Materials Characterisation Programme

Polymers: Multiscale Properties

FIRST INDUSTRIAL ADVISORY GROUP MEETING

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

Wednesday 6th September 2006

AGENDA

10.00	**Tea/Coffee**
10.30	**Welcome and Introduction**
10.45	**Materials 2007+ and Other Programmes**
	Bill Nimmo/Clive Scoggins
11.00	**Challenge of Nanocomposites**
	Dr Ian Robinson, Lucite International UK Limited
11.30	**SM06 - Louise Crocker**
	Knowledge Based Design of Plastics
12.00	**SE02 – Nigel Jennett**
	Improved Design and Manufacture of Polymeric Coatings Through the Provision of Dynamic Nano-Indentation Measurement Methods
12.30	**SM10 – Bill Broughton**
	Characterising Micro- and Nano-scale Interfaces in Advanced Composites
13.00	**Lunch**
14.00	**Discussion – Future Direction**

Any Other Business

14.30	**Laboratory tour of Nanomechanical Facility (optional)**
15.30	**Closure of Meeting**

Multiscale Properties

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Attendees:

Mr Stuart Bates	Altair Engineering
Mr Darren Hodson	AstraZeneca
Dr Mogon Patel	AWE, Aldermaston
Dr Paul Morrell	AWE, Aldermaston
Dr John Meegan	AWE, Aldermaston
Dr Tol Purewal	Bespak
Mr Paul Abraham	DuPont (UK)
Mr John Clark	Fine R& D
Mr Neil McDermott-Evans	GKN Aerospace
Dr Ian Robinson	Lucite International UK
Dr Ben Beake	Micro Materials
Dr Chris Stevens	NGF Europe (Chair)
Mr Andrew Hulme	RAPRA
Mr Kevin Jackson	Visteon
Mr Mike McCarthy	Wilde FEA
Bill Broughton	NPL
Louise Crocker	NPL
Bruce Duncan	NPL
Greg Dean	NPL
Nigel Jennett	NPL
Tony Maxwell	NPL

Apologies:

Mr John Hartley	Fibreforce Composites
Dr Georges Limbert	FIRST Numerics

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://www.npl.co.uk/materials/programmes/characterisation/pw/polymers/meetings.html

Introduction
Bill Broughton

Bill Broughton welcomed everyone to the meeting and thanked them for coming. The scope and aims of the current work programme were presented to the IAG members and the IAG informed that all documentation and information from the projects would be placed on the NPL website – the webpage address and the password needed to access it were given at the IAG. Bill Broughton then introduced Dr Chris Stevens (NGF Europe) who chaired the meeting.

Materials 2007 Project Formulation
Bill Nimmo

Bill Nimmo gave a short presentation outlining the changes that have been made to the project formulation process. One of the main changes is that the material and thermal programmes will be merged together. As a consequence, there is particular interest in formulating projects that build on our expertise in both of these fields, such as the measurement of moisture in materials and the measurement of materials thermal properties. The key requirements of such projects are that they should be both innovative and contribute to the UK's knowledge base. The current formulation process is due to be completed in March with the projects starting in April 2007. In future it is intended to have a rolling formulation process with projects being developed throughout the year.

Challenge of Nanocomposites
Dr Ian Robinson, Lucite International UK Ltd

Dr Ian Robinson gave a short presentation explaining the reasons why there's growing interest in the use of nano-composite fillers and the technological and scientific challenges that have to be overcome. The properties of conventional glass and carbon fibre filled composites are now well known and can be predicted reasonably well using formula based on the rule-of-mixtures. However, the properties that have been obtained from nano-composites are very different and cannot be explained using conventional composite theories. There is an exponential increase in the number of papers that are being published with claims that:

- there are significant increases in the toughness and strength of polymers due to the presence of nano-fillers,
- the T_g of certain polymers is significantly increased by nano-fillers,
- there are significant changes to the rheological behaviour of the polymer,
- improved barrier properties and minimal loss of optical properties.

The main difference between nano-composites and conventional composites would appear to be the extremely large surface area of the filler. It is, however, essential to obtain good dispersion of

the filler in the polymer to maximise the impact of the filler as it is believed that the nano-particles actually change the polymer chain dynamics.

Ian Robinson felt that the key issue that will have to be addressed is to ensure the long-term stability of these materials, through the development of reliable accelerated testing protocol. This is a significant issue for all polymers as the life-expectancy of polymers has traditional been obtained assuming that they are to be used in typical European climates whereas today's growing markets are in Asia.

Questions

Bill Nimmo: Are the thermodynamics of nano-materials the same as that of bulk materials?

Ian Robinson: The thermodynamics are different.

Ben Beake: Is exfoliation necessary?

Ian Robinson: Exfoliation is essential as it is the surface area of the nano-particles that alter the properties of the polymer.

Tol Purewal: Is there anything that may point you in the right direction to obtain suitable properties from a nano-composite?

Ian Robinson: Not at the moment but the field is developing all the time.

Knowledge Based Design of Plastics (SM06)

Louise Crocker

Louise Crocker described the overall aims of the project along with the benefits for industry. There are two main deliverables in the project that have been divided into five key stages. The objectives of stage 1 were outlined and progress discussed. It has been agreed that Polyacetal (POM) and polybutylene terephthalate (PBT) will be studied in the project. Dupont is currently supplying these materials, although material from Ticona may also be obtained. The format of the case study has been agreed –the creep of a bend specimen will be predicted as this will contain stresses that vary with location and time. It is also a geometry that can be reliably tested in the lab. The data acquisition will be carrying out by creep tests in tension, compression and shear. Stage 1 has been completed, and Louise explained that Stage 2 would be planned after the Stage 1 project board review meeting on the 21st September (Note: this meeting has since been held, and it was agreed that Stage 1 was complete, the project was slightly ahead of plan, and Stage 2 could continue with no major change in scope or direction).

Greg Dean then presented the modelling approach that is to be used. He gave an overview of the behaviour of the POM material Delrin. Greg defined a creep compliance function and showed how it could used to model the tensile creep behaviour of Delrin. The parameter τ, a mean retardation time, was introduced and it was shown that this parameter decreased with increasing stress, and the variation with stress could be modelled. Comparison of tensile compliance data with that from tests under compression and shear loading indicated that to extend the model to multiaxial stress states the variation of the parameter τ needed to be modified to account for the

effective stress state. This modified function was shown to accurately predict the creep behaviour in shear.

Questions

Mogon Patel: Will be applicable to foams and foams filled with silica?

Greg Dean: The model should be suitable for systems with fillers as the fillers should behave like crystals.

Ian Robinson: Is the modelling procedure to be published?

Greg Dean: The modelling procedure is to be published as a paper.

Ian Robinson: Will you be including temperature effects on creep?

Louise Crocker: Not in this current project. Temperature effects are the subject of a proposal that has been put into the Materials 2007+ formulation, but we don't know whether it has been successful. (Update: the project is not being considered for funding this year, but as part of the rolling formulation system has been placed in a hopper for consideration next year).

Improved design and manufacture of Polymeric Coatings Through the Provision of Dynamic Nano-indentation measurement methods (SE02)

Nigel Jennett

Nigel Jennett introduced himself and gave an explanation of the aims, motivation and objects of the project. The main aim in this work is to develop indentation measurement techniques that are capable of measuring the mechanical properties of a polymer at localised points within a component. This is of particular interest where only small volumes of material are available such as in micro-mouldings and coatings or where the surface properties have been altered during processing or subsequent use. Nanoindentation has been used extensively to determine the properties of non-time dependent materials such as metals and ceramics where NPL has been involved in the development of test protocol for international standards. The testing of plastics is significantly more complex due to their visco-elastic behaviour. As a consequence of their time-dependent nature it is preferable to use a dynamic rather than static indentation method. This involves superimposing a small oscillating load on to the indenter and measuring both the corresponding displacement and the time lag between the two signals. The objectives of the project are therefore to:

- Validate indentation protocols for the measurement of mechanical properties in visco-elastic materials for ISO standardisation and certified reference materials.
- Compare the methods to measure polymer properties as a function of frequency and temperature.
- Develop ultra-rapid indentation and creep-relaxation measurement methods for characterisation of visco-elastic materials.

Questions

Paul Morrell: Can you use the same materials as in SM06 and the data that was obtained in previous studies.

Nigel Jennett: Yes previously obtained data and materials can be used.

Nigel Jennett: What are the key parameters that are needed from these test methods?

Chris Stevens: Time dependent data is required, for rubbers these are mostly obtained at a frequency of around 10 Hz.

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

Bill Broughton

Bill Broughton explained that the aim of his project was to develop quantitative techniques for the characterisation of interfacial properties in dispersed and continuous filled polymeric materials, such as fibre-reinforced polymers, nano-composites and toughened adhesives. The first stage of the project has been to produce a review of measurement and modelling techniques. The techniques covered include; coupon tests, micromechanical interfacial test methods (single-fibre fragmentation test, single-fibre pull-out test, microbond test, microindentation), nanoindentation, nanoscratch, Raman and ultrasonics. Bill described the main advantages and disadvantages of the different measurement and modelling techniques in his presentation. The single-fibre fragmentation test replicates the stress transfer characteristics in the fibre well but the results are difficult to interpret due to the complex stress states that occur at the fibre/matrix interface. Coupon tests only provide an indirect measure of interfacial strength. Micromechanical interfacial test methods tend to be relatively straightforward in regard to specimen preparation and testing. Data analysis methods are oversimplified, assuming constant shear assumptions when non-uniform stress and strain distributions and mixed mode failures occur in all cases. Generally, there is a high degree of scatter in the data generated. Raman spectroscopy is capable of strain mapping with high resolution but the relationship between the Raman signal and the stress and strain is complex. Bill asked whether anyone was using these techniques in industry but no one was aware of them being used. The next stage of this project will involve evaluating the different measurement and modelling techniques in a series of case studies involving three different polymer composite systems; GRP pultruded rods, glass-flakes and nanocomposites. Fibreforce composites, NGF Europe and Lucite International have kindly promised to provide material for the case studies.

Questions

Tol Purewal: What properties will be considered in the case studies?

Bill Broughton: Fracture strength and stiffness will be examined, particularly the effects that the sizing has on the properties.

John Clark: Would it be possible to develop one of the simpler test methods as this may be more appropriate for smaller companies?

Neil Mcdermott-Evans: The Problem with simpler tests is that it is difficult to interpret the results due to the complex stress states that are obtained in such tests.

Tol Purewal: There are really two different types of test required in industry, quick simple tests that can be used for quality control and the more complex tests that allow you to really understand the properties of the material.

Discussion – Future Direction

Bruce Ducncan

Bruce Duncan asked the IAG for suggestions and comments on potential formats for future meetings – among the suggestions put forward were:

- focussing on one specific project at each meeting in turn;
- a general overview of all the projects at the start of each meeting, followed by the IAG breaking up into separate smaller groups for each project;
- interim meetings for each individual project.

Questions

Ian Robinson: The handouts that have been provided are extremely useful but increasing the number of meetings would be too time consuming for most people from industry to attend.

John Meegan: Can information from the meeting be posted on the website?

Bruce Duncan: All the presentation will be placed on the NPL website on password protected pages.

Any other business

Date for the next IAG will be the 7th March 2006.