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Contents Lists and Abstracts from the Journal of the Adhesion Society of Japan

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The Cross-linking Reaction of a Thermoplastic Polyester Using a Radical Initiator

Keiichi UNO

Toyobo Research Center 1-1, Katata, 2-chome, Ohtsu, Shiga, 520-02 Japan

Abstract

A saturated, thermoplastic polyester can be made to cross-link by heating with organic peroxides such as benzoyl peroxide or dicumyl peroxide.

As a model reaction of a saturated, thermoplastic polyester and a radical initiator, various types of dibenzoates of glycol and dimethyl esters of long-chain aliphatic dicarboxylic acid were made to react with dicumyl peroxide at high temperatures.

The comparative reactivity of H-abstraction in the components of polyesters was estimated by measuring quantities of 2-phenyl-2-propanol and acetophenone formed under the reaction of the model compound with dicumylperoxide.

It was found that hydrogens of methylene further from the β -position to -0 or -C0 were easily abstracted using the cumyloxy radical.

Based on these results, the mechanism of the cross-linking reaction utilizing a radical initiator in a saturated and thermoplastic polyester was discussed.

(Received: November 26, 1988)

ABSTRACTS J. ADHESION SOCY. JAPAN

Hot Melt Adhesion between Different Polymer-adherends by the Blended Polymers Filled with Super Fine Fillers

Shunji OHNISHI

Ohnishi Laboratory 3-15-14 Ninomiya, Tsukuba-city, 305, Japan

Abstract

Possibility and strength of adhesion between the two different materials of polymer A and B as adherend by the blended polymers of A and B and the blended polymers filled with super fillers as adhesive have been investigated. As polymer, HDPE, LDPE, PP, PS, PMMA have been selected and as a filler, ferite powder (Fe₂O₃) with particle size 0.5 μ m and alumina powder (Al₂O₃) with particle size 0.4 and 0.01 μ m have been used. As adhesives, the blended mixture of two kind of polymer as non-filled type and of two kind of polymer and filler as filled type which filling ratio are 3.85 vol.% have been used for melt process at 200 ~ 240°C and strength of adhesion have been evaluated by tensile strength.

By using blended polymer of non-filled or filled type as adhesive, possibility of adhesion have been recognized on the combination of adherend in specific range of polymer mixing ratio (=PMR) of PS-HDPE: PS-PDP: HDPE-PMMA: HDPE-PP, but not recognized on PP-PMMA. By using filled type, generally PMR have spreaded and strength have enhanced at specific range more than non-filled type, for instance, on PMR 20 vol.% of HDPE-PS, strength have been $40 \sim 50 \text{ Kg/cm}^2$.

(Received: December 17, 1988)

Generalization of Tearing Energy*

Yoshio MIKI

Production Engineering Laboratory Nitto Denko Corporation 18 Hirayama, Nakahara, Toyohashi, Aichi, 441-31, Japan

Abstract

The tearing energy by Rivlin is defined only for the elastic materials under conditions of constant displacement. The concept of tearing energy is generalized in this paper by defining the generalized tearing energy \mathcal{T} as the mechanical energy release rate itself, which was defined in the previous paper. As a result, it is shown theoretically that \mathcal{T} is independent of shape of test-pieces and test methods, and that \mathcal{T} depends on test rate and has a critical value as the lowest limit.

It is clarified that Rivlin's method is applicable to any materials for form's sake if the total deformation work done is used instead of the strain energy density.

It is pointed out that the existing fracture mechanics are premised on the hypothesis of brittle fracture condition. We deduce the relation between the tearing energy and Rice's *J* integral and show how to calculate without the above hypothesis.

(Received: June 5, 1989)

• A Study of Mechanics of Adhesion (VI)

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The Syntheses and Cross-linking of a Polyethyleneterephthalate (PET) Copolyester

Keiichi UNO

Toyobo Research Center 1-1, Katata, 2-chome, Ohtsu, Shiga, 520-02 Japan

Abstract

A new photosensitive monomer, N,N'-Bis (hydroxy-ethyl) benzophenone tetracarboxylic acid diimide (HEB-TI), which is polymerized with ethyleneterephthalate in a molten state, is synthesized, identified and polymerized with ethyleneglycol and terephthalic acid.

A copolymerized PET with $1 \sim 5 \text{ molar}\%$ of HEBTI is formed into a film utilizing an extrusion method similar to that of conventional PET.

The copolymerized PET containing HEBTI units can, under solidstate, develop certain interchainlinking reactions through irradiation with ultra-violet light or with an electron beam, resulting in a cross-linking between the molecules or an increase in the molecular weight.

It was investigated and ascertained that the molar% of HEBTI, the volume of irradiation energy, the temperature of the films undergoing irradiation, and the effective wave lengths of irradiation could affect the efficiency of the interchain-linking reactions.

(Received: December 11, 1988)

Effect of The Temperature of Polymer Substrate at Vacuum Deposition on The Adhesion of Metal Thin Film on Polymer Film*

Satoshi TANIGAWA, Yoshinori IMOTO, Kenichi AZUMA, Katsuhiko NAKAMAE and Tsunetaka MATSUMOTO

> Department of Industrial Chemistry, Faculty of Engineering Kobe University Rokkodai-cho, Nada-ku, Kobe-shi, 657 Japan

* Studies on Recording Magnetic Materials and Magnetic Composites XXV.

Abstract

Metal thin films were prepared on polymer films by vacuum deposition technique at various substrate temperature. The effect of the substrate temperature at vacuum deposition on the adhesivity of the deposited metal thin films on the polymer films was investigated. The following results were obtained.

Adhesion of the deposited metal thin film on polymer film changed drastically at a particular temperature of polymer substrate at vacuum deposition. This temperature was different in each polymer substrate. The change of the adhesion at various temperature of substrate was examined from the chemical or physical properties of polymer films. The substrate temperature at which the maximum adhesion was obtained is consistent with the glass transition temperature of that polymer substrate. The magnetic characteristics (coercivity etc.) of the cobalt thin films which deposited at the substrate temperature of glass transition of polymer films greatly improved.

(Received: June 24, 1989)

Permeabilities of Composite Membranes Containing Polyvinyl Alcohol and Polyvinyl Esters

Toyoji TSUCHIHARA, Chiharu KASAI, Masaru YAMAGUCHI and Kazuhiko MATSUZAWA

Faculty of Engineering, Yokohama National University 156, Tokiwadai, Hodogaya-ku, Yokohama-shi 240, Japan

Abstract

Asymmetric composite membranes were prepared through mixing of hydrophilic and hydrophobic polymers. Crosslinked polyvinyl alcohol was used as hydrophilic polymer, while polyvinyl esters of various aliphatic side chains were used as hydrophobic polymers.

Ultrafiltration through the membranes containing various mole ratios of hydrophilic and hydrophobic polymers were examined, and permeabilities of water and solutes of molecular weights ranged in $1000 \sim 10000$ were studied.

The composite membranes containing polyvinyl esters were found to have good water permeability, but low solute permeation selectivity.

(Received: July 31, 1989)

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The Cross-linking of a Polyethyleneterephthalate (PET) Copolyester and the Properties of Its Cross-linked Film

Keiichi UNO

Toyobo Research Center 1-1, Katata, 2-chome, Ohtsu, Shiga, 520-02 Japan

Abstract

Cross-linked films of polyethyleneterephthalate (PET) were produced by the UV-irradiation of films from PET copolymerized with $1 \sim 5 \text{ molar}\%$ of a new photosensitive monomer, N,N'-Bis-(hydroxyethyl) benzophenone tetracarboxylic acid diimide (HEBTI) and the properties of the cross-linked films were studied.

The crystallization of PET was inhibited enormously by the cross-linking. Using this behavior, I estimated the true melting point based upon the inherent structure of the film. That is, the films which were heat-treated under various conditions were measured on a Differential Scanning Calorimeter (DSC) after cross-linking the film to inhibit melting and restructuring it while undergoing DSC measurement.

The glass transition temperature (Tg), the resistance against thermal deterioration, the thermaldimensional stability, and the folding endurance of the cross-linked film were enhanced in comparison with conventional non-cross-linked PET film.

(Received: December 10, 1988)

Adsorption Behavior of Silane-Coupling-Agent-Containing Polymers on $\gamma\text{-Fe}_2O_3$ and the Properties of Magnetic Coatings*

Katsuhiko NAKAMAE, Satoshi TANIGAWA and Takeo KITAYAMA

Department of Industrial Chemistry, Faculty of Engineering, Kobe University 1, Rokkodai-cho, Nada-ku, Kobe, 657 Japan

Abstract

Copolymers consisted of γ -methacryloxypropyltrimethoxysilane (γ -MPS) and methyl methacrylate (MMA) [P(MMA- γ -MPS)] were synthesized as a model binder for magnetic tapes. The adsorption behavior of such polymers on γ -Fe₂O₃ particles were investigated. The dispersion of these particles in the magnetic tapes and mechanical properties of the tapes were also examined. (1) The adsorption behavior of P (MMA- γ -MPS) on γ -Fe₂O₃ was Langmuir type adsorption. The saturated adsorbance increased with increase in γ -MPS content in the polymers, and kept constant over 0.5 mol% of γ -MPS

Studies on Recording Magnetic Materials and Magnetic Composites XXVI.

content. (2) Methanol which was produced by the hydrolysis reaction of methoxy group in γ -MPS molecule was detected by gas-chromatography. (3) Using P (MMA- γ -MPS) as a magnetic binder, the dispersion of magnetic particles and the mechanical property of magnetic films were improved. (Received: June 24, 1989)

SN-ratio Estimation of Optimum Test Conditions of Rolling Ball Tack Method of PSA Tape

Zenichi MIYAGI, Kazuosa KUMA and Kentaro YAMAMOTO

Department of Precision Engineering, Meiji University 1-1-1 Higashi-mita, Tama-ku, Kawasaki-shi 214, Japan

Abstract

Optimum test conditions of rolling ball tack test of ASTM type are determined in conformity to initial rolling rate, calculated from initial position of the rolling ball, and diameter of the ball by the aid of SN-ratio. A new testing apparatus is developed to improve accuracy of determination of stopping distance free from parallax error by the aid of plane mirror, by which the sighting needle, the ball and their images can be seen through, and the calculated initial rolling rate is enlarged up to 2000 mm/s.

Stopping distance of five kinds of double-coated tapes on the market are tested under the following test conditions; 14/32, 20/32, 26/32, and 32/32 inch in ball diameter, and from 100 to 1400 mm/s in calculated initial rolling rate in 100 mm/s intervals.

The results of SN-ratio analysis are summarized as follows:

and Constanting of Cold Parts

1) SN-ratio increases as initial rolling rate increases, but the ratio becomes constant when the rate is greater than 900 mm/s.

2) Maximum and minimum SN-ratio appear at 26/32 and 14/32 inch in diameter of rolling ball respectively, and their difference is 8.69 db.

3) When the rolling ball tack test is applied for quality control, as is recommended in the ASTM standards, it is advisable to adopt a rolling ball of 26/32 inch in diameter and calculated initial rolling rate of 900 mm/s or more.

(Received: July 10, 1989)

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The Syntheses and Cross-linking of Self Cross-linkable Polyesters Containing Oxy-rings

Keiichi UNO

Toyobo Research Center 1-1, Katata, 2-Chome, Ohtsu, Shiga, 520-02 Japan

Abstract

New monomers containing both an oxy-ring and functional groups capable of polyesterification were synthesized and copolymerized with other glycols and acids to make polyesters with a pendent oxy-ring such as an oxirane-ring, an oxetane-ring, or a tetrahydrofuran-ring.

Polyesters containing an oxy-ring were made to cross-link by themselves by heating in the presence of catalysts.

The reactivity of the self cross-linking reaction of polyesters containing various oxy-rings decreases in the following order.

oxetane > tetrahydrofuran >> oxirane

According to model reactions, it was ascertained that the oxetane-ring can readily react with a carboxylic group in the presence of sodium phenoxide and that the tetrahydrofuran-ring can readily react with a hydroxy group in the presence of acid anhydrides. From these results, the self cross-linking reaction of polyesters containing an oxy-ring is assumed to take place through the biting of the pendent oxy-ring by the carboxylic or the hydroxy end-groups of polyesters.

(Received: December 14, 1988)

Application of Macromonomer for the Pressure Sensitive Adhesives I. Adhesive and Surface properties

Katsuhiko NAKAMAE, Tatsuo SATO and Tsunetaka MATSUMOTO

Faculty of Engineering, Kobe University, Rokkoudai-cho, Nada-ku, Kobe-shi 657, Japan

Abstract

Adhesive and surface-chemical properties of poly (polystyrene macromer-buthyl acrylate) copolymers, poly (styrene-polybutyl acrylate) copolymers and poly (styrene-butyl acrylate) random copolymers were investigated. The results are as follows;

(1) The adhesive properties of the copolymers which were prepared by macromer-technique were different from the random copolymers.

(2) It was found that there were differences in the surface and molecular structure between the copolymers which were prepared by macromer-technique and the random copolymers by the results about surface free energy of copolymers and interfacial tension of water/toluene in the presence of copolymers.

(3) The reason why there were differences in the surface and molecular structure seemed that the copolymers which were prepared by macromer-technique had graft chains and had different inner-structure from the random copolymers. Therefore it was suggested that the graft copolymers which were prepared by macromer-technique had the phase-separated structure.

(Received: June 24, 1989)

Surface Analysis for Microwave Plasma Treated Fluororesins by ESCA

Tomoyuki KASEMURA, Yoshikatu OKADA and Masayuki FUJII

Department of Applied Chemistry, Faculty of Engineering, Gifu University 1-1 Yanagido Gifu-shi 501-11 Japan

Abstract

Three fluoreresins (PDVF, PTFE and FEP) were treated with the microwave plasmas which were generated by an electronic cooking range and the surfaces of treated resins were analyzed with ESCA.

The large signal of O_{1_s} at 533 ev and the broad continuous spectrum of C_{1_s} from 295 to 285 ev appeared in the ESCA spectrums for plasma treated resins. The amount of oxygen, which was introduced by the treatment, increased with the lowering of gas pressure in the plasma treating glass vessel. It was shown that the resin was treated more effectively by the lower treating gas pressure.

In order to obtain the depth profiles, two techniques were employed: (i) argon ion etching and (ii) by varying the electron take-off angle (θ) .

i) The oxygen introduced by plasma treatment decreased rapidly with the going on of the ablation caused by the etching.

ii) For the FEP immediately after the treatment, the amount of oxygen increased with the decreasing of θ (with the shallowing of the analyzable depth) and fluorine decreased. After leaving the treated FEP in the desiccator for 70 hours, the amount of fluorine increased in the region of $15^{\circ} < \theta < 60^{\circ}$ and oxygen decreased, since the fluorine-containing segments, which had a low surface free energy, migrated to the surface of the resin.

From the depth profiles, it was found that the plasma treatment reached to a half of the depth which could be analyzed by ESCA at $\theta = 90^{\circ}$.

(Received: July 20, 1989)