

# **Polymers: Multiscale Properties**

## **THIRD INDUSTRIAL ADVISORY GROUP MEETING**

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

**Thursday 8<sup>th</sup> November 2007**

### **AGENDA**

- |              |  |
|--------------|--|
| <b>10.00</b> | <b>Tea/Coffee</b>  |
| <b>10.30</b> | <b>Welcome and Introduction</b>  |
| <b>10.45</b> | <b>Materials 2008</b>  |
|              | <b>Bill Nimmo/Clive Scoggins</b>   |
| <b>11.00</b> | <b>In Silico Analysis of Medical Devices and Material Research</b>   |
|              | <b>Darren Hodson, AstraZeneca R&amp;D Charnwood</b>  |
| <b>11.30</b> | <b>SM06 – Greg Dean</b>  |
|              | <b>Knowledge Based Design of Plastics</b>  |
| <b>12.00</b> | <b>SE02 – Nigel Jennett</b>  |
|              | <b>Improved Design and Manufacture of Polymeric Coatings Through the Provision of<br/>Dynamic Nano-Indentation Measurement Methods</b> |
| <b>12.30</b> | <b>SM10 – Bill Broughton</b>   |
|              | <b>Characterising Micro- and Nano-scale Interfaces in Advanced Composites</b>  |
| <b>13.00</b> | <b>Lunch</b>   |
| <b>14.00</b> | <b>AM11 – Bill Broughton</b>   |
|              | <b>Diagnostics for Measuring and Modelling Dispersion in Nanoparticulate Reinforced<br/>Polymers</b>                                   |
| <b>14.30</b> | <b>Discussion – Future Direction</b>   |
|              | <b>Any Other Business</b>  |
| <b>15.00</b> | <b>Closure of Meeting</b>  |

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**Attendees:**

Dr Chris Stevens	NGF Europe (Chair)
Dr Mark Lees	Hexcel
Dr Hassan Menay	Hexcel
Mr Darren Hodson	AstraZeneca
Mr Andi Clements	MIRA
Dr Stephen Grant	Ansys
Dr Mark Robinson	Ansys
Dr Ian Wands	Du Pont
Dr Paul Morrell	AWE, Aldermaston
Dr Ian Robinson	Lucite International UK
Bill Broughton	NPL
Louise Crocker	NPL
Greg Dean	NPL
Nigel Jennett	NPL
Tony Maxwell	NPL
Miguel Monclus	NPL
Martin Rides	NPL
Tim Young	NPL

**Apologies:**

Bill Martin	DIUS
Hardyal Kalsi	Kohler Mira Ltd
Stuart Bates	Altair UK
Chris Price	Security Composites Ltd
David Mason	NGF Europe
Tol Purewal	Bespak
Anis Zakaria	AKZO Nobel Nippon Paint Ltd
John Hartley	Exel Composites UK Ltd
Neil McDermott-Evans	GKN Aerospace Kings Norton
John Meegan	AWE, Aldermaston
Paul Abraham	DuPont (UK)
Ben Beake	Micro Materials
Andrew Hulme	RAPRA

***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 informed the IAG that all the documentation and information from current and past projects could be found on the new NPL website (web address and password are given at the top of this page).

**Materials 2008+ and Roadmapping**

**Bill Nimmo and Neil Campbell**

Bill Nimmo introduced himself and then 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. It is therefore essential to have a strategy for developing new projects and to construct a roadmap. The DIUS wants roadmaps to be produced by the end of April 2008 across all of the NMS programmes. The roadmaps cover the research that is required to achieve particular goals over the next 15 years. Neil Campbell gave an example for the energy industry based on a European roadmap.

**Questions**

Ian Robinson: Can you circulate a copy of the roadmap.

Bill Nimmo: Neil will send a copy out.

Ian Robinson: Can you provide us with a copy of the government target so that we can build on them.

Bill Nimmo: Bill and Neil will summarise the government's aims set out by the Technology Strategy Board and distribute it to the IAG members.

Ian Robinson: Lucite produced a "Mind map" on the requirements for the development of new materials that he will send to Bill.

Andi Clements: Could a draft roadmap be sent out by e-mail, which can be updated and then sent out again in a revised form.

Ian Robinson: Some issues such as the degradation of materials in harsh environments are extremely important to industry but may not be seen in the high level goals.

Chris Stevens reminded everyone that these roadmaps are critical to the development of new programmes and asked everyone to contribute to them.

## **In Silico Analysis of Medical Devices and Material Research**

### **Darren Hodson, AstraZeneca R&D Charnwood**

Darren Hodson described the procedures that are being developed to assess the performance of medical devices for regulatory purposes. Polymeric medical devices such as inhalers and sprays are produced in tens of millions and distributed throughout the world so performance is essential. Typical products can contain up to 25 parts, are typically made of POM and polypropylene and can be used for up to a year. A series of case studies were then described examining the modulus and variance of different polymers over a range of temperatures from 20 to 60°C. Future requirements for the in silico analysis of polymer devices include improvements to the modelling of the material and the development of rapid measurement techniques.

#### **Questions**

Ian Robinson: Is the modelling conducted every time a change to the design is made?

Darren Hodson: Everytime a new materials supplier is used new creep data is required hence there is a great need for accelerated testing methods.

Martin Rides: How different are the material properties in small devices compared to bulk properties?

Darren Hodson: Properties of completely different when it comes to modelling creep in small components.

## **Knowledge Based Design of Plastics (SM06)**

### **Greg Dean**

The object of project SM06 is to develop the capability to design polymeric components for use under long-term loading conditions. The first phase is to develop models to determine long-term deformation behaviour under multiaxial stress states. The next phase includes developing and evaluating code for implementing the model in the ABAQUS software package to enable long-term stress analyses to be carried out for arbitrary load histories. Dupont and Ticona have supplied the materials for the project. Specimens have been tested of both Polyacetal (POM) and polybutylene terephthalate (PBT). Some modifications to the creep model have been made to take account of a secondary relaxation peak that occurs in the PBT specimens. Sub-routines were then written to allow the ABAQUS FEA package to model non-linear viscoelastic behaviour. The next stage of the project will be to evaluate the FEA code using step loading, flexural creep and stress relaxation tests as case studies.

#### **Questions**

Ian Robinson: Developing models and test protocol to determine the long-term properties of polymeric materials is essential to UK industry particularly in the health care and packaging industries.

The IAG expressed concern that a creep project, proposed for funding in the 2008 NMS programme with the support of some of the members this IAG, had not been selected by NPL for inclusion in the project proposals put forward to the MAC.

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

**Nigel Jennett**

The main objectives of this project are to validate protocols for dynamic indentation measurements suitable for ISO standardisation; to compare the methods as a function of frequency and temperature; and to develop ultra rapid test methods. The project has now reached its midpoint and considerable amounts of data are currently being processed. Sensitivity studies have been conducted which have shown that it is important to choose the correct values for the dynamic parameters. Nigel showed also the effect different parameters have on the response of restitution in dynamic micro-impact tests. Viscoelastic models are also being developed for these test methods with Cambridge University. Nigel concluded that the test methods are sensitive to calibration but are reasonably robust and repeatable.

### **Questions**

Andi Clements: Do you get large energy releases using the micro-impact?

Nigel Jennett: Yes the advantage of the micro-impact over other techniques is that you can control the energy of the impact, which can be extremely large over the small area of the impact.

Hassan Menay: How does this differ from the AFM?

Nigel Jennett: In principle the theories are the same.

## **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. An initial review of measurement and modelling techniques has been produced and is available on the NPL website. The next stage is to conduct case studies to examine adhesion in a range of three different composite systems.

The first case study examined the interfacial adhesion in GRP pultruded rods, which have been supplied by Fibreforce and Vectrotex. One good and one poorly bonded composite have been examined. The modulus and the strength of the poorly bonded composite are both significantly lower than the well-bonded system. SEM images indicated that there was a band approximately 200nm thick present at the interface of the poorly bonded fibres. Force displacement AFM has been used to measure adhesion and modulus on the nanoscale in both the good and poorly bonded composites. TEM with focused ion beam (FIB) has been used to examine 100 nm thick specimens in order to view the adhesive interface. Bill Broughton then described how the composites are being modelled to take into account the damaged interfaces.

The second case study to be conducted in this project involves examining 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 mechanical properties of the poorly bonded flakes were all lower than for the well-bonded flakes. SEM examination of the flakes indicated that there might be an interphase region surrounding treated glass flakes of the order of 200 nm thick. Residual stress

measurements have been made using thermal expansion and hole-drilling measurement techniques. Significant differences were observed using the hole-drilling technique between the reinforced and unreinforced polypropylene samples and with different levels of interfacial bonding. A range of different models have been considered for modelling the mechanical properties of the glass flake reinforced polypropylene composites, however, these are limited due to the fact that they assume perfect bonding. A new model is therefore being developed.

PMMA nanocomposites were used for the third case study. This has proved to be the most technically difficult of the three case studies. DMA tests have been conducted, but it was not possible to detect the onset of possible cracking at sub-zero temperatures.

### **Questions**

Paul Morrell: Is the FIB apparatus based at NPL?

Tim Young: No, it's at Surrey University?

Paul Morrell: How long does it take to cut a specimen using FIB?

Tim Young: It takes at least 1.5 hours per specimen.

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

**Bill Broughton**

AM11 is a new project that will be examining methods for measuring and modelling the dispersion of nanoparticles in polymers. A review is being conducted and a range of different measurement techniques will be examined including dielectrics, TEM, XRD, AFM, light scattering, ultrasonics and mechanical techniques. Dispersion of the nanoparticles will also be modelled. Neil McCartney has suggested using Maxwell's far-field method to model the elastic properties of clustered particles. Bill Broughton asked the IAG to suggest suitable case studies for the project.

### **Questions**

Andi Clements: How were the specimens produced?

Ian Robinson: They were cast.

There was general agreement that this project is valuable and worth pursuing.

### **Any other business**

Date for the next IAG will be the 22<sup>nd</sup> April 2008.