

MINUTES

POLYMERS MULTISCALE PROPERTIES

FORTH INDUSTRIAL ADVISORY GROUP MEETING

Polymers: Multiscale Properties

FORTH INDUSTRIAL ADVISORY GROUP MEETING

**Globe Room, Bushy House
National Physical Laboratory, Teddington, Middlesex**

Tuesday 22nd April 2008

AGENDA

- | | |
|--------------|--|
| 10.00 | Tea/Coffee |
| 10.30 | Welcome and Introduction |
| 10.45 | Materials 2009 |
| | Bill Nimmo/Martin Rides/Bill Broughton |
| 11.15 | Overview of Elastomer Research at AWE: New Advanced Materials for the 21st Century |
| | Paul Morrell, Senior Scientist, AWE |
| 11.45 | SM10 – Bill Broughton/Tim Young |
| | Characterising Micro- and Nano-scale Interfaces in Advanced Composites |
| 12.15 | AM11 – Bill Broughton |
| | Diagnostics for Measuring and Modelling Dispersion in Nanoparticulate Reinforced Polymers |
| 12.30 | SE02 – Nigel Jennett/Miguel Monclus |
| | Improved Design and Manufacture of Polymeric Coatings Through the Provision of Dynamic Nano-Indentation Measurement Methods |
| 13.00 | Lunch |
| 14.00 | SM06 – Greg Dean |
| | Knowledge Based Design of Plastics |
| 14.30 | Discussion – Roadmap for Polymers and Nanocomposites |
| | Any Other Business |
| 15.00 | Closure of Meeting |

Multiscale Properties
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Attendees:

Darren Hodson	AstraZeneca UK
Paul Morrell	AWE
Paul Spencer	Bradford University
John Sweeney	Bradford University
Martin Watson	Crown Packaging UK PLC,
Andrew Lockley	Crown Packaging UK PLC
Bill Martin	DIUS
Judith Elder	Durham University
Steven Breen	GKN Aerospace Kings Norton
Hassan Menay	Hexcel Composites Ltd
Emile Greenhalgh	Imperial College
Angelika Menner	Imperial College
Ian Robinson	Lucite International Group
Ben Beake	MicroMaterials
Chris Stevens (Chair)	NGF Europe
Peter Cox	Peter Cox Associates
Andrew Hulme	Rapra
Chris Price	Security Composites Ltd

Bill Broughton	NPL
Louise Crocker	NPL
Greg Dean	NPL
Maria Iodeiro	NPL
Tony Maxwell	NPL
Miguel Monclus	NPL
Nigel Jennett	NPL
Martin Rides	NPL
Monica Saavedra	NPL
Jeremy Wormington	NPL
Tim Young	NPL

Note: Presentation can be viewed on the NPL website, by clicking the title in the minutes and entering the username "multiscale" and password "iagmember".

Website address:

<http://resource.npl.co.uk/materials/programmes/characterisation/pw/polymers/meetings.html>

Introduction

Bill Broughton

Bill Broughton welcomed everyone to the meeting and presented the agenda. He described the projects that were to be discussed and reminded the IAG that all the documentation and information from current and past projects can be found on the NPL website (web address and password are given at the top of this page).

Materials 2009

Bill Nimmo/Martin Rides/Bill Broughton

Bill Nimmo gave a short presentation outlining the changes that have been made to the project formulation process. Materials research is now divided into two different programmes with projects being developed every year under rolling formulation. This will involve NPL together with the IAG developing a roadmap to plan future work. Current projects will be reviewed against the roadmap and new projects developed. This will allow a long-term strategy to be developed and prevents projects being forced into a rigid 3-year structure, as is currently the situation. It is therefore essential for the IAG to develop a strategy for new projects and to construct a roadmap.

Overview of Elastomer Research at AWE: New Advanced Materials for the 21st Century

Paul Morrell, Senior Scientist, AWE

Paul Morrell from AWE at Harwell gave an overview of the research that they are conducting on elastomers. AWE is responsible for the maintenance and disposal of British nuclear weapons and for maintaining the capability of building nuclear warheads within the UK. At present there is considerable interest in the use of fillers in polymers including nanotubes. The main challenges are characterising the dispersion, concentration and location of the particles in the polymer and assessing the effect that they have on the processing and properties of the polymer. In addition, AWE is also conducting considerable work on the characterisation of polymeric foams. Using 3D x-ray microtomography AWE have been able to image foam structures down to a resolution of 6 microns and have used this data to develop a model of the foam.

Questions

Greg Dean: Does adding a filler reduce the elasticity of the material.

Paul Morrell: Some elasticity will be lost, but they are still capable of meeting the mechanical properties stated in the specifications.

Characterising Micro- and Nano-scale Interfaces in Advanced Composites (SM10)

Bill Broughton and Tim Young

The aim of this project is to develop quantitative techniques for the characterisation of interfacial properties in dispersed and continuously filled polymeric materials.

The first case study examined GRP pultruded rods, which have been supplied by Exel Composites Ltd and Owens Corning. One good and one poorly bonded composite have been examined. The interlaminar shear and longitudinal flexural strength of the poorly bonded composite are both significantly lower than the well-bonded system. SEM images showed blistering on the poorly bonded composite specimens but not on the well bonded specimens. Ultrasonic measurements failed to detect debonding in the poorly bonded continuous fibre composites and only a very small change in longitudinal flexural stiffness was observed using impact excitation.

The second case study examined the adhesion of glass flakes that have been embedded in a polypropylene matrix. The glass flake composite samples, supplied by NGF Europe, contained chemically treated glass flakes in order to alter the adhesion properties. The fracture toughness and strength of the poorly bonded flakes were all lower than for the well-bonded flakes. No significant difference was observed in volume fraction, density, crystallinity or hardness between the well and poorly bonded flake specimens. Differences were, however, observed between the well-bonded and poorly bonded flakes by conditioning the specimens at low temperatures to debond the flakes and using ultrasonics to detect the debonding. Residual strain measurements have been made using an incremental slitting method and also by hole-drilling using DIC. These techniques revealed that the poorly bonded specimens had higher residual strains than the well-bonded specimens.

Questions

Ian Robinson: Is the slitting method standardised?

Bill Broughton: The technique is being developed as an ASTM standard for metals.

Diagnostics for Measuring and Modelling Dispersion in Nanoparticulate Reinforced Polymers (AM11)

Bill Broughton

In this project a range of techniques are being assessed for measuring and modelling the dispersion of nanoparticles in reinforced polymers. The development of such techniques is essential in order to produce well-dispersed, cluster-free nanocomposites. Clusters may possibly form either during production or in service due to electrical, thermal or chemical exposure. A review of different measurement techniques has already been completed and a range of techniques selected for further study. The following four case studies will be considered in the project:

- Nanoclay/Polyamide (nylon 6 PA-6)
- Silica/PMMA
- Nanoclay/PMMA
- CNT/Polypropylene

Initial test work has been conducted using the nanoclay/polyamide and silica/PMMA nanocomposites. No significant difference was observed in the mechanical properties of the unreinforced polymer, and the well-dispersed and poorly dispersed nanoclay and silica reinforced nanocomposites. Nanoclay/polyamide showed differences in mechanical properties due to changes in nanoparticle loading. A reduction was observed in the melting point and the crystallinity of the polymer, but this was insufficient to use as a test method. Significant differences were, however, observed in the optical transparency of the nanoclay/PMMA nanocomposites.

Questions

Ben Beake: Why aren't changes observed in the mechanical properties of the polymer.

Ian Robinson: Fracture properties are improved, but the quantity of nano-particles in the polymer is insufficient to alter the stiffness of the polymer.

Improved Design and Manufacture of Polymeric Coatings Through the Provision of Dynamic Nano-indentation Measurement Methods (SE02) **Nigel Jennett and Miguel Monclus**

The aim of the project is to identify the agreement between methods as a route to validation of indentation derived viscoelastic property measurements.

The first part of the talk given by Nigel Jennett dealt with the investigation of dynamic nanoindentation experiments performed on the NanoII and Nanotest instruments looking at the effects of varying oscillation frequency. Modulus measurements with the NanoII at three discrete frequencies and preliminary results of multi-frequency tests performed on the NPL modified Nanotest were reported. With the current Nanotest set up, the pendulum damping is adjustable and the system is able to measure ~200 frequencies per hour. The oscillation response when damped and in contact with standard polymeric materials PS-1 and PS-3 was presented. The next step in the project will be to validate the calibration procedure and calculate modulus to compare results with NanoII and DMA.

The second part presented by Miguel Monclus dealt with viscoelastic analysis of nanoindentation creep data. A viscoelastic conical analysis (being developed with Cambridge University) was applied to PS-1 and PS-3 materials using pyramidal and conical indenters of different radius and varying applied forces. Modulus and viscoelastic properties deduced from the analysis were found to be load-independent for sharper indenters. It was highlighted the importance of stating time frames when reporting modulus values of polymers using different test methods.

Questions

Ben Beake: Are the results affected by the length of hold period fitted?

Miguel Monclus: The model diverges from the data over long times and so the fit to the hold period is kept as short as possible.

Knowledge Based Design of Plastics (SM06)

Greg Dean

The objective of SM06 is to develop a capability to design with polymers under long-term load. In the first stage of the project a model has been developed to describe the long-term deformation behaviour of polymers under a multiaxial creep stress.. Theoretical expressions have also been developed that enable the model to be used in applications where the stress or strain history varies with time. In initial attempts to develop code for implementing the model in a finite element system, the equations were coded as a user subroutine in Abaqus by reducing integral equations to differential equations and fitting them into existing subroutines. In subsequent evaluation studies, the FE analysis was found not to give the correct prediction for a step loading history. Investigations found that in the process of reducing the equations to differential equations, an error had been made in the analysis so that the solutions obtained were not appropriate. Due to the limited funds within SM06, coding the creep model into a finite element system now falls outside the scope of this project. The project deliverables have been revised.

The project will now evaluate the model and the theory for the simple case of uniaxial tensile loads, for which solutions can be obtained by numerical methods. Results from these analyses will be compared with the results of experimental tests in one case where the stress is changed in discrete steps and in another case where the strain is held constant (a stress relaxation test).. Early results from a stress relaxation test have been compared with the numerical solution, and the correlation is very good.

The work on coding the creep model into an FE system will now be carried out within a new project. Bradford University is helping us to explore methods for obtaining solutions to our equations within Abaqus.

Discussion – Roadmap for Polymers and Nanocomposites

Roadmaps for the development of projects in future research programmes have been developed for nanocomposites and polymeric materials. Martin Rides asked the IAG members to contribute ideas on a regular basis for inclusion in the roadmaps.

Questions and Comments

Ian Robinson: Design of polymers should be included in the roadmap.

Darren Hodson: Development of a greater understanding of the underlying science is needed for the modelling of polymers.

Chris Price: Measurement of nanotube alignment in polymers is required for FEA simulations.

Ian Robinson: One of the main requirements of the industry is to develop techniques for processing polymers at low temperatures that will not compromise the properties of the product or produce residual stresses. By reducing the temperature you use less energy and save money. Martin Rides asked whether supercritical CO₂ processing was a solution?

Ian Robinson: The biggest impact would be achieved by improving traditional techniques.

Andy Lockley: The use of recycled polymers would be of interest.

Bill Broughton: Do we need to include rubber systems into the roadmap?

Darren Hodson: The tribology and leaching of drugs and components in contact with elastomers is extremely important.

Chris Stevens: IoM³ produced a roadmap on rubbers and will provide a copy for Bill Broughton.

Chris Stevens: The migration of nanocomposites fillers is also an issue that needs considering.

Post-Meeting: A copy of the IoM³ roadmap on rubbers was received from Chris Stevens.

Any other business

Date for the next IAG will be the 15th October 2008. In future this IAG will be combined with the Polymer Processing IAG.