA-Japan Seminar on Corrosion, March 11-14, 1985, Nikko

Laser Raman Spectroscopy for In Situ Study of Thin Corrosion Films on Iron

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Laser Raman spectroscopy and the ellipsometry were applied for the detection and characterization of thin corrosion films formed on iron in air at a temperature range from 100°C to 150°C. It is found that the oxidation of iron in dry air leads to the formation of a surface oxide film which is composed of magnetite and that the water vapor in air accelerates the formation of hematite. The ratio of magnetite to hematite in the surface oxide film appears to increase with increasing water vapor pressure, temperature, and oxidation time.

5th International Conference on Surface and Colloid Science, Clarkson University, Potsdam, New York, U. S. A., June 24-28, 1985

Characterization of MnO₂ by Zn²⁺ Ion Adsorption

Hiroki TAMURA and Masaichi NAGAYAMA
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Adsorption of Zn²⁺ ions on MnO₂ samples was examined as a function of solution pH and Zn²⁺ ion concentration. The amount of adsorbed Zn²⁺ increased with the pH and the Zn²⁺ concentration in solution. The behavior was described with a Frumkin type isotherm in which retardation of adsorption due to electrostatic repulsion or steric hindrance of adsorbed Zn²⁺ is taken into consideration. The isotherm contains an equilibrium constant and a constant for the retardation effect. The values of the constants clearly reflected the different properties of oxide prepared under different conditions.

Symposium on 'Anodic Oxidation', the 176th Meeting of the Electrochemical Society, May 12-17, 1985 Toronto, Canada

Anodizing of Aluminum Covered with Hydrous Oxide Films

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Anodizing of aluminum treated with boiling water produces oxide films with excellent dielectric properties and the process has been used in manufacturing electrolytic capacitors. In this investigation, attempts were made to examine (1) the amount and composition of hydrous oxide formed by the boiling treatment, and (2) the structure and properties of anodic oxide films formed on boiled specimens. The oxide films are shown to consist of two layers capable of sustaining high voltages and the mechanism by which these layers are formed is explained.

Fifth World Hydrogen Energy
Conference, Toronto, Canada,
July 15-20, 1984.

Catalytic Decomposition of Hydrogen Sulfide and Methanol over Molybdenum Disulfide

By Masatoshi SUGIOKA and Kazuo AOMURA
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The decomposition of hydrogen sulfide and methanol over molybdenum disulfide catalyst were investigated to produce hydrogen and syngas ($\text{CO} + \text{H}_2$) by use of a closed circulation reactor system at 500°C and 400°C , respectively. The catalytic activity of MoS_2 for the decomposition of H_2S was greatly enhanced by the reduction treatment with hydrogen but was hardly increased by the evacuation treatment at high temperatures ($500^\circ\text{C} - 800^\circ\text{C}$). A possible mechanism was proposed for the catalytic decomposition of hydrogen sulfide over MoS_2 where the coordinatively unsaturated site of MoS_2 surface formed by the reduction with hydrogen acts as the active site.

In the decomposition of methanol over MoS_2 catalyst, the catalytic activity and selectivity of MoS_2 for the decomposition of methanol were remarkably changed by the addition of a small amount (5wt%) of alkali metal ions (Li, Na, K, Rb and Cs). Especially, the catalytic activity of MoS_2 was considerably increased by the addition of potassium ion and the main product was syngas. It was concluded that there is a possibility to develop an effective sulfur-tolerant MoS_2 catalyst for the production of syngas from methanol by the modification of MoS_2 with a small amount of potassium ions.

8th International Congress on Catalysis, Berlin (West), 2-6 July 1984

The Differences in Acidity between High-Silica Zeolites Characterized by Calorimetry, ESR, and MASNMR

M. NITTA, M. YAMASHITA, M. SHIOTANI, K. AOMURA,
Y. NOZAWA, M. HATANO

The heat of immersion of HZSM-5 and HZSM-11 zeolites with the same Si/Al ratio of 60 pretreated at temperatures from 473 to 1073K are used to investigate their acidity differences and to compare them with the acidity of low-silica HY zeolite. Dependence of heat of immersion on the pretreatment temperature is considerably different between the two zeolites. The heat of immersion of HZSM-5 in pyridine and in n-butylamine are higher than those of HZSM-11. The heat values per site indicate that the acid strength in the former is higher than that of the latter. ESR spectra of adsorbed benzene revealed the formation of three kinds of radicals. The surface hydroxyl hydrogen (Brønsted acid sites) takes part in the formation of cyclohexadienyl radicals. HRMAS-Si (29)-NMR spectra of HZSM-5 and HZSM-11 show three chemical shifts at -107, -115, and -118ppm, and the lines will be also discussed in relation to the acidity. These suggest that a delicate structural change occurs in the channel framework of HZSM zeolites with pretreatment at higher temperature.

19th International Conference on Coastal Engineering, Houston, Texas, U. S. A., Sept. 3-7, 1984.

Evolution of Interfacial Waves Along an Unsteady Salt Wedge

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Field measurements of the receding salt wedge were made at the mouth of the Ishikari River in October 1981. The gradually growing interfacial waves and the diffusion pattern of salinity are visualized by an Ultrasonic Method. The evolution mechanisms of waves are analysed in detail by calculating the power spectra of wave records. It is pointed out that the wave breaking, the wave-eddy interaction and the nonlinearity of waves are factors dominating the evolution.

The nonlinear property of interfacial waves is studied further by watertank experiments.

10th International Symposium on
Nonlinear Acoustics, July 24-28,
1984 Kobe, Japan

Nonlinear Transport Theory of the Surface Acoustic Waves -Surface Acoustic Soliton-

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The purpose of this paper is to suggest theoretically the possible existence of the surface acoustic soliton and to give a fundamental method to amplify or reshape it through the interaction with electrons. Starting from the equation of motion for the anharmonically interacting surface phonons, the theory of the surface acoustic soliton in a semiconductor is developed based on the coherent-state representation. The two-dimensional displacement field at the surface is shown to satisfy the nonlinear integro-differential equation with a damping term. With the aid of the reductive perturbation method, the equation can be reduced to the nonlinear Schrödinger equation with a damping term whose coefficient is the attenuation rate of the surface phonons.

An approximate solution is derived on the basis of the first integral and compared with the numerical solution to reveal an excellent agreement. In the presence of an applied dc field, it is shown that the amplification of the surface acoustic soliton can be attained as the electron drift velocity exceeds the sound velocity.

The 13th Congress of the Inter-
national Commission for Optics,
August 20-24, 1984, Sapporo.

Interferometric Spectral Imaging

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A method is described for efficiently obtaining comprehensive information of a polychromatic radiator. Under certain conditions both the spatial and spectral details of the radiative object can simultaneously be recovered from the 3-D spatial coherence function in the diffraction region. The recovery of object information is based on a Fourier transform relationship derived from the well-known formula describing the general propagation law for the mutual coherence of polychromatic partially coherent light. A new type of interferometer is proposed for the efficient collection of the spatial coherence data. Experimental results of the spectral-image recovery are also presented.

The 13th Congress of the International Commission for Optics,
August 20-24, 1984, Sapporo.

Acoust-Optic Coherent-Incoherent Conversion for Partially Coherent Optics

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Hokkaido University, Sapporo 060, Japan

A proposal is presented for producing a secondary, spatially incoherent light source from a coherent laser source by use of an optical Bragg cell. If the first-order diffracted beam of light is selected and modulated in frequency, such an incoherent source results over the back focal plane of a Fourier forming lens. The theoretical background is given together with some preliminary experimental results.

Sino-Japanese Joint Meeting on
Optical Fiber Science and Electro-
magnetic Theory, Beijing, China,
May 16-19, 1985

Measurement and Analysis of Spatial Coherence at the end of Optical Fiber Excited by a Laser Beam

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Modulus and phase of the complex degree of spatial coherence at the exit end of a coherently excited optical fiber was analyzed by taking into account the mode-coupling between propagating modes. The pointwise correlation between two axially symmetrical points across the fiber core exhibits an oscillatory behavior obeying the form of $\cos(\beta_r - \beta_n)z$. It was found that the most probable value for the modulus and phase can be obtained by taking an average over its period of oscillatory envelope. Such theoretical considerations were confirmed by the experiment using a computer-aided wavefront-reversing interferometer.

2nd International Conference on
Optical Fiber Sensors, Stuttgart,
FRG, Sept. 6-7, 1984

**Polarization Noise in Birefringent Single-Mode Fiber Link
with Imperfect Joints**

M. IMAI and Y. OHTSUKA

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Hokkaido University, Sapporo 060, Japan

Angular misalignment between birefringent axes of two connecting fibers as well as misaligned coupling of linearly polarized light into one of the birefringent axes of input fiber gives rise to polarization noise and yields a reduction of maximum 7 dB in the signal-to-noise ratio.

The 13th Congress of the Inter-
national Commission for Optics,
Sapporo, Japan, August 20-24,
1984

**Spatial Coherence in a Coherently Excited Optical Fiber
and its Application to Mode Analysis**

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Department of Engineering Science, Faculty of Engineering
Hokkaido University, Sapporo 060, Japan

When a spatially coherent light such as He-Ne laser is used to excite multimode optical fibers, intermodal interference or beating between modes plays an important role in evaluating pointwise correlation of the optical fields. Theoretical considerations show that the modulus and phase of the complex degree of spatial coherence exhibit an oscillatory behavior along the propagation distance of optical fiber. Spatial coherence at the exit end of optical fibers was investigated experimentally by means of a computer-aided wavefront-reversing interferometer and the modal structures of a weakly guiding fiber were analyzed by comparing the experimental plots with the theoretical predictions.

The 13th Congress of the International Commission for Optics, Sapporo, Japan, August 20-24, 1984

Multiple Logic Operations by use of an Optical Interference System

Yoh IMAI, Yoshihiro OHTSUKA, and Keiji TANAKA*

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Optical logic gates operating in parallel can be implemented by using an optical interference system. Nonlinear transformation between optical inputs and outputs is based on a shifting operation of an output fringe due to photorefractive nonlinearity. Multiple-output scheme of gates is achieved by utilizing a dividing technique on the output fringe, taking into account of the fringe shift. For example, maximal eight kinds of gates ; AND, B, OR, OR, XOR, and their complements, are executed simultaneously at the positions divided over one period of the fringe under the phase conditions of $\pi/2$ and $2\pi/3$ for respective inputs. The number and sorts of the logic operations performed are adjustable via the shift and/or the division of the output fringe.

10th International Symposium on Nonlinear Acoustics, 24-28 July 1984, Kobe Japan

Nonlinear Sound Waves and Shock Waves Radiated from a Pulsating or Oscillating Sphere

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Shinya ISHII and Tomio OKIGAMI

Department of Mechanical Engineering, Faculty of Engineering Science, Osaka University, Toyonaka 560, Japan

The propagation of nonlinear waves emitted from a pulsating sphere is of fundamental importance in nonlinear acoustics. A representation of the exact solution for the far field equation in a first approximation is presented, which is closely related to the approximate solution so far obtained by the renormalization technique. A uniformly valid solution is then sought for in a second approximation. The shock formation distance is evaluated from the result.

Also, investigated are nonlinear waves emitted from an oscillating rigid sphere. In the true multidimensional case, a similar representation to the former can only be derived for the radial velocity. The tangential velocity can successfully be determined by a simple method. The formation and propagation of shock waves is also studied in the limit when the coefficient of viscosity vanishes.

The First Sino-Japanese Physical
Metallurgy Symposium, Beijing,
China, Nov. 8-11, 1984

Effect of Carbon Atoms on Vacancy Migration in Pure Iron and Fe-C Alloy

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**Beijing University of Iron and Steel Technology, Beijing, China

In the present work, the "in situ" observation on vacancy clustering was carried out using a high voltage electron microscope (HVEM) in order to study the behaviour of defect clustering caused by vacancy migration in Fe and carbon containing steel (0.1160 at% C). Irradiation was performed by a HVEM operating at 1000 kV and at an average damage rate of 2×10^{-3} dpa s^{-1} in the temperature range between 300 K and 623 K. When pure iron was irradiated, interstitial dislocation loops were initially formed and they grew with the irradiation resulting in the high dislocation density. The dislocation density was higher at lower irradiation temperature. Void formation was recognized after few dpa in the whole temperature range. On the other hand, when the iron with carbon was irradiated, voids were also formed at the same temperature range as in pure iron, but irradiation dose up to void appearance increased in comparison with pure iron. Furthermore, by post-irradiation annealing at temperatures below stage IV after irradiation at room temperature the void growth was recognized in both specimens. These results suggest that vacancies produced by electron irradiation can migrate in stage III.

The First Sino-Japanese Physical
Metallurgy Symposium, Beijing,
China, Nov. 8-11, 1984

Behaviour of Hydrogen in Iron Alloys

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Metals Research Institute, Faculty of Engineering,

Hokkaido University, Sapporo 060, Japan

To investigate the behavior of hydrogen in iron at high temperatures, electron-irradiation was carried out in a high voltage electron microscope after H^+ preinjection. In the H^+ injected iron interstitial type loops were observed at room temperature, however, fine vacancy type loops were developed during annealing at above 670 K and were shrunk by further electron irradiation. The fine defect clusters produced by the injection are inferred to the complex of hydrogen and vacancy. In the He^+ injected 316 steel, the nucleation of dislocation loops was enhanced. By the electron irradiation at high doses, void number density increased strongly in both H^+ and He^+ injected specimens. From these results, it is considered that hydrogen can interact with vacancies and enhance the nucleation of loop and void formation.

Third ASIA-Pacific Congr. on
Electron Microscopy, Singapore,
Aug. 24-Sept. 2, 1984

Instability of Precipitation in Ni-based Alloys under Irradiation by HVEM

Taro TAKEYAMA, Heishichiro TAKAHASHI and Hisashi OSANAI
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Hokkaido University, Sapporo 060, Japan

The instability and microstructural development of γ' in Ni-based alloys during electron irradiation were investigated. These alloys were previously heat-treated at 723-1073 K to obtain precipitations, and then irradiated by a high voltage electron microscope at 1000 kV in the temperature range of 723-823 K. In the case of Ni-11.5 at% Si alloy, by irradiating the alloy, plate-like precipitates were formed. The plate-like precipitates were identified as Ni_3Si nucleated as a result of Si-segregation at the dislocation loops during irradiation. The large pre-existing precipitates started to dissolve from the outside and small precipitates were renucleated near the dissolved γ' as the resolution proceeds. When the Ni-13.5 at% Al alloy containing Ni_3Al was irradiated, very small precipitates were newly nucleated with a high number density in the matrix, and they continued to grow with further irradiation, while the majority of large pre-existing precipitates began to be destroyed and finally lost their shapes, which suggests the resolution of the γ' . From the present study it was clarified that instability of precipitates may be strongly associated with the solute size and precipitate misfit effects. In Ni-Al alloy with positive misfit precipitates the solute could be diffusing away from the precipitate. In Ni-Si alloy with negative misfit precipitation vacancy-Si interaction may occur firstly at the interfaces of the precipitates.

Twelfth International Symposium
on Effects of Radiation on Materi-
als, Williamsburg, Va., USA, Jun
18-20, 1984

Solute Segregation and Void Formation on Grain Boundaries in Electron Irradiated 316 Stainless Steel

Soumei OHNUKI, Heishichiro TAKAHASHI and Taro TAKEYAMA
Metals Research Institute, Faculty of Engineering,
Hokkaido University, Sapporo 060, Japan

Radiation-induced segregation and void formation on grain boundaries in Ti-modified 316 steel were investigated by electron irradiation and EDX analysis. Strong enrichment and depletion of solutes were observed on grain boundaries during irradiation. Moreover, preferential void formation on grain boundaries occurred and grain boundary migration was enhanced by the irradiation. These facts indicate that grain boundaries act as preferential sinks for radiation-induced point defects, and the sink behavior may be changed by the development of the segregation under irradiation.

First International Conference on
Fusion Reactor Materials, Tokyo,
Japan, Dec. 3-6, 1984

Study of Cavity Formation in 316 Stainless Steels by Means of HVEM/Ion-Accelerator Dual Irradiation

Taro TAKEYAMA, Soumei OHNUKI and Heishichiro TAKAHASHI
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Hokkaido University, Sapporo 060, Japan

To investigate the effect of helium on the dislocation structural evolution and cavity formation during dual irradiation, a HVEM/ion-accelerator irradiation system was developed. This facility composed of 1300 kV HVEM and 300 keV ionaccelerator enables the in-situ observation of dual irradiation. The results of simple electron irradiation, dual irradiation and preinjected irradiation in the same grain were compared. On the dislocation loop structure, the presence of helium caused an increase in the number density and a decrease in the size. On the cavity formation, the increment of one order in the number density was confirmed, and the swelling of dual irradiation was larger than that of the simple electron irradiation. These results indicated that the helium injected in 316 steel during dual irradiation may enhance the cavity nucleation process without suppression of its growth.

First International Conference on
Fusion Reactor Materials, Tokyo,
Japan, Dec. 3-6, 1984

The Effect of Implanted Hydrogen on the Effect of Cluster Formation in Electron Irradiated Austenitic Stainless Steel

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Metals Research Institute, Faculty of Engineering,
Hokkaido University, Sapporo 060, Japan

To investigate the effect of hydrogen on the formation of dislocation loops and voids, electron-irradiation was carried out in a high voltage electron microscope after H⁺ preinjection. Fine defect clusters produced by H⁺ preinjection at room temperature rapidly disappeared during the low fluence of electron irradiation (<0.1dpa) at 670-770K, and by further irradiation, other defect loops developed. It is inferred that the fine defect clusters produced by the preinjection is a complex of hydrogen and vacancy. By the irradiation in a high dose, H⁺ preinjection increased also in void number density strongly. From these results, it may be considered that hydrogen in a high concentration will interact with vacancies and enhance the void nucleation.

First International Conference on
Fusion Reactor Materials, Tokyo,
Japan, Dec. 3-6, 1984

The Damage in Structural Behaviour by Electron Irradiation of Mn-Cr Austenitic Stainless Steels

Heishichiro TAKAHASHI*, Taro TEKEYAMA*, Kanji TANIKAWA* and Ritsu MIURA**

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**The Japan Steel Works, Ltd., Muroran 050, Japan

A simulation experiment by electron irradiation using a HVEM was carried out at a dose rate of about 2×10^{-3} dpa s^{-1} . This was done at a temperature of 723 K on high manganese-chromium austenitic steels with various solute concentrations, i. e., 19 wt% Mn, 16 wt% Cr, 0.1 wt% C and 0.03-5.9 wt% Ni. Voids formed in all specimens and their distribution was different depending on Ni solute concentration. For specimens with higher Ni concentration, void formation tended to be retarded, and the incubation irradiation dose for void nucleation became longer in comparison with the specimen with lower Ni concentration. The resultant void swelling at 13 dpa were 1.3% and 0.3% for the specimens with the lowest and the highest Ni contents, respectively. In addition to their void swelling observations we also observed the formation of new phases with strain contrast in the vicinity of the grain boundaries during irradiation. The results of the compositional analysis of these regions including the new phases showed segregation of Si and Ni, and depletion of Mn and Cr. From these results it is suggested that compositional change of solute due to segregation and/or depletion induces an austenite phase instability.

International Symposium on
Characterization of Heavy Crude
Oils and Petroleum Residues,
Lyon, France June. 25-27 1984

Characterization of Carbonization Reaction of Petroleum Residues by Means of High-Temperature ESR and Transferable Hydrogen

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Sapporo 060, Japan

Seiji SHIMOMURA* and Toshio IMAMURA*

*General Planning Department, Koa Oil Co., Ltd,
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It was attempted to characterize the carbonization reaction of petroleum residues through the measurements of high temperature ESR, hydrogen donor ability and optical texture of resultant cokes. Good correlations were found between hydrogen donor ability and change in spin concentration. Residues

forming cokes with large size of optical texture have a high ability as hydrogen donor and show low spin concentrations at high temperatures. Similar results were obtained also for some model compounds. A model for the mechanism of carbonization is proposed on the basis of these observations.

International Symposium on
Characterization of Heavy Crude
Oils and Petroleum Residues,
Lyon, France June. 25-27 1984

Enhancement of Hydrogen Transfer in Petroleum Residue with Catalysts during Carbonization

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The influence of the addition of metal oxide catalysts on transferable hydrogen in petroleum residue was studied by $^1\text{H-NMR}$ technique with a particular focus on the mesophase development. Solid catalysts such as $\text{SiO}_2\text{-Al}_2\text{O}_3$, $\text{MoO}_3\text{-TiO}_2$ and $\beta\text{-Al}_2\text{O}_3$ decreased the transferable hydrogen from Wafra vacuum residue on heating up to 673K and reduced the size of optical texture of the resultant cokes.

On the other hand, SeO_2 markedly revealed the promotion of hydrogen transfer in vacuum residues and considerably enlarged the size of optical texture. The catalytic activity in vacuum residue/ D_2O systems over catalyst was investigated by D-NMR technique. Deuterium incorporation of D_2O into petroleum residue in the region of aliphatic hydrogens was recognized with SeO_2 catalyst, whereas exchange in the aromatic region was noted for $\text{SiO}_2\text{-Al}_2\text{O}_3$ catalyst. Transferable hydrogen associated with the skeleton of hydrocarbons is one of the keys controlling the carbonization reaction of petroleum residues and pitches.

International Carbon Conference
"Carbon 84", Bordeaux, France
July. 2-6 1984

High-Pressure, High-Temperature ESR in the Early Stage of Carbonization of Pitch and Coal

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High temperature, high pressure e. s. r. measurements of the hydrogenation reaction of Taiheiyo coal in the presence of catalysts were carried out to understand the stabilization of thermally and/or catalytically induced free radicals.

A decrease in free radical concentration with the increasing temperature was observed for $ZnCl_2$ and $SnCl_2 \cdot 2H_2O$ catalysts at 10 MPa under hydrogen gas. High pressure modified single-cell d. t. a. and p. d. a. equipment augmented the uniquely designed high temperature, high pressure e. s. r. cell. The hydrogenation reaction was monitored under the same experimental conditions as e. s. r. From the results of the combination of high temperature, high pressure e. s. r. with high pressure d. t. a. and p. d. a., it was established that H_2 molecules can react efficiently with free radicals from coal molecules created by the presence of $ZnCl_2$ and $SnCl_2 \cdot 2H_2O$ catalysts.

International Carbon Conference
"Carbon 84", Bordeaux, France
July. 2-6 1984

Carbonization Behaviour of Pitch in the Presence of Inert Material

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The influence of silica gel ($<45\mu$ m) as an inert additive on the carbonization reactions of A240 pitch was investigated by optical microscopy, measurements of transferable hydrogen, high-temperature e. s. r. and high-temperature 1H n. m. r. Additions of silica gel have the effect of reducing the size of the optical texture of the mesophase which appears in the early stages of carbonization. The higher the concentration of silica gel, the smaller is the amount of transferable hydrogen. The spin concentration of pitch increases with the increasing silica gel content. Measured values of $\Delta H_{1/2}$, using high-temperature 1H n. m. r., suggest that the molecular motions in the pitch become restricted on addition of silica gel. The effect of

silica gel on the physical properties and chemical reactivity of carbonizing pitch is discussed.

17th Biennial Conference on Carbon, Lexington, Kentucky, U. S.
A. June. 16-21 1985

Investigation of the Early Stage of Carbonization for Petroleum Pitch By Means of High-Temperature ¹H-NMR and ESR

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The early stage of carbonization of petroleum pitches was investigated by an in-situ high-temperature ¹H-NMR and an ESR spin probe. Well resolved ¹H-NMR spectra throughout the pyrolysis of pitches provided detailed information about mesophase formation. The ESR spin probe method also provided useful information about mesophase generation during the cooling process.

17th Biennial Conference on Carbon, Lexington, Kentucky, U. S.
A. June. 16-21 1985

Coal Evaluation Method Based on Compatibility of Coal and Pitch

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A new evaluation method for coking coals is proposed. The distribution of optical textures of coke modified with coal tar pitch were obtained by an automatic measuring system based upon image analysis. Hydrogen donor and acceptor abilities were also estimated. Coals are evaluated in terms of the extent of the development of anisotropic textures.

17th Biennial Conference on Carbon, Lexington, Kentucky, U. S.
A. June. 16-21 1985

Development of Anisotropic Texture in Co-Carbonization of Low Rank Coal and Pitch

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Sapporo 060, Japan

The extent of development of anisotropic texture in coke made from various coals or blends with pitch in co-carbonization systems was evaluated in terms of the hydrogen donor (D) and acceptor (A) abilities of coals, pitches and for various blending ratios of coal-pitch systems.

Gieseler plastometry and high temperature $^1\text{H-NMR}$ were also used to obtain information regarding the mobility in the co-carbonization systems. A good correlation was found between the extent of development of anisotropic texture, solidification temperature and the value of D/A for single or blend systems.

2nd China-Japan Symposium on Fluidization, Kunming, China April 10-15 1985

Prediction of Solid Mixing in Liquid Fluidized Beds of Binary Solid Particles

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When a bed of size-and density-different binary particles are fluidized by a liquid in laminar regime, it stratifies vertically into two layers ; the upper layer consisting of almost a pure component while the lower of essentially complete mixture of both components. In this paper a mathematical model to predict the composition of the lower mixing layer is developed on the basis of momentum equations for each component of particles. The voidage function involved in the equations is independently given from a unit cell model which assumes a creeping flow around the particle within the fictitious spherical cell and a potential flow outside the cell. The applicability of the unit cell model is confirmed by comparison with observed expansion characteristics of monocomponent beds. A segregation map is illustrated which gives the composition of any given binary solid and in turn predicts the occurrence of the layer inversions.