25th Short Course on Polymer Characterization

in conjunction with 25th POLYCHAR 2017, Kuala Lumpur

Theme: **Polymer characterization**

Date: **Monday, 9th October 2017**

Time: **8.15 am to 5.15 pm**

Venue: **Putra World Trade Centre, Kuala Lumpur, Malaysia**

Synopsis

The Course on polymer characterization has been developed for students, researchers and industry practitioners from around the world to update their knowledge. The lectures shall provide the understanding of the methods of characterization of polymers and polymer-based materials (PBMs) as well as the basic science behind those methods. Thought-provoking insights into the experimental design and the research results will be presented. This will provide a basis also for understanding research reports during the following days of the Forum. The instructors might share their power point slides on the website of the IUPAC sub-committee on Polymer Education accessible to the general public.

Who should attend

Students, researchers on PBMs and engineers in relevant manufacturing industries.

Fees for 1-day Short Course on Polymer Characterization

1. Complimentary for the participants of 25th POLYCHAR **OR**
2. RM400 / USD100 for solely attending 1-day Short Course on Polymer Characterization only

Payment mode

Please refer to this web link
http:// www.25POLYCHAR.org.my

Tentative program

The short course is a set of lectures by experts in the field describing different aspects of interest to the participants of 25th POLYCHAR 2017. The following topics will be presented:

Time	Lecture	Speaker
8.00 am – 8.30 am	Registration	
8.30 am – 9.15 am	Electrochemical characterization of polymer electrolytes	Chin Han Chan
9.15 am – 10.00 am	Thermal analysis used to analyze the glass transition phenomenon	Jean-Marc Saiter
10.00 am – 10.15 am	Tea break	
10.15 am – 11.00 am	Viscoelastic properties of polymers	Michael Hess
11.00 am – 11.45 am	Simple teaching for mathematical treatments about diffraction and scattering of X-ray and visible light beams	Masura Matsuo
11.45 am – 12.30 pm	Determination of thermodynamic quantities by scattering techniques	Volker Abetz
12.30 pm – 2.00 pm	Lunch	
2.00 pm – 2.45 pm	Complex branched glucose polymers and human health	Robert G. Gilbert
2.45 pm – 3.15 pm	Polymer tribology	Witold Brostow
3.15 pm – 4.00 pm	Micromechanics of polymers: Electron microscopic methods of investigation	Sven Henning
4.00 pm – 4.15 pm	Teak break	
4.15 pm – 4.45 pm	Characterization of crosslinks in vulcanised rubbers: From simple to advanced techniques	Aik Hwee Eng
4.45 pm – 5.00 pm	Closing of short course	

For more information, please contact

Organization Secretariat

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under the auspices of
the Scientific Committee of World Forum on Advance Materials

MORE with IUPAC Polymer Education

Abstracts

Chin Han Chan and Hans-Werner Kammer

Authors present a phenomenological view on dielectric relaxation in polymer electrolytes, which is monitored by Electrochemical Impedance Spectroscopy. Molecular interaction of polymer chains with salt molecules (or dipole-dipole interaction between segments and salt molecules) leads to polar molecular entities. Frequency-dependant impedance spectra are the key quantities of the interest for determination of electric properties of materials and their interfaces with conducting electrodes. Salt concentration serves as parameter. Bulk and interfacial properties of the samples are discussed in terms of impedance (Z^*) and modulus (M^*) spectra. We focus on two different classes of systems, *i.e.* high molar mass of poly(ethylene oxide) (PEO) + salt and epoxidized natural rubber (ENR) + salt. Impedance spectra with salt content as parameter tell us that we have interaction between dipolar entities leading to dispersion of relaxation times. However as scaling relations show, dispersion of relaxation times does not depend on salt content in the PEO system. The relaxation peak for the imaginary part of electric modulus (M'') provides information on long-range motion of dipoles. Summarizing the results from imaginary part of impedance spectrum (Z''), tanδ (imaginary/real of permittivities) and M'' for the two systems under the discussion, PEO behaves like a mixture of chains with dipoles. There are interactions between the dipoles, but they are relaxing individually. Therefore, we see PEO-salt system as a polymer electrolyte where only a tiny fraction of added salt molecules becomes electrically active in promoting conductance. However, ENR-salt system behaves just as a macroscopic dipole and it can't display electrode polarization or electric relaxation because there is no mobility of individual dipoles. Hence, ENR-salt does not form a polymer electrolyte in the classic sense.

Volker Abetz

Many properties, like the molecular weight, size and shape of macromolecules, the thermodynamic interactions between different components in blends in the single phase and also under phase separating conditions are, as well as the order in single and phase separating systems can be investigated by scattering techniques. In this contribution it will be focused on the relationship between thermodynamic properties like compressibility in single phase systems and the thermodynamic interaction between different components in multi-component (mainly two-component) systems. These relationships will be discussed in terms of the structure factor and its relationship to experimentally measurable quantities in light, X-ray and neutron scattering. Also from the size of polymers in the matrix of similar or other components information about the thermodynamic interactions can be obtained. The lecture will include mixing theory for polymers in solutions and blends (Flory-Huggins-Staverman), theory of spinodal decomposition (Cahn-Hilliard) and scaling relationships between the size of polymer chains and their molecular weight (Flory).

Jean-Marc Saiter

It is common to read papers concerning glassy materials (organic or inorganic polymers) in which a value of glass transition temperature Tg is determined and proposed as a material characteristic. This temperature theoretically characterizes the transition between a non-equilibrium state (the glass) for $T < T_g$ from a metastable state (like liquid state) for $T > T_g$. The mean consequence is the existence of a molecular disorder for T<Tg leading to a molecular dynamic responsible of a physical ageing process, which, with other words, indicates that all the properties of a material kept at $T < T_g$ are time and temperature dependent. The problem is that the glass transition is not a material constant but depends upon the thermal history of the studied material.

During this short course I will show how to make a good T_g measurement by means of thermal analysis method. The first part of the presentation will be focused to the definition of T_g, the second part will be focused on the description of what is a wrong and what is a good T_g measurement.

Witold Brostow

Historically, much effort in Materials Science and Engineering (MSE) has been devoted to mechanical properties—for understandable reasons. By comparison, the available information on tribological properties of materials is lean. Tribological properties include friction, scratch resistance and wear. Wear is defined as the unwanted loss of material from solid surfaces due to mechanical interactions. Worldwide economic losses to industry caused by wear run into billions of US dollars. In metals one can use liquid lubricants. This is not doable for polymers and polymer-based materials (PBMs). A liquid between two polymer surfaces is likely to be soaked up by the polymers—leading to swelling of the materials involved. In the absence of suitable lubricants, excessive wear in service necessitates the replacement of both facing components. Needless to say, the replacement is a palliative only, and possibly a costly one. The operation has to be repeated periodically for the entire lifetime of the device or the structure. We shall discuss friction, scratch resistance and wear: methods of determination of these quantities and also methods of their improvements for PBMs.

Reference: W. Brostow & H.E. Hagg Lobland, Materials: Introduction and Applications, Ch. 20, John Wiley & Sons, New York 2017.

Sven Henning and Goerg H. Micher

The main aims of micromechanical analyses are the identification of property determining micro- and nanostructures and the description of the principal deformation phenomena that occur under mechanical load, as there are, for instance, crazing, homogeneous deformation, shear band formation, micro- and nanovoid formation, fibrillation, etc. These processes can be correlated to a brittle, semiductile or ductile material behavior on the macroscopic scale. In that sense, the description of micromechanical phenomena can contribute to the formulation of material laws predicting the response of a material to mechanical load. Micromechanical processes are the link between the morphology (predicted by synthesis, composition, and processing), loading conditions and resulting mechanical properties.

There are a number of different micromechanical techniques that can be adapted with respect to the actual problem. First of all, there are micromechanical in situ methods characterized by the application of tensile load, compressive or bending force using miniaturized testing devices inside the TEM, (E)SEM or AFM. Alternatively, microscopic analyses of deformation zones can be performed after mechanical testing ex situ with correlation to actual states of deformation. That approach also includes the inspection of fracture surfaces and, to some extent, problems of failure analysis. Thirdly, micromechanical techniques also include the application of miniaturized mechanical testing devices for small samples (e.g., micro-parts, biological samples, nanofibers) or for mechanical testing of small amounts of materials from new syntheses. Finally, the micromechanical approach allows the measurement of local mechanical properties in microscopic regions of interest.

The presentation also covers different preparation routes with a special emphasis on preparation by means of (cryo-)ultramicrotomy. The advantages of the different techniques are illustrated by a number of examples covering semicrystalline polymers, block copolymers, composites and nanocomposites with different filler geometries (particles, layered structures, carbon nanotubes) and biological/biomedical materials (bone, electrospun nanofibers).

Masura Matsuo

Most of undergraduate students have learned spectra resulting from electron transfer and they know that electromagnetic wave property with wave-particle duality depends on their wavelength. This note is concerned with diffraction and scattering for X-ray beam and visible light beam as electric magnetic rays. The wavelength of X-ray beam is much shorter than that of visible light beam and then the particulate is predominant in comparison with wave nature. Hence X-ray beam and visible light beam provide different characteristics.

Difference between the two is related to the different fluctuations of system dependent upon the absolute values of scattered intensity. That is, X-ray scattering intensity is due to mean square value of electron density fluctuation, while light scattering intensity, mean square value of refractive index. Accordingly, the scattered intensity distributions are given by Fourier transform of these fluctuations. The wavelengths (λ) of X-ray beam generated from Cu and Mo targets are 0.154 and 0.06198 nm, respectively, while the wavelength of He-Ne gas laser is ca. 650 nm. The large difference is attributed to photon energy (ε) given by

$$\varepsilon = \frac{hc}{\lambda} = h\nu \quad (c: \text{velocity of light}, \nu: \text{frequency}, h: \text{Planck's constant})$$

When an incident X-ray beam is entered, the electrons in atoms behave as free electrons because of high photon energy. On the other hand, when visible light is entered, electrons of atom cause vibration on bottom of the potential box. Considering vibrating dipole, the potential field is not isotropic and electrons move easily along C-C polymer main chain axis in comparison with the direction perpendicular to the C-C axis, when an incident wave interact with electrons belonging to C-C covalent bonds in main chains. That is, for an incident beam, the vibrating dipole moment along C-C axis becomes larger than that perpendicular to the C-C axis. Accordingly, scattered wave is sensitive to polarization condition of an incident beam. Accordingly, the diffraction and scattering of X-ray and visible light beams provide similar and different characteristics about diffraction and scattering.

The present talk is focused on easy understood commentary in terms of the experimental and theoretical concepts.

Robert G. Gilbert

Starch and glycogen are similar complex branched glucose polymers, with (1→4)-a linear links and (1→6)-a branch points. Both function as glucose storage polymers: starch for plants and glycogen for animals. Glycogen also contains a small but significant amount of protein. Starch compromises about half our food energy and is digested in our gastrointestinal tract to glucose (blood sugar), which is stored in our bodies as glycogen until needed for energy. Starch contains two forms of glucans: amylose with molecular weight ~ 10^6, comprising a small number of long-chain branches, and amylopectin with a much higher molecular weight, ~ 10^8, with a vast number of short-chain branches; these spacing between branch points in the latter is biologically regulated so that native starch comprises alternate crystalline and amorphous lamellae, with the crystallinity arising from shorter amylopectin chains forming helices. Glycogen has similar structure to amylopectin except slightly shorter chains, and lacks the regular spacing between branch points so that it does not crystallize. In muscles, glycogen takes the form of relatively small β particles, whose small size is ideal for rapid energy release [1], while in the liver, these small β particles are linked together to form composite a particles, with an aggregate appearance under TEM. Using proteomics, we have proved that the β particles are linked together in a particles by a protein 'glue'. Starch-based foods have impacts our human health: in the modern diet, digestion to glucose is rapid, with adverse implications for propensity to obesity, diabetes and colo-rectal cancers [2]. Using polymer theory [3, 4], we have created means to develop plants producing starches which are more slowly digested but still retain acceptable palatability. Diabetes is characterized by uncontrolled glucose release, and we have discovered that in diabetes, the glycogen a particles in liver are fragile, readily degrading to smaller β particles, which are more rapidly enzymatically degraded to glucose [5], with an obvious link to the symptoms of diabetes. Understanding the complex molecular structure of these two polymers requires a range of techniques, especially multiple-detection size-exclusion chromatography and fluorophore-assisted carbohydrate electrophoresis, together with advanced polymer theory for reducing the resulting data to a small number of physically and biologically meaningful parameters. This is leading to a wide range of outcomes, including indicating new drug targets for diabetes [6] and understanding food palatability [7].

Keywords: starch, glycogen, branched, nutrition, diabetes.

1. Deng B, Sullivan MA, Li J, Tan X, Zhu C, Schulz BL, and Gilbert RG. Glycoconjugate Journal 2015;32(3-4):113-118.
2. Topping D. J. Cereal Sci. 2007;46(3):220-229.
3. Wu AC, Morell MK, and Gilbert RG. PLoS ONE 2013;8(6):e65768.
4. Li C, Wu AC, Go RM, Malouf J, Turner MS, Malde AK, Mark AE, and Gilbert RG. PLoS One 2015;10(4):e0125507.
5. Jiang X, Zhang P, Li S, Tan X, Hu Z, Deng B, Li E, Wang K, Li C, Sullivan MA, and Gilbert RG. European Polymer Journal 2016;82(1):175-180.
6. Sullivan MA, Harcourt BE, Xu P, Forbes JM, and Gilbert RG. Current Drug Targets 2015;16(10):1088-1093.
7. Li H, Prakesh S, Nicholson TH, Fitzgerald MA, and Gilbert RG. Food Chem. 2016;196:702-711.

Michael Hess

The theoretical background of the viscoelastic behaviour of polymers is presented in short explaining the most important terms and experimental techniques. Numerous examples of application will be presented for pure polymers and blends.

This contribution complements the Short Course contribution of Jean-Marc Saiter.

Aik Hwee Eng

Crosslink density is an important property that affects the functional performance of a vulcanized rubber. It is normally determined by the solvent swelling method using the Flory–Rehner equation which describes the interaction of polymer and liquid molecules in equilibrium state. While the information on the total crosslink density is useful, when more than 1 type of crosslinks are present in a vulcanized rubber, further characterization work is necessary to understand the contribution of these crosslinks to the performance properties of the rubber. Therefore, the ionic and covalent crosslinks of carboxylated nitrle butadiene rubber and natural rubber can be quantified individually using solvents of different polarities. By using suitable chemical probes, such as thiolate ion, this technique also allows the separate determination of the mono, di and polysulphic crosslinks in vulcanized rubbers. The crosslink density can also be determined using the Mooney-Rivlin equation based on stress-strain data of vulcanized rubbers. By swelling vulcanized rubbers in styrene, polymerizing the styrene, and observing the microtomed rubbers under a transmission electron microscope, crosslinks that formed within a latex particle, or intra-particle crosslinks, and those of inter-particle crosslinks can be visualized and determined semi-quantitatively. This characterization technique is useful for one to understand the extent of crosslinking reactions in the latex and dry latex film.

Speakers' profile

Chin Han CHAN is an associate professor at the Faculty of Applied Sciences of MARA University of Technology, Malaysia. Her research interest is devoted to physical properties of macromolecules and polymer blends, specifically to thermoplastic elastomers, solid polymer electrolytes *etc*. She was appointed as Visiting Scientist at China University of Petroleum, Beijing, China (2011 – 2012), as Chair Professor at Mahatma Gandhi University, Kottayam, India (2014), She has published more than 70 papers in international and national refereed journals and more than 30 invited lectures for international conferences. She is one of the editors of books published by Royal Society of Chemistry (2013) entitled "Natural Rubber Materials" (2 Volumes), and Apple Academic Press (2014 & 2016) (distributed by CRC Press) entitled "Physical Chemistry of Macromolecules - Macro to Nanoscales" and "Functional Polymeric Composites", respectively. She peer-reviews many international journals on polymer sciences.

Jean-Marc Saiter is the director of the scientific pedagogy for Onyx, Groupe Nutriset Company, France and Prof. Emeritus, lab SMS, University of Rouen Normandy, France. He published over 200 refereed publications and presented more than 200 oral or poster presentations. He was awarded the Paul J. Flory award for research distinction of the Society of Polymer Characterization, the highest award of distinction in 2007. He is leading interdisciplinary research project related to materials sciences focused on disordered solid materials, development of method to characterize the value of the characteristic time of the molecular dynamic at the glass transition, development of new methods of investigation of the effect of disorder on the physical properties of glassy materials and leading development of an undergraduate and graduate program in Materials sciences

Michael Hess born in Germany, graduated (Diplom degree in Chemistry, Physical Chemistry) at the Rheinisch-Westfälische Technische Hochschule (RWTH) Aachen, Germany. He was promoted Dr.rer.nat. at the RWTH in the year 1976 (Physical Chemistry). In 1977 he became Senior Scientific Officer at the Gerhard-Mercator-University Duisburg, Germany (Department of Physical Chemistry), 2004 University Duisburg-Essen (Essen, Germany), 2008-2012 University of Siegen (Siegen Germany), retired.

He is Distinguished Adjunct Professor at the University of North Texas, Denton, Texas (Department of Physics) and since 1998 Visiting Professor at the Chosun University, Gwangju, Republic of Korea (Department of Polymer Engineering and Science). In 1995 he became member of the IUPAC Polymer Division (Commission IV.1 – Polymer Terminology), which he chaired from 2000 to 2002. Since 2008 he is Secretary of the Polymer Division (IUPAC = International Union of Pure and Applied Chemistry). He has more than 30 years experience in Macromolecular Chemistry and Physics (research and teaching) with about 130 publications and a number of tutorials and lectures in this field. He was one of the founders and presently is Vice-President of the annual POLYCHAR Conference (World Forum on Advanced Materials) and contributes each year to its tutorial Short Course on Polymer Characterization, next one (POLYCHAR 23) May 11-15, 2015, Lincoln (Nebraska), USA. In 2014, he was organizer of the Session on Polymer Characterization of the World Polymer Congress MACRO 2014, July 2014, Chiang Mai, Thailand, and MACRO 2016 in Istanbul, Turkey.

From 01.04.2013 – 31.01.2014, he was Visiting Professor at the University of Antioquia, Medellin, Colombia. He is presently acting secretary of the IUPAC Polymer Division and Associate Member.

Masaru Matsuo has completed his PhD at Kyoto University in Japan and he was a professor of Nara Women's University. After his retirement, he became a full time professor of Dalian University of Technology in China. Since September 1st 2014, he is a visiting professor of Dalian University of Technology. He has published more than 200 papers in refereed journal articles. He is IUPAC fellow and Certificate of Membership Award of ACS (July 2015 ~ July 2018). He received "The Award of the Society of Fiber Science and Technology of Japan" on May 1990, "Paul Flory Polymer Research Prize" on April 2010 and "Certificate of Friendship Award of Liaoning Province in China" on September 2011.

Volker Abetz studied chemistry at the University of Freiburg, where he received his diploma in 1987 and his doctoral degree in 1990 under the supervision of Prof. Dr. Reimund Stadler. In his doctoral thesis he developed dichroic and birefringent methods to study orientation behavior of multicomponent polymer melts and networks under shear and uniaxial extension, partly in collaboration with Prof. Gerry Fuller at the Stanford University, California. Then he joined the polymer physics group at the Max-Planck-Institute for Polymer Research in Mainz, where he worked on structure and dynamics of polymer blends until 1993. After working on the structure formation of interpenetrating polymer networks during a half year visit with a stipend from the European Capital and Humanity Program at the Institut Charles Sadron in Strasbourg he went to the Institute of Organic Chemistry at the University of Mainz, where he started to work on the morphological behavior of block copolymers. In 1997 he moved to the University of Bayreuth, where he finished his habilitation on the synthesis and morphological behavior of ternary block copolymers at the Chair for Macromolecular Chemistry in 2000. In 2004 he was appointed an associate professor position for Polymer Chemistry at the University of Potsdam and in the same year he accepted the position as a head of the Institute of Polymer Research of the Helmholtz-Zentrum Geesthacht, together with a position as a full professor position at the Faculty of Technology at the University of Kiel. He continued the work in the area of tailor made block copolymers and extended the scope of these materials successfully to membranes, besides initiating the institute's present activities in nanostructured materials for structural applications. In 2009 he was offered a chair position of the Leibniz-Institute for New Materials together with a full professor position for Chemical Material Sciences at the University of Saarland in Saarbrücken, which he declined. Presently he holds an offer for a full professor position for Physical Chemistry at the University of Hamburg. He is coordinator of several European projects, edited two volumes in Advances of Polymer Science and is author of several book chapters and more than 200 original publications.

Robert G. Gilbert is Research Professor at the Centre for Nutrition and Food Sciences, University of Queensland, and also at both YangZhou University and the Biolake hi-tech campus in Wuhan, China, under the Chinese Government 1000-Talents Foreign Experts program. He received his undergraduate training at Sydney University in 1966, and his PhD from the Australian National University in 1970. He carried out postdoctoral work at MIT in the US from 1970 to 1972, and then returned to the University of Sydney, where he held a personal chair in polymer chemistry and was Director of the Key Centre for Polymer Colloids. In 2006, he took up a position in the Centre for Nutrition and Food Sciences at the University of Queensland. He is a Fellow of the Australian Academy of Science, is author of about 450 papers, 6 patents, and 2 books. He is fluent in French and German as well as his native English, but only speaks limited Chinese.

Witold Brostow
RESEARCH: materials for electronics industry including nanocomposites and liquid crystals; effects of irradiation or magnetic fields on properties of polymer-based composites; molecular dynamics computer simulations of materials; sustainable materials & their processing; drag reduction and flocculation; new approaches to instruction in Materials Science & Engineering.

BOOKS, PUBLICATIONS and PATENTS: Science of Materials in 2 American, 1 Spanish and 2 German editions. Materials: Introduction and Applications, jointly

with H.E. Hagg Lobland, John Wiley & Sons, New York 2017. 415 refereed research papers; 5 patents. Editor of: Mechanical Properties of Polymer Liquid Crystals (1998) and Performance of Plastics (2000).
GRADUATE DEGREES: MS (in Chemistry) and DrSc (in Physics), University of Warsaw; DSc (in Chemistry), Polish Academy of Sciences, Warsaw.

Sven Henning is senior scientist at the Fraunhofer Institute for Microstructure of Materials and Systems (IMWS) in Halle (Saale), Germany, and lecturer in Polymer Materials Science at the Technical University Ilmenau, Germany. He studied physics at the Martin Luther University Halle-Wittenberg and received his PhD in Physics in 2006. The focus of his scientific work is on electron microscopy of polymers and biomaterials and the development and application of techniques to study micro- and nanostructures and micromechanical mechanisms of deformation and fracture of advanced materials. He is author of about 50 papers, 8 contributions to books, and 3 patents.

Aik Hwee Eng obtained his doctorate in material engineering from the Tokyo University of Agriculture and Technology, Japan in 1994. He was a researcher of the Rubber Research Institute of Malaysia from 1994 to 2001 and subsequently worked as a module director at the R&D center of a rubber glove company and has recently retired. He was awarded the Japan Science & Technology Fellowship in 1997, the Malaysian Young Scientist Award in 1998. He has published 35 scientific journal papers, filed 6 patents, co-authored 4 books and is a fellow of the Malaysian Institute of Chemistry.

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REGISTRATION FORM

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