

# **MATERIALS SCIENCE AND TECHNOLOGY NEWSLETTER**

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## **FOCUSING ON POLYIMIDES, ADHESION, CONTACT ANGLE AND PARTICLES**

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## EDITORIAL COMMENTS

As the Fall season is upon us the attention of the NEWSLETTER must now focus on two upcoming symposia on **POLYIMIDES AND HIGH TEMPERATURE POLYMERS** and **ADHESION ASPECTS OF THIN FILMS** which will be held in Orlando Florida the week of November 5, 2007. In addition, we need to announce two new symposia coming up in the Summer of 2008 on **CONTACT ANGLE** and **PARTICLES ON SURFACES**.

Though this seems to be quite a range of topics they are all in fact intimately related by the simple fact that they depend critically on surface phenomena. The polyimide materials for instance are commonly used as coatings especially in the electronics industry where they are favored due to their high temperature and chemical stability as well as their excellent insulator properties. However, in order to be useful they must adhere properly to a number of other materials including metals, silicon, ceramic and glass. Adhesion is of course a surface phenomenon and is one of the main reasons the polyimides have been chosen over other materials as insulators. From my own experience in the microelectronics industry I know that we considered a number of high temperature polymers to be used as thin film insulators. Many of these materials had superior thermal stability and electrical properties than the polyimides, but invariably one of the main reasons they were not chosen was because of their inability to adhere properly to a number of critical substrate materials. The adhesion and contact angle topics need no further discussion as their dependence of surface phenomena is transparent. Particles on surfaces are also clearly affected by the surface forces acting between the particles and the surface they reside on. The whole problem of removing particles from surfaces is clearly dependent on understanding the nature of these surface interactions.

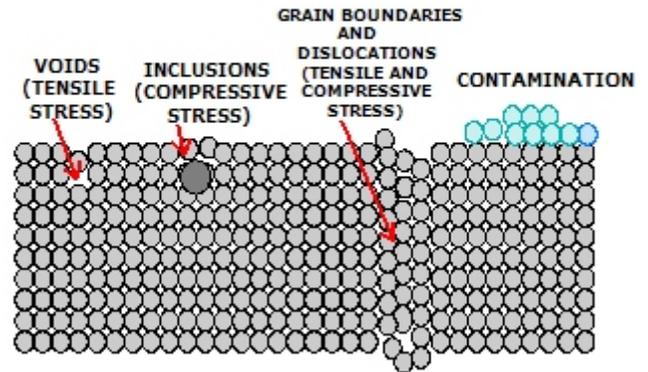
Thus, speaking of interrelated surface phenomena, the subject of this issue's essay is a paper that involves nearly all of the topics mentioned above except that the polymers involved were not of the high temperature variety but rather more prosaic engineering thermoplastics. However, the methodologies employed could have been equally well applied to the polyimides or any other polymer for that matter.

## IN SEARCH OF THE TRUE SURFACE ENERGY

### Problematic Nature of Solid State Surface Energies

Measuring the surface energy of a solid has to be

A VARIETY OF DEFECT STRUCTURES AND IMPERFECTIONS MAKE DETERMINING THE SURFACE ENERGY/SURFACE TENSION SOLIDS VERY DIFFICULT



**Figure 1** Sample surface defects of a solid that affect the surface energy.

one of the most challenging problems of surface science and the one topic where there is little agreement on what the true surface energy of any particular solid might be. Table 1 gives a listing of the most common methods for measuring the surface tension of liquids and solids. What is directly noticeable is that there are nearly twice as many methods for measuring the surface tension for liquids as opposed to solids. What the table does not show, however, is that whereas there is a general consensus that the methods used for liquids are reliable and accurate there is little such a consensus for those methods in the solids column.

The situation has been summed up rather succinctly by Adamson and Gast<sup>1</sup>

*"There is a rather limited number of methods for obtaining experimental surface energy and free energy values, and many of them are peculiar to special solids or situations. The only general procedure is the rather empirical one of estimating a solid surface tension from that of the liquid"*

The basic problem comes down to the fact that whereas liquids retain a high degree of mobility at the atomic and molecular level solids do not.

<sup>1</sup> "Physical Chemistry of Surfaces: Sixth Edition", Arthur W. Adamson and Alice P. Gast (John Wiley & Sons, Inc., New York, 1997) p. 278

## Surface Force Apparatus as a Direct Approach for Surface Energy Measurements

Given the unencouraging situation, it is interesting to note that there are advanced methods for looking directly at the surface energetics of solids. In particular, I would like to call the reader's attention to what I feel is a seminal piece of work on the surface energies of poly(ethylene terephthalate) and polyethylene by Mangipudi, Tirrell and Pocius<sup>2</sup> hereafter referred to as MTP. These authors decided to in effect "take the bull by the horns" and directly measure the surface energy of their samples by using the Surface Force Apparatus or SFA. The basic experiment first came to light some 38 years ago out of the pioneering work of Tabor and Winterton<sup>3</sup> and was further refined to what is essentially its modern configuration by Israelachvili and Tabor<sup>4 5</sup> a short while later.

Figure 2 gives a schematic representation of the SFA apparatus. Everything about this device approaches the limits of machine metrology. Using interferometric methods the gap spacing can be measured to a resolution approaching 0.3 nm and using a carefully fabricated cantilever forces can be detected with a sensitivity of 10 nano Newton. Further details on this remarkable apparatus can be found in the volume by Kendall<sup>6</sup>. For our present purposes we only need to note that MPT decided that the Surface Force Apparatus would be a very effective way of getting at the

<sup>2</sup> "Direct Measurement of Molecular Level Adhesion between Poly(ethylene terephthalate) and Polyethylene Films: Determination of Surface and Interfacial Energies", V. Mangipudi, M. Tirrell and A. V. Pocius, in Fundamentals of Adhesion and Interfaces, Ed. D. S. Rimai, L. P. DeMejo and K. L. Mittal (VSP, Utrecht, The Netherlands, 1995) p. 205.

<sup>3</sup> D. Tabor and R. H. S. Winterton, Proc. R. Soc. A **312**, 435 (1969).

<sup>4</sup> J. N. Israelachvili and D. Tabor, Nature, **236**, 106 (1972).

<sup>5</sup> J. N. Israelachvili and D. Tabor, Proc. R. Soc. A **331**, 19 (1972).

<sup>6</sup> "Molecular Adhesion and its Applications", Kevin Kendall (Kluwer Academic/Plenum Publishers, New York, 2001) See chapter 4 p. 63 for a most engaging account of the history and development of the SFA apparatus.

**TABLE 1: Methods for measuring the surface tension (surface free energy) of solids and liquids\***

LIQUIDS	SOLIDS
Capillary rise method	Contact angle method
Drop shape analysis	Work of cleaving method
Drop weight method	Skapski method
Oscillating jet method	Grain boundary groove analysis
Pulsating bubble method	-
Ring method	-
Wilhelmy slide method	-
*Data from: "Physical Chemistry of Surfaces: Sixth Edition", Arthur W. Adamson and Alice P. Gast (John Wiley & Sons, Inc., New York, 1997)	

Thus a given liquid has the ability to spontaneously reorganize at the surface to minimize the total surface free energy thus giving rise to a well defined surface configuration which, to all intents and purposes, is smooth down to nearly the atomic level. Thus the surface free energy or surface tension of any liquid is well defined and amenable to precise measurement. The surface configuration of a typical solid is quite another matter being highly dependent on the thermal-mechanical history that lead to its formation. Nearly every detail of the solidification process comes into play, whether solidification was rapid or slow, what mechanical loading conditions were active during and after the solidification process, what impurities were present and what was their precise concentration and spatial distribution, ... etc. Thus any given surface can have a range of defects and structures which affect its surface free energy. A sample of some of the most common imperfections is displayed in figure 1. In particular voids and inclusions can induce local tensile and compressive stress fields that will alter the local surface tension and if wide spread the total surface energy. Grain boundaries emerging at the surface have roughly the same effect. Thus all the details of a polycrystalline solid come into play. Finally the presence of contaminant layers will disguise the underlying surface and thus make the measurement of the true surface energy nearly impossible even if only a monolayer is present.

surface energies of polymer films. However this is rather easier said than done since there is a lot of work in sample preparation. MPT decided to look at films of polyethylene and poly(ethylene terephthalate). Films of the two polymers on the order of 3 to 5 micrometers thick were obtained by extrusion methods and then silvered on one side so as to create a reflection surface for the interferometric measurements. The films then had to be carefully mounted onto near perfect quartz cylindrical lenses using an adhesive. Even the choice of adhesive proved to be very fussy since certain adhesives gave rise to viscoelastic effects which distorted the surface force measurements. For further details the reader should consult the original MPT paper.<sup>2</sup>

Since the surface force apparatus measures the force required to separate the surfaces in the absence of any applied compressive load we still need to relate this measured force to the surface energy holding the surfaces together. For this MPT turned to the JKR theory of surface forces which gives this force in terms of the radius of curvature of the contacting surfaces  $R$  and the work of adhesion between the two surfaces  $W$  as:

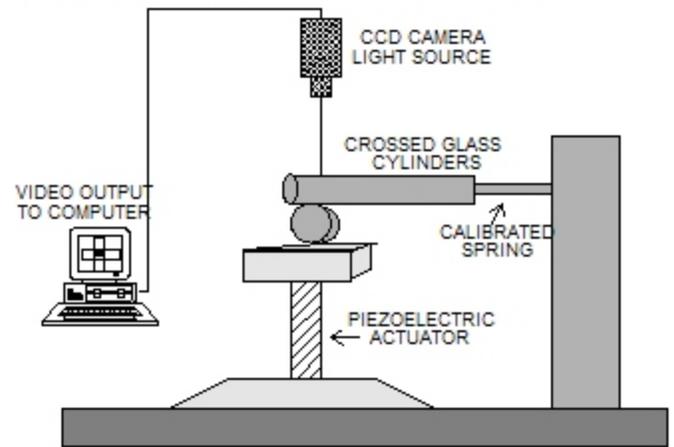
$$P_s = \frac{3\pi WR}{2} \quad (1)$$

In addition the JKR theory makes a number of predictions concerning the details of the deformations incurred in the contact region as shown in figure 3. These predictions could now be verified directly using the SFA apparatus. The first test shown in figure 4 demonstrates that the energy required to separate the surfaces is independent of the time that the surfaces remain in contact before being separated. This rules out the presence of any possible interdiffusional behavior of the polymer surfaces which could cause the separation force to increase dramatically. In addition, the data shown in figure 5 further show that the separation energy does not depend appreciably on the rate of separation for the relatively slow separation rates used in the experiment. Finally, figure 6 demonstrates that the JKR theory can accurately predict the details of the surface deformation occurring in the contact region.

### Comparison with Contact Angle Method

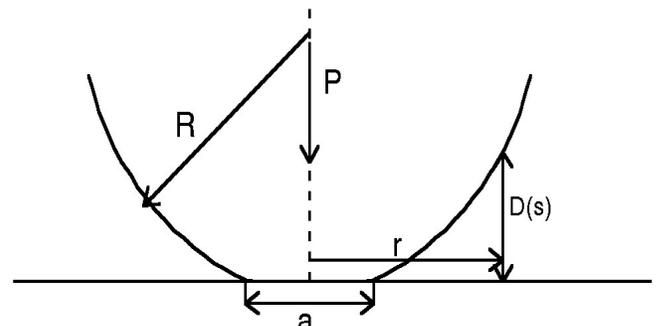
With confidence in the surface force measurements now established MPT then went on to measure the surface energies of their samples using the contact angle method which was essentially the only alternate method available

### SCHEMATIC OF SURFACE FORCE APPARATUS



**Figure 2:** Apparatus for sensitively measuring the force of attraction between two crossed cylinders. The gap between the cylinders is delicately adjusted by a piezo electric actuator, the force is determined by monitoring the deflection of a carefully calibrated spring and the gap between the cylinders is monitored by interferometry.

### JKR CONTACT MECHANICS



$$a^3 = (R/K)[P + 3\pi WR + (6\pi WR P + (3\pi WR)^2)^{1/2}]$$

$$D(s) = (a^2/\pi R)[(s^2 - 2 + (4/3)(a_0/a)^{3/2})\tan^{-1}(s^2 - 1)^{1/2}]$$

$$s = r/a \quad K = (2E)/(3(1-\nu^2))$$

$a_0$  = Contact diameter at  $P = 0$

$E$  = Young's Modulus;  $\nu$  = Poisson's ratio

**Figure 3** Schematic of the load - deformation behavior of a spherical surface in contact with a rigid substrate according to the Johnson, Kendall, Roberts (JKR) theory.

given the nature of the samples being used. However, whereas making contact angle measurements is significantly less sophisticated and difficult than performing surface force experiments there arises nonetheless the thorny problem of interpreting the data in order to determine the solid surface energy. The problem of interpreting contact angle data was discussed in some detail in a previous issue of the newsletter

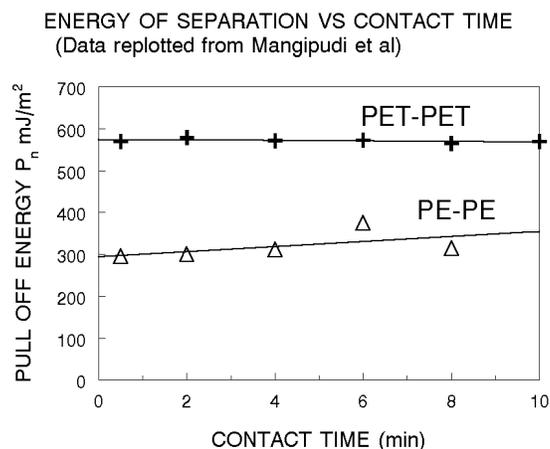
([www.mstconf.com/Vol2No3-2005.pdf](http://www.mstconf.com/Vol2No3-2005.pdf)) For current purposes, however, there are at least 4 separate theories which attack this problem which we list in roughly chronological order:

1. **Zisman method:** Earliest pioneering attempt at estimating the solid surface energy by extrapolation to zero contact angle which implies complete wetting.
2. **Good, Girifalco, Fowkes, Young equation:** Attempted improvement over Zisman method which uses the geometric mean approximation to estimate the solid-liquid surface energy from the liquid-vapor and solid-vapor energies.
3. **van Oss, Choudhury, Good approach:** Seeks to improve on Good, Girifalco, Fowkes, Young by explicitly recognizing the diverse nature of the intermolecular forces present in terms of dispersion forces, acid-base interactions and hydrogen bonding.
4. **Equation of State Theory:** Seeks a wholly different interpretation based on liquid state thermodynamics.

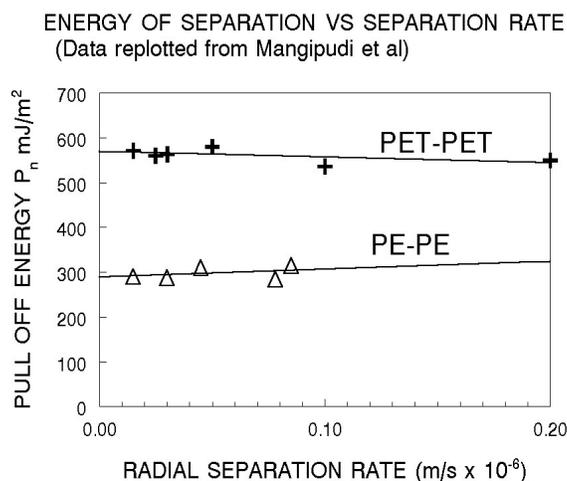
It is well known that all of the above theories have limitations and in addition the complications discussed above in terms of imperfections in nearly all solid surfaces will affect all of the above theories more or less equally. As a rule, assuming well characterized surfaces, all of the above theories will prove adequate if only short range dispersion forces are dominant. Thus, since MPT's samples are very well characterized and should not be dominated by any complex interactions, there should be a reasonable chance for getting agreement between the SFA measurements and contact angle data.

<b>TABLE 2:</b> Comparison of surface energies for PET and PE polymers measured by the SFA apparatus and contact angle methods. Data averaged from Ref. 2		
POLYMER	SFA MEASUREMENT (mJ/m <sup>2</sup> )	CONTACT ANGLE MEASUREMENTS (mJ/m <sup>2</sup> )
PET	61.2	41.4
PE	33.3	35.9
PE-PET	77.3	-

MPT performed contact angle measurements on both samples using 13 different liquids and



**Figure 4** Plot of separation energy vs time remaining in physical contact for poly(ethylene terephthalate) PET and polyethylene PE films as measured by the SFA apparatus.



**Figure 5** Same as figure 4 except that now the rate of separation is the independent variable.

interpreted their data using 3 of the above mentioned theoretical procedures. The results are given in table 2.

In table 2 the contact angle data are for advancing angle measurements only and are averaged between results derived from theories 2 and 4 above. The results for PET are clearly disappointing and no doubt reflect the more complex surface chemistry of this polymer which apparently confounds the contact angle measurements. For PE however, the results can be considered near exact by surface measurement standards. This no doubt reflects the fact that PE is the simplest polymer known from a chemistry standpoint. The third entry in table 2 is the surface interaction energy of PE and PET which is

listed as an example of the power of this direct measurement method. Contact angle measurements could possibly be used to estimate this interaction using polymer melts but it would be devilishly tricky and if the results in table 2 are anything to go by it is unlikely that interpreting the data would be very straightforward.

So where does all this leave us. The results for PE clearly indicate that if the surfaces are carefully prepared and well characterized and involve only the simplest forces as described by the classical van der Waals interactions then contact angle data can give highly accurate results which are comparable to direct measurements. On the other hand if the surface forces become more complex then all bets are off. Thus the search for the true surface energy of a solid is within sight but still very much on the distant horizon.

### **POLYIMIDES AND ADHESION ASPECTS OF THIN FILMS: PROGRAMS FOR ORLANDO FLORIDA, NOVEMBER 5-9, 2007**

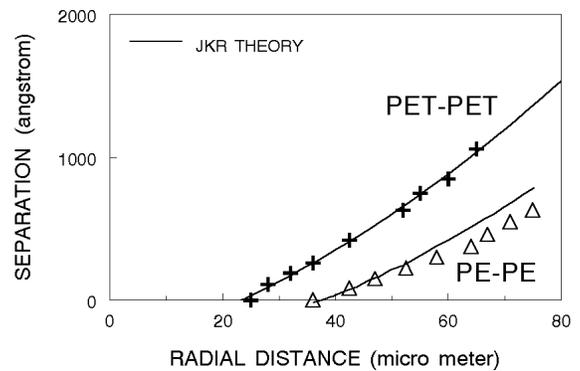
We are happy to announce the preliminary programs for the upcoming November symposia on Polyimides and Adhesion Aspects of Thin Films coming up in November.

#### **PRELIMINARY PROGRAM FIFTH INTERNATIONAL SYMPOSIUM ON POLYIMIDES AND OTHER HIGH TEMPERATURE POLYMERS SYNTHESIS, CHARACTERIZATION AND APPLICATIONS**

To be held November 5-7, 2007 in Orlando, Florida USA

This symposium is the fifth in a series the first of which was held in Newark, NJ in 1999. As with its predecessors, this symposium will be concerned with all aspects of polyimides and other high temperature polymers. These materials have found applications in such diverse areas as the aerospace industry and microelectronic components. A unique combination of physical and chemical properties makes these materials highly attractive for demanding applications where chemical inertness, high temperature stability, low dielectric constant, mechanical toughness and processability are primary concerns. This symposium is organized to bring together scientists, technologists and engineers interested in all aspects of high temperature polymers, to review and assess the current state of knowledge, to provide a forum for exchange and cross-fertilization of ideas, and to define problem areas which need intensified efforts. The invited

CONTACT SEPARATION PROFILE  
(Data replotted from Mangipudi et al)



**Figure 6** Cylinder deformation profile near contact region. Comparison of SFA measurements and JKR theory. Refer to formula for deformation profile  $D(s)$  given in figure 3.

speakers have been selected so as to represent widely differing disciplines and interests, and they hail from academic, governmental and industrial research laboratories. This meeting is planned to be a truly international event both in geographic coverage as well as in spirit. The technical program will contain both invited overviews and contributed original research papers. It is planned to chronicle the transactions in a hard-bound volume of archival quality (to match or exceed the standards of the journal literature) which will serve as a reference work for future generations of investigators. The following is a **PARTIAL** list of papers to be presented.

### **CHARACTERIZATION AND DURABILITY STUDIES**

**Thomas Bahners**, Uwe Schloßer and Eckhard Schollmeyer; Deutsches Textilforschungszentrum 47798 Krefeld, GERMANY; **Some Aspects of Aging of High-Performance Fibers and Lifetime Prediction and Enhancement**

**Thuy. D. Dang** and N. Venkatasubramanian; AFRL, Bldg 654, 2941 Hobson Way, Wright-Patterson Air Force Base, OH 45433; **Rigid-rod Polybenzimidazoles (PBIs): A Review of Their Synthesis, Properties, Processing and Applications**

**Satoru Iwamori** and Akihiro Uemura; Kanazawa University, Kanazawa City 920-1192, JAPAN; **Polymer Thin Films Sputtered with Polyimide Targets**

**Satoru Iwamori** and Masaaki Mizoguchi; Kanazawa University, Kakuma-machi, Kanazawa City, 920-1192, JAPAN; **Adhesion of Poly(tetrafluoroethylene) (PTFE) and Poly(vinyl alcohol) (PVA) Composite Thin Films on the Nickel-Titanium Substrates**

**Rong-Ho Lee**, Hsun-Lien Lin, Shenghong A. Dai, and Ru-Jong Jeng; National Yunlin University of Science & Technology, TAIWAN; **Thermally Stable NLO Poly(amide-imide)s via Acylurea Intermediates**

**J K Quamara**, Maneesha Garg and Geetika Goyal; National Institute of Technology, Kurukshetra-136119, INDIA; **Multiple Relaxation Processes in Swift Heavy Ion Irradiated Kapton-H Polyimide**

**M. Sarojadevi** and S. Bhuvana; Department of Chemistry, Anna University, Chennai - 600025, INDIA; **Structure Property Relationship of Processable Heat Resistant Poly (Amide-imide-imides) for High Temperature Applications**

## ELECTRONIC APPLICATIONS

**Wai Kin Chan**; The University of Hong Kong, Pokfulam Road, Hong Kong, CHINA; **Heterocyclic Aromatic Conjugated Polymers For Opto-Electronic Applications**

**Kuni Cherenack** and S. Wagner; Department of Electrical Engineering, Princeton University, Princeton, NJ 08544; **Developing a 300C Fabrication Process on Clear Plastic**

Xiaojuan Zhao, Shiyong Yang and **Lin Fan**; Institute of Chemistry, Chinese Academy of Sciences, Zhongguancun, Beijing 100080, CHINA; **Synthesis and Characterization of Multifluorinated Aromatic Polyimides for Photonic Applications**

Ryousuke Tamura, Eunju Lim, Shuhei Yoshita, Takaaki Manaka and **Mitsumasa Iwamoto**; Department of Physical Electronics, Tokyo Institute of Technology, Tokyo 152-8552, JAPAN; **Organic Field Effect Transistor as a Maxwell Effect Element Using Polyimide and Polyvinylidene Poly(vinylidene fluoride) Films**

**Tomonori Minegishi** and Noriyuki Yamazaki; Hitachi Chemical Co., Ltd., 13-1 Higashi-cho 4-chome, Hitachi-shi, Ibaraki 317-8555 JAPAN; **A Novel Photodefinable Poly(benzoxazole) Using A Negative-working Chemical Amplification System**

Hongjun Zuo, Jiansheng Chen, Lin Fan and **Shiyang Yang**; Chinese Academy of Sciences, Zhongguancun, Beijing 100080, CHINA; **Aromatic Polyimide Resins for High Density Packaging Substrates**

**Bao-Ku Zhu** and You-Yi Xu; Institute of Polymer Science, Zhejiang University, Hangzhou Yuquan 310027, P. R. CHINA; **Preparation of Ultra-low Dielectric Constant Polyimide Foam via Phase Inversion Process**

## NANO COMPOSITES, BLENDS AND NOVEL APPLICATIONS

**Zongwu Bai**, Shane B. Juhl, Narayanan Venkat, and Thuy D. Dang; University of Dayton Research Institute, University of Dayton, Dayton, OH 45469; **High Performance SPBI/SPTES Composite Proton Exchange Membranes for H<sub>2</sub>/O<sub>2</sub> Fuel Cell Applications**

Wan-Jung Chou and **Chuh-Yung Chen**; Department of Chemical Engineering, National Cheng-Kung University, Tainan 70101, TAIWAN; **Characteristic of Polyimide-based Nanocomposites Containing Nano-scale Filler**

Ying Du, **Guo-Dong Dang**, Hong-Wei Zhou and Chun-Hai Chen, Department of Chemistry, Alan G. Macdiarmid Institute, Jilin University, Changchun, P. R. CHINA, 130012; **New Kind of Carbon-Fiber-Reinforced Phenylethynyl-Terminated Polyimide Composites**

**Anne Jonquière**s, Michel Awkal, Robert Clément, and Pierre Lochon; Laboratoire de Chimie Physique Macromoléculaire, Nancy Université, 54 001 NANCY cedex, FRANCE; **New Ion-containing Polyimides for the Purification of One of the Most Promising Bio-fuels by a Membrane Separation Process**

**Benalia Kouini**; University of Boumerdes, LRME, Boumerdes 35000, ALGERIA; **The Effect of Nanoclays on the Properties and Morphology of Injection Molded Polypropylene/ Polyamide 66 Nanocomposites**

**Elvira T. Krutsko**, Tamara A. Zharskaj and Nicolai R. Prokopchuk Belarussian State Technological University, Department of Polymers, BELARUS; **Polyimide Blends**

**R. Misiego**, C. Cano and R.B. Pipes; School of Chemical Engineering, Purdue University, West Lafayette, IN; **Polyimide Foam Nanocomposites from Powder Precursors**

**Morton Litt**; Department of Macromolecular Science, Case Western Reserve University, Cleveland, OH 44106; **Rigid Rod Polymers with Frozen in Free Volume: Application to Polyelectrolyte Membranes**

**Evgeniya Sanzhieva**, Fedosya Kalinina, Larisa Radnaeva, Vladislav Borovik, Oleg Shkurko, Dmitry Mogonov; Buryat State University, Smolina st., 24a, Ulan-Ude, RUSSIA; **Novel Pyrimidine Containing Polyimide and Films for Fuel Cells**

**M. Sarojadevi**, L. S. Jayakumari, V. Thulasiraman, G. Anuradha and P. S. Sampath; Department of Chemistry, Anna University, Chennai - 600025, INDIA; **Structural Characterization and Mechanical and Thermal Properties of New Cyanate/ Epoxy and Cyanate/BMI Blends and Composites**

**M. Sarojadevi**, P. Selvakumar and K. Padmini; Department of Chemistry, Anna University, Chennai - 600025, INDIA; **Phthalocyanine Terminated Polyimides for High Temperature Composites Applications**

#### **NOVEL SYNTHESIS AND STRUCTURE-PROPERTY RELATIONSHIPS**

**Susanta Banerjee**; Materials Science Centre, Indian Institute of Technology, Kharagpur - 721302, INDIA; **Synthesis of Novel Semifluorinated Poly(ether imide)s and Structure-Property Relationship**

Meruert Nurakhmetova, Maria Abilova, Victor Solomin, Valentina Kravtsova and **Rinat Iskakov**; Kazakh British Technical University, 55 Tolebi Avenue, 050000, Almaty, KAZAKHSTAN; **A New Heat-resistant Alicyclic Polyimide on the Basis of Simple Oil-Refined Hydrocarbons**

**Ying-Ling Liu** and Shi-Yih Chen; Department of Chemical Engineering, Chung Yuan Christian University, Chung-Li, Tao-Yuan 32023, TAIWAN; **Temperature-Responsive Polyimides from Diels-Alder Reactions**

**Brigitte Mutel**; Laboratoire GÉPIFRÉM - Bât. C5, Cité Scientifique, 59655 Villeneuve d'Ascq cedex, FRANCE; **Thermochemical Behaviour of Organosilicon Films Elaborated by a Cold Plasma Polymerization Process**

**H. S. Patel** and S.N.Desai; Department of Chemistry, Sardar Patel University Vallabh Vidhyanagar - 388120, Gujarat - INDIA; **Novel Polyimide System Based on Nitro Displacement / Diels-Alder Reaction - III**

**M. Sarojadevi**, R. Hariharan and N. Amutha; Department of Chemistry, Anna University, Chennai - 600025, INDIA; **Synthesis and Properties of Novel Bismaleimides, Polyaspartimides and Organosoluble Polyimides**

**Saqiba Sheerazi**; Quaid-e-Azam University, Islamabad, PAKISTAN; **Synthesis and Characterization of Polyimide-Epoxy Hybrid Films**

**Dennis W. Smith, Jr.**; Department of Chemistry, Clemson University, Clemson, SC 29634; **Semi-Fluorinated Polymers Derived from Aromatic Trifluorovinyl Ethers**

**Yusuke Tsuda**; Department of Biochemistry & Applied Chemistry, Kurume National College of Technology, Kurume, Komorino 1-1-1, Fukuoka 830-8555 JAPAN; **Soluble Polyimides Based on Aromatic Diamines Bearing Long-Chain Alkyl Groups**

#### **AEROSPACE APPLICATIONS**

**Christine Allen**; NASA Goddard Space Flight Center, Greenbelt, MD 20771; **Application of Polyimide Microwires for Thermal Isolation of a Cryogenic Transistor Module**

**Theo J. Dingemans**, Erik S. Weiser and Terry L. St.Clair; NASA Langley Research Center, AMPB, Hampton, VA 23681-0001; **Novel All-Aromatic Liquid Crystal Polyesters and Polyetherimides for Aerospace Applications**

**Sara R. Halper**, Shabnam Virji and Randy M. Villahermosa; Space Materials Laboratory, The Aerospace Corporation, El Segundo, CA; **Metal-containing Polyimides for Contamination Sensing and Prevention in Space Applications**

**Ching-Hsuan Lin**; Department of Chemical Engineering, National Chung Hsing University, Taichung, TAIWAN; **Polyimides for Potential Space Applications**

Martha K. Williams, **Trent M. Smith**, James E. Fesmire, and Erik S. Weiser; Kennedy Space Center, FL 32899; **Polyimide Foam Composites for Enhanced Thermal and Acoustical Insulation**

**Sandra J. Tomczak**, Michael E. Wright, Vandana Vij, Timothy K. Minton, Amy L. Brunsvold, Brian J. Petteys, Andrew J. Guenther, Gregory R. Yandek, and Joseph M. Mabry; AFRL/PRSM, Materials Applications Branch, Air Force Research Laboratory, 10 E. Saturn Blvd, Bldg. 8451, Edwards AFB, CA 93524; **Space-Survivability of Main-Chain and Side-Chain POSS-Kapton® Polyimides**

**Shabnam Virji**, Sarah Halper, Judy Ying, Rika Anderson, and Randy Villahermosa; The Aerospace Corporation, Space Materials Laboratory, 2350 E. El Segundo Blvd., M2-248, El Segundo, CA 90245; **Zeolite Modified Polyimides as Absorption Materials for Molecular Contamination**

**Kristopher E. Wise**, Dennis C. Working, Jae-Woo Kim, Peter Lillehei, Sharon Lowther, Cheol Park, Godfrey Sauti, and Emilie J. Siochi; National Institute of Aerospace, Hampton, VA 23666; **Polymer Nanocomposites for Aerospace Applications**

**Dezhen Wu**; Collage of Materials Science and Engineering; Beijing University of Chemical Technology, Beijing 100029, CHINA; **Preparation of Highly Reflective And Conductive PI/Ag Composite Films by Ion-exchange and Self-Metallization Techniques**

**Kumiko Yokota**, Masahito Tagawa, Eiji Miyazaki, Mineo Suzuki, Minoru Iwata, and Rikio Yokota; Department of Mechanical Engineering, Faculty of Engineering, Kobe University, Kobe 657-8501 JAPAN; **Durability of a Silicon-Containing Polyimide in a Simulated Low Earth Orbit Space Environment**

**Masahito Tagawa** and Kumiko Yokota; Department of Mechanical Engineering, Faculty of Engineering, Kobe University, Kobe 657-8501 JAPAN; **Fluorination of PMDA-ODA Polyimide using Hyperthermal Atomic Fluorine Beams**

**D. G. Abreu**, T. Debies, A. Entenberg and G. A. Takacs; Chemistry, Center for Materials Science and Engineering, School of Physical Sciences, Rochester Institute of Technology, Rochester, 14623 NY; **Modification of Fluoropolymer Surfaces to Contain Copper Fluoride: Relevance to Adhesion**

## **PRELIMINARY PROGRAM**

### **THIRD INTERNATIONAL SYMPOSIUM ON ADHESION ASPECTS OF THIN FILMS (INCLUDING ADHESION MEASUREMENT AND METALLIZED PLASTICS)**

To be held November 7-9, 2007 in Orlando, Florida, USA

This symposium is the third in a series dealing with adhesion aspects of thin films, adhesion measurement and metallized plastics. The first symposium with this title was held in Orlando, FL in 2003 with the intent of integrating key aspects of three separate symposia which treated these topics singly in the past. The main idea was to provide a broader venue for the discussion and exploration of these three closely related fields of endeavor. The main part of the symposium focuses on those aspects of thin film technology that have a direct bearing on film adhesion to the substrate. This is a topic of both fundamental interest to all aspects of thin film technology and of great practical concern in applications where films of high stress are involved. The coating of diamond films onto machine tools is one of many applications where thin film adhesion is a critical factor in coating durability. The second part of the symposium will deal with the ability to accurately measure the adhesion of coatings to surfaces. This is always a crucial part of development and manufacturing processes dealing with coatings and films. Finally, metallized plastics are a burgeoning technology heavily dependent on thin film adhesion with applications ranging from decorative design to optical coatings to advanced thin film wiring schemes in the microelectronics industry. Metallized plastic films allow the technologist to capitalize on the favorable properties of two disparate classes of materials to create new and unique products which transcend the performance and usefulness that can be obtained by either class alone.

The invited speakers have been selected so as to represent widely differing disciplines and interests, and they hail from academic, governmental and industrial research laboratories. The following is a **PARTIAL** list of papers to be presented.

**Robert C. Cammarata**; Department of Materials Science and Engineering, Johns Hopkins University, 102 Maryland Hall, 3400 North Charles Street, Baltimore, MD 21218-2689; **Recent Advances in the Theory and Measurement of Thin Film Stress**

**Alexander Fedorov**, Willem-Pier Vellinga and Jeff De Hosson; Department of Applied Physics, Netherlands Institute for Metals Research, University of Groningen, Nijenborgh 4, 9747 AG Groningen, THE NETHERLANDS; **Degradation of Adhesion in Deformed Polymer-metal Interfaces Studied by Laser Induced Delamination Technique**

**J. Friedrich**, R. Mix, A. Meyer-Plath, S. Wettmarshausen, H. Sturm; Bundesanstalt für Materialforschung, und Prufung, Unter den Eichen 87, D-12200 Berlin, GERMANY; **A new Concept of Adhesion Promotion in Metal-Polymer Composites by Introduction of Covalently Bonded Hydrophobic Spacers at Interface**

**J. Friedrich**; Bundesanstalt für Materialforschung, und Prufung, Unter den Eichen 87, D-12200 Berlin, GERMANY; **New Plasma Techniques for Polymer Surface Modification with Monotype Functional Groups**

**M. Ignat**, C. Malhaire, G. Ravel and E. Quesnel; SIMAP INP Grenoble, FRANCE; **Cracking and Deadhesion of Thin Metal Films on Mechanically Modified Polymer Surfaces**

Meruert Nurakhmetova, Maria Abilova, Victor Solomin, Valentina Kravtsova and **Rinat Iskakov**; Kazakh British Technical University, 55 Tolebi Avenue, 050000, Almaty, KAZAKHSTAN; **A New Heat-resistant Alicyclic Polyimide on the Basis of Simple Oil-Refined Hydrocarbons**

**Satoru Iwamori** and Masaaki Mizoguchi; Graduate school of natural science & Technology, Kanazawa University, Kakumamachi, Kanazawa city 920-1192, JAPAN ; **Adhesion and Mechanical Properties of Poly(tetrafluoroethylene) Poly(vinyl Alcohol) Composite Thin Films Prepared by Spin Coat Method**

**K. S. Kim**; Department of Mechanics, Division of Engineering, Box D, Brown University, Providence, RI 02912; **Nano-scale Solid-interface Strength Measured by Hybrid Method of Nano-testing and Finite Element Analysis**

**Seok-Keun Koh**, Jun-Sik Cho, Sung Han, Ki-Hwan Kim, Younggun Han, Junghwan Lee, ChulSoo Lee, Jinwoo Seok and Jaeho Joo; R&D Center, P&I Corporation, Shinnae Technotown #405, 485 Sangbong-Dong, , Seoul, KOREA; **Industrialization and New Applications in Ion Assisted Reaction**

**Noboru Kyouno**; RHESCA Co. Ltd., 15-17, Hino-Honmachi 1 Chome, 191-0011, JAPAN; **Application of FFT-based Signal Analysis to Micro-Scratch Testing for Adhesion Strength Measurement of Thin Films and Measured Results Based on Different Measuring Parameters**

**Brigitte Mutel**; Laboratoire GÉPIFRÉM, Bâtiment C5, Université des Sciences et Technologies de Lille, 59655 Villeneuve d'Ascq cedex, FRANCE; **Polymer Functionalization and Thin Film Deposition by Cold Remote Nitrogen Plasma Process**

**R. J. Narayan**; Department of Biomedical Engineering, University of North Carolina, Campus, Box 7575, Chapel Hill, NC, USA 27599-7575; **Biofouling-resistant Materials for Medical Applications**

**Young-Bae Park**, Sung-Cheol Park, Su-Hwan Cho, Hyun-Cheol Jung, Jae-Woo Joung, Kyu-Hwan Lee; School of Materials Science and Engineering, Andong National University, KOREA; **Interfacial Adhesion of Inkjet-Printed Ag on Flexible Polyimide Substrate**

E. T. Krut'ko, T.A. Zharskaya and **I.N. Prokopchuk**; Byelorussian State Technological University; **A New Method of Obtaining Epoxy Waterproof Coatings**

**Brian W. Sheldon**; Division of Engineering, Brown University, Providence, RI 02912; **Grain Boundary Induced Stresses in Polycrystalline Coatings and Thin Films**

**Willem-Pier Vellinga**, Alexander Fedorov, Jeff De Hosson Department of Applied Physics, Netherlands Institute of Metals Research, University of Groningen, Nijenborgh 4, 9747 AG, Groningen, THE NETHERLANDS: **Residual stress and delamination front geometry on polymer-metal interfaces**

**H. Willeck**, W. Eberhardt , H. Kueck; Hahn-Schickard-Institute of Microassembly Technology HSG-IMAT, Stuttgart, GERMANY; **A New Device for Testing the Adhesion of Conductors on Polymers**

## **SUMMER 2008 SYMPOSIA ON CONTACT ANGLE WETTABILITY AND ADHESION**

We are happy to announce the continuation of two symposia that have always attracted much attention in the past and are still as popular as ever. The contact angle measurement procedure has to be by far one of the oldest methods of experimental science dating back nearly to the inception of the scientific method in the 17<sup>th</sup> century with the work of Francis Hauksbee who performed some of the earliest observations and experiments in 1709. These were repeated in 1718 by James Jurin who observed that the height of fluid in a capillary column was a function only of the cross-sectional area at the surface, not of any other dimensions of the column.

Later Thomas Young set out in descriptive terms the principles governing the contact between fluids but never actually wrote down his famous equation in his 1804 paper "An Essay on the Cohesion of Fluids". Pierre Simon Laplace followed this up in *Mécanique Céleste* with a formal mathematical description which reproduced in symbolic terms the relationship described earlier by Young. Laplace accepted the idea propounded by Hauksbee in the *Philosophical Transactions* for 1709, that the phenomenon was due to short range forces. The part which deals with the action of a solid on a liquid and the mutual action of two liquids was not worked out thoroughly, but ultimately was completed by Gauss.

Particle contamination technology on the other hand only really started to garner scientific interest toward the mid 20<sup>th</sup> century with the advent of the microelectronics industry. Specifically as the 1970's rolled around and the drive for large scale integration began in earnest. As the electronics industry started to cram more and more circuitry onto a small square of silicon, it was realized that particles on the order of the diameter of a human hair in size could wreak havoc on microcircuits and decimate the yield attainable from a silicon wafer. From that time the need to control particle contamination became a holy crusade since the entire economics of the microelectronics industry depended on steadily improving the yield of good chips that could be garnered from a single wafer. The need to control ever smaller particle contamination has only grown more urgent as manufacturers have shrunk the feature size of transistors down to the level of 40 nanometers. Furthermore, the need to understand particle interactions with surfaces has expanded well

outside the microelectronics industry to such applications as Xerography where controlling the interactions of toner particles is a key concern. In addition, the pharmaceutical industry has found particles to be very useful as drug delivery packages and again the interaction between the particle and the drug molecule is a controlling factor in this technology. All of these issues are apropos to the Particle symposium and we expect papers to be presented across the entire spectrum of applications.

### **CALL FOR PAPERS** SIXTH INTERNATIONAL SYMPOSIUM ON **CONTACT ANGLE, WETTABILITY AND** **ADHESION**

To be held at the University of Maine, Orono, Maine USA, July 14-16, 2008

In his opening remarks at the first symposium in this series Professor Robert Good pointed out that Galileo in the 17<sup>th</sup> century was quite likely the first investigator to observe contact angle behavior with his experiment of floating a thin gold leaf on top of a water surface. Since that time contact angle measurements have found wide application as a method for determining the energetics of surfaces. This, in turn, has a profound effect on the wettability and adhesion of liquids and coatings to surfaces.

We are indeed happy to announce that this the 6<sup>th</sup> symposium in the series will be organized in collaboration with Prof. Douglas Gardner in the Advanced Engineered Wood Composites Center at the University of Maine, Orono, Maine. Prof. Gardner is well acquainted with problems of wettability and adhesion having published on the *Dynamic Wettability of Wood* and also serving on the editorial board of the *Journal of Adhesion Science and Technology* which is edited by the Conference Director Dr. Mittal. This symposium will be concerned with both the fundamental and applied aspects of contact angle measurements. Issues such as the applicability and validity of various measurement techniques and the proper theoretical framework for the analysis of contact angle data will be of prime concern. In addition, a host of applications of the contact angle technique will be explored including but not limited to: wettability of powders, fibers, wood products, papers, polymers and monolayers. Further focus will be on the use of contact angle data in evaluating surface modification procedures, determining relevance of wettability to adhesion, the role of wettability in bioadhesion, ophthalmology, prosthesis and in the control of dust in mining and milling applications. The primary focus of this

symposium will be to provide a forum for the discussion of cutting edge advancements in the field and to review and consolidate the accomplishments which have been achieved thus far.

## **SYMPOSIUM TOPICS:**

### **Factors Influencing Contact Angle Measurements:**

- ◆ Static and dynamic contact angles, effect of surface flaws and surface roughness on wetting.
- ◆ Effect of pore size distribution
- ◆ Effects of velocity and viscosity of liquid on solid-liquid interfacial behavior.
- ◆ Interaction forces including: van der Waals, Acid-Base, Hydrogen bonding, ...etc

### **Wettability Behavior and Surface Characterization of Various Materials:**

- ◆ Contact angle interpretation and hysteresis.
- ◆ Wettability of various material surfaces including but not limited to: wood, elastomers, silicon wafers, pharmaceutical powders, metals, polymers, paper, particles, fibers... etc.
- ◆ Surface treatments to modify wettability behavior.
- ◆ Patterned surfaces
- ◆ Superhydrophobic effect

### **Wettability, Adhesion and Applied Aspects of Contact Angle Measurements:**

- ◆ Effect of surface energetics on adhesion.
- ◆ Biological applications including protein and bacterial adhesion.
- ◆ Fine particle adhesion and control of dust.
- ◆ Other technological applications including: printing, agriculture, pharmaceuticals, textiles and paper.

## **CALL FOR PAPERS**

### **ELEVENTH INTERNATIONAL SYMPOSIUM ON PARTICLES ON SURFACES: DETECTION, ADHESION AND REMOVAL; University of Maine, Orono, Maine USA, July 16-18, 2008**

This will be the eleventh event in the series of symposia on particles on surfaces initiated as part of the Fine Particle Society meeting in 1986. Particles are yield detractors in the manufacture of sophisticated and sensitive electronic components and are very undesirable in many other technologies. Contamination of optical

surfaces and shorting of microelectronic circuits by conducting particles, among other concerns, underscore the importance of particle detection, adhesion and removal. On the other hand, however, in certain instances particle adhesion to surfaces is necessary. The purpose of this symposium is to address the vast ramifications of particles on solid surfaces by bringing together specialists in many allied fields to discuss their latest findings and to identify areas for further investigation. Various types of substrates and particles --metals, oxides, glass, and polymers-- will be covered. The technical program will comprise both invited and contributed papers ranging from topical overviews to original research and industrial applications.

## **SYMPOSIUM TOPICS:**

- ▶ **Sources and mechanisms of particle contamination**
  - ▶ Intrinsic and extrinsic
  - ▶ Nanometer to micrometer scale
- ▶ **Factors that influence particle adhesion:**
  - ▶ chemistry, topography, shape, size, relative humidity, medium, etc.
- ▶ **Particle adhesion measurement techniques**
  - ▶ Forces affecting adhesion: JKR theory, Hamaker theory
- ▶ **Detection, identification and characterization of particles on surfaces**
  - ▶ Micrometer scale
  - ▶ Nanometer scale
- ▶ **Techniques for particle removal**
  - ▶ Challenge of nanoscale removal
  - ▶ Fluid dynamics of particle removal
- ▶ **Implications of particle contamination**
  - ▶ Microelectronic applications
  - ▶ Biomedical applications
  - ▶ Optics and precision tool applications
- ▶ **Thermodynamics of particle removal** including interactions with fluids, electrolytes and solvents
- ▶ **Detection/Removal of bacteria/viruses considered as particles**

These symposia is being organized under the direction of Dr. K. L. Mittal, Editor, Journal of Adhesion Science and Technology by MST Conferences, LLC. A proceedings volume is planned for each and further details will be provided in due course. Please notify the conference chairman of your intentions to present a paper as early as possible. An abstract of about 200 words should be sent by **January 15, 2008** to the conference chairman by any of the following methods:

E-mail: [rhl@mstconf.com](mailto:rhl@mstconf.com)

FAX: 212-656-1016

Regular mail:

Dr. Robert H. Lacombe  
Conference Chairman  
3 Hammer Drive  
Hopewell Junction, NY 12533

Contact by phone:

845-897-1654

845-227-7026

Full conference details and registration via the Internet will be maintained on our web site:

Details of the **CONTACT ANGLE** symposium will be maintained at:

<http://mstconf.com/contact6.htm>

Details of the **PARTICLE** symposium will be maintained at:

<http://mstconf.com/particle11.htm>

## REGISTRATION INFORMATION

### **NOVEMBER 5-7, 2007: FIFTH INTERNATIONAL SYMPOSIUM ON POLYIMIDES AND OTHER HIGH TEMPERATURE POLYMERS**

### **NOVEMBER 7-9, 2007: THIRD INTERNATIONAL SYMPOSIUM ON ADHESION ASPECTS OF THIN FILMS (INCLUDING ADHESION MEASUREMENT AND METALLIZED PLASTICS)**

### **NOVEMBER 10, 2007: SHORT COURSE ON ADHESION MEASUREMENT METHODS**

#### **LOCATION:**

Regal Sun Resort Hotel  
1850 Hotel Plaza Blvd.  
P.O. Box 22202  
Lake Buena Vista, FL 32830-2202  
Tel: Reservations (USA, Toll free): 1-800-624-4109  
Tel: (Overseas): 1-407-842-1029  
FAX: 1-407-828-6547  
Web site: ([www.regalsunresort.com](http://www.regalsunresort.com))

#### **REGISTRATION:**

Speaker/student \$395 each; regular attendee \$595 each. A 20% discount applies if you are also attending both symposia. An additional 10% discount applies if more than 1 person is participating from the same organization.

**HOTEL:** Please make room reservations directly with the Regal Sun Resort. A block of rooms has been set aside for conference registrants until October 5, 2007. After this date the hotel will accept reservations on a space available basis and they cannot guarantee that the special conference rates of \$89 per day will apply. Make your reservations early and be sure to mention that you are attending one of the MST symposia in order to receive the reduced conference hotel rate.

#### **TO REGISTER FOR SYMPOSIA:**

BY PHONE: 845-897-1654 or 845-227-7026  
BY FAX: 212-656-1016 or 845-897-2361  
E-mail: [rhlacombe@compuserve.com](mailto:rhlacombe@compuserve.com)

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MST Conferences  
3 Hammer Drive  
Hopewell Junction, NY 12533-6124, USA

## SHORT COURSE ON ADHESION MEASUREMENT TECHNIQUES, November 10, 2007:

Associated with its symposia MST gives a short course on adhesion measurement methods. Since nearly all of the symposia have some relation to adhesion phenomena, the ability to quantify the adhesion of one material layer to another is clearly one of the unifying themes. This course is designed to mesh with the topical symposia by presenting an overview of the most useful adhesion measurement techniques which are being used to evaluate the **PRACTICAL ADHESION** of coatings and laminate structures. Emphasis will be given to methods which can be carried out in a manufacturing environment as well as in the lab and which give results that are directly relevant to the durability and performance of the coatings. The effects of material elastic properties and residual stress are considered as well as other external influences which affect coating adhesion.

**Audience:** Scientists and professional staff in R&D, manufacturing, processing, quality control/reliability involved with adhesion aspects of coatings or laminate structures.

**Level:** Introduction and technical overview

**Prerequisites:** General background in chemistry, physics or materials science.

**Duration:** 1 day

**Registration fee:** \$595: Includes complete set of lecture notes and a copy of **ADHESION MEASUREMENT METHODS: THEORY AND PRACTICE** (CRC PRESS, 2006).

#### **How You Will Benefit From This Course:**

**Understand** advantages and disadvantages of a range of adhesion measurement techniques.

**Gain insight** into mechanics of adhesion testing and the role of sample intrinsic stress and material properties.

**Learn optimal methods** for setting adhesion strength requirements for coating applications.

**Learn how to select** the best measurement technique for a given application.

**Gain perspective** from detailed discussion of actual case studies of product manufacturing and development problems.

**CANCELLATIONS:** Registration fees are refundable, subject to a 15% service charge, if cancellation is made by **Oct. 5, 2007**. **NO** refunds will be given after that date. All cancellations must be in writing. Substitutions from the same organization may be made at any time without penalty. MST Conferences reserves the right to cancel either of the symposia or the short course if it deems this necessary and will, in such event, make a full refund of the registration

## REGISTRATION FORM

<b>POLYIMIDES AND OTHER HIGH TEMPERATURE POLYMERS</b> November 5-7, (speaker/student)	\$395
<b>POLYIMIDES AND OTHER HIGH TEMPERATURE POLYMERS</b> November 5-7, (regular attendee)	\$595
<b>ADHESION ASPECTS OF THIN FILMS</b> , November 7-9 (speaker/student)	\$395
<b>ADHESION ASPECTS OF THIN FILMS</b> , November 7-9 (regular attendee)	\$595
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Deduct 20% if you are attending both symposia. Deduct additional 10% if more than 1 participant from same institution.	
Short Course on <u>Applied Adhesion Measurement Methods</u> : November 10, 2007	\$595
<b>TOTAL REGISTRATION FEE</b>	

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