NATIONAL PHYSICAL LABORATORY

INDUSTRIAL ADVISORY GROUP MEETING ON MEASUREMENT METHODS RELATING TO THE PROCESSING AND PERFORMANCE OF PLASTICS

Minutes of the 7th Industrial Advisory Group Meeting held at NPL (Auditorium, Module 16) on Wednesday 19th October 2005 at 10.30 am

Participants:

Vicky Joss	Amcor Flexibles
Darren Hodson	AstraZeneca
Martyn Bennett	Avon Rubber
Adrian Kelly	Bradford University
Graham Reed	Department of Trade and Industry
Don Fleming	Fleming Polymer Testing & Consultancy
John Hockey	Hi-Technology Group
Amarjit Grewal	Jaguar and Landrover
Upul Ratnayake	Loughborough University
Ian Robinson	Lucite Int. UK
Stephen Gray	MIRA
Peter Cox	Peter Cox Associates
Alec Barron	RAILKO
Tim Biswell	RAILKO
Jas Mahey	TA Instruments Ltd
Bruce Duncan	NPL
Martin Rides	NPL
Angela Dawson	NPL
Paul Tomlins	NPL
Greg Dean	NPL
Colin McKechnie	NPL
Richard Mera	NPL

Apologies for absence were received from:

Brian Powell	Alpha Technologies UK
Julian Cubitt	Anglian Windows
Daven Chamberlain	Arjo Wiggins
Roy Carter	Celsum Technologies Ltd
Martin Watson	Crown Cork & Seal Co Inc
Bill Martin	DTI
Dilwyn Jones	Emral Ltd
David Pindar	Federal-Mogul
Barry Hennessey	Ford Motor Company Ltd
Patrick Leevers	Imperial College

Gary Pitman	Innovene
Richard Brown	Landrover
Bryan Cass	Linpac Plastics
Barry Haworth	Loughborough University
Jan Czerski	Materials Information Centre
Oliver Tomlin	MIRA Ltd
Chris Stevens	NGF Europe
Roger Stewart	PERA International
Richard Simpson	RAPRA Technology
Paul Maley	Selex Communications Ltd
Holger Norenberg	Technolox Ltd
John Harrigan	University of Manchester
J Mohanraj	University of Leeds
Gary Waller	Visteon Customer & Tech. Centre
Steve Lackovic	Wallace Instruments

CONTENTS

1. INTRODUCTION AND MINUTES OF THE PREVIOUS MEETING

Peter Cox (Peter Cox Associates) welcomed all participants to the 7th IAG meeting of the DTI funded MPP (**M**easurements for the **P**rocessability and **P**erformance of Materials) research programme. Peter thanked Darren Hodson of Astra Zeneca for coming along to give a presentation on the "Application of Computational Techniques during the development of Packaging and Medical Devices" to the IAG. Martin Rides gave a brief overview of the planned IAG meeting on processing and performance of plastics and described its overall purpose. He stated that the meeting would split into two parallel sessions of equal length, referred to as Theme 1 and Theme 2. Theme 1 would centre on the projects "Modelling the Behaviour of Plastics for Design Under Impact" (MPP 7.9) and "Measurement of Softness" (MPP 7.7). Theme 2 would centre on the projects "Flow Properties of Filled Materials and Rheological Standards" (MPP 7.4) and "Heat Transfer in Polymers" (MPP7.1).

The minutes of the 6th IAG meeting held on 27th October 2005 at Loughborough University were accepted as written.

The date of the 8th IAG meeting was set for 29 March 2006.

2. CHARACTERISATION PROGRAMME FORMULATION

Bruce Duncan gave an outline of the proposed polymer projects in the Characterisation Programme. These are; "Modelling Plastics Materials for Design" which is a development of Greg Dean and Louise Crocker's work to investigate the long term multi-axial performance of plastics, "Measurement of Naturalness" which will develop methods for characterising the feel properties of a wider range of synthetic and natural materials, and "Characterisation of Interfaces in Continuous and Dispersed Materials Systems". Bruce then went on to outline proposed projects in the polymer matrix composites area. These are "Validation of Composite Component Design", "Embedded Sensor Metrology For Condition Monitoring" and "Traceable Measurements to Improve Quality in Composite Materials". It was explained that the process is being lead by Bill Martin, DTI and Brian Chapman, NPL. More details can be found at www.dti.gov.uk/nms.

3. MODELLING THE BEHAVIOUR OF PLASTICS FOR DESIGN UNDER IMPACT (Project MPP 7.9)

Greg Dean (NPL) gave an introduction to the objectives and the current status in the project.

Three elastic plastic models have been used to predict deformation behaviour of Land Rover door trim components. The determination of experimental values for the properties and parameters required by each of the three elastic-plastic models were described. Testing issues such as obtaining data at high rates were also discussed. Greg's talk referred to recent work on measurement of failure at high strain rates in a waisted specimen, using image correlation software.

The difference in material behaviour between injection moulded specimens and those from the door panels was highlighted. Results from calorimetry, chromatography and melt flow were presented. These confirmed that the melt flow index results supported the different behaviours observed in materials. Orientation could also be responsible for some of the differences observed. In answer to a question, Greg Dean commented that X-ray measurements had been carried out to investigate crystallinity effects, but nothing could be seen due to the presence of the filler.

In clarifying the criterion for cavitation initiation, Greg Dean explained that the volumetric strain needed to reach a critical strain for initiation. However, particle size distribution leads to a strain dependent cavitation envelope. Greg Dean was asked about generation of data for cavitation model. He replied that data from three tests (tensile, shear and compression) are needed, whereas data from only two are needed for the linear Drucker-Prager model. Shear curves are used as hardening curves in cavitation model.

Louise Crocker (NPL) described the benefits of using FE analysis in the design of automotive components that could be subject to passenger or pedestrian impact. Two component parts cut from the Land Rover door trim have been analysed, the top trim and armrest. FE predictions of the top trim and armrest obtained using three models; von Mises, linear Drucker-Prager and the cavitation model, were presented. There was good comparison between experimental data and the cavitation model. The linear Drucker-Prager model gave the next closest prediction followed by the von Mises model.

Using high-speed photography, photographs of the underside of the top trim component had been obtained during testing at 0.1 mm/s. The photographs were used to analyse the changes in slope of the load-displacement plot observed experimentally and in FE predictions. The deformation prediction obtained using the cavitation model compared well with photographs.

Predicted load-displacement curves for the armrest matched experimental data well up to 15mm displacement then, with all models, the predicted curves peak. This is not observed experimentally. A study has been carried out to understand the cause of this difference. Photographs of the armrest component during testing show a different deformed shape to that found with FE, which predicts a partial collapse of the component. It had been suggested at a previous IAG that variable sample thickness might be the cause, as the thickness was treated as a constant in the FE analysis. Changing the thickness at the 'corners' of the armrest increased the stiffness of the component but did not alleviate the collapse. Friction was also thought to be a possible cause of the difference. Initial FE analyses showed no difference in predictions when friction was included. However, the friction value assumed was thought to be suspect. The IAG members were asked for their advice. NOTE: FE analyses with more realistic coefficient of friction values have now been rerun, and the problem has been overcome.

Sensitivity analyses have been carried out. In both components, the impact location can affect the initial stiffness of the force-displacement curve. When testing the armrest component, the jig is held at an angle to allow indentation of the flat surface of the component. Analyses have shown that small differences in this tilt angle have little effect of the force-displacement curve. Replacing the hemispherical indenter with a cylindrical indenter had a large effect of the predicted force-displacement curve. Discussion continued on the effect of friction between the indenter and the component on predictions (a value of 0.2 for the friction coefficient was used) and a suggestion was made of trying to lubricate the surface of the indenter, e.g. using PTFE, to identify the effect on measurements. A series of analyses looked at the sensitivity of predictions (force-displacement curve, peak stresses and strains) to the values of the cavitation parameters. It was observed that the modulus and rate-dependence parameters had the largest effect on predictions. In answer to a question, the effect of mesh size had also been investigated briefly: a fine mesh has been selected for the investigations performed.

The recent and future outputs from the project were listed.

There then followed a discussion of the proposal submitted to the new Characterisation Programme, due to start in April 2006. Louise Crocker gave a brief summary of the proposal, a principal output of which is an FE method that will assist in the design of plastics components for long-term, load-bearing applications. Members of the IAG stated that they would like to see polypropylene, PBT, acetal studied. PMMA was suggested as another suitable material for study. In answer to a question, the project will probably use ABAQUS initially, but the materials model could be coded for Ansys and LS Dyna as well. The comment was made that ABAQUS is a very good 'all around' package. It was commented that non-linearity is included in isochronous curves. It was suggested that an assessment of residual strength could be included, and that properties depend on injection moulding conditions (e.g. residual stress). Also the effect of temperature would be of interest. It was suggested that acceleration of creep through UV exposure/leaching of additives should also be investigated.

4. MEASUREMENT OF SOFTNESS (Project MPP 7.7)

The final phase of this project has involved modelling the physical indentation of soft rubber-like materials using finite element methods. The finite element approach requires a materials model and data that are specific to the materials being modelled, both of which have been obtained through mechanical testing. Greg Dean spoke about the stress relaxation and dynamic mechanical measurements and the derivation of models to describe the behaviour of the materials. Louise Crocker described the method used to obtain the finite element predictions for the response of a block of thermoplastic elastomer subjected to an indentation that mimics the Shore hardness method. Good agreement was obtained between the predicted Shore hardness values and those measured experimentally. This correlation can be exploited in design: finite element calculations can be used to predict the Shore hardness of the thin coatings of thermoplastic elastomers that are used to impart a soft-touch feel to everyday objects. The value of this approach is its predictive capability. The expected feel of the material can be assessed in reality by exploiting the very strong psychophysical relationship that have been obtained linking perceived hardness of rubber-like materials to measured Shore hardness. This relationship shows a one-to-one correspondence between perception and reality.

In answer to a question, the contribution from friction in predicting the displacement of the rubber during indentation has not yet been incorporated. Further calculations will be carried out to map the displacements and look for areas where friction between the indentor tip and the surface may be a problem.

In reply to a question, Paul Tomlins commented that the information will be disseminated to the design community through targeted press releases, articles in the trade press, newsletters, through active participation at exhibitions i.e. Interplas and through direct discussions with companies.

5. RHEOLOGY OF MULTIPHASE MATERIALS AND POLYMER STANDARDS (Project MPP 7.4)

Martin Rides outlined the project “Processing behaviour of multi-phase materials”. Tasks in the DTI project were outlined and major objectives stated. He then reported on the development of methods for the measurement of the slip behaviour of multi-phase materials. Extrusion pressure versus apparent shear rate, and apparent shear viscosity versus apparent shear rate data for a range of materials was presented. The materials were extruded through dies of differing diameter in order to investigate the measurement of the slip behaviour of these materials. AAEHH005 was HDPE with a high carbon black loading, whereas AAEHH002 was a highly filled EVA.

In reply to comments about whether slip or melt fracture were being observed, Martin Rides said that both mechanisms were observed and that this is an issue that further complicates the reliable measurement of the materials’ behaviour. If slip happens after melt fracture then there should be a geometry dependence of behaviour. Martin Rides pointed out that if shear viscosity data from the non-slip region were used for modelling purposes, without incorporation of methods to describe the slip behaviour at higher rates, then errors in predictions would occur. Martin Rides stated that a factor of 2 reduction in pressure drop was seen for material H005 when entrance pressure was plotted against apparent shear rate due to the slip phenomenon that is stress related.

An intercomparison was being organised in which Bradford University, Loughborough University, Fleming Polymer Testing and Consultancy and NPL were participating. The intercomparison was open to others to also participate. It was expected that the results of the intercomparison, using AAEHH002 and AAEHH005 as the materials, would be reported at the next IAG meeting.

Martin Rides then presented results obtained using a flow cell in which the polymer flow can be observed visually through a glass window. Good agreement between the slit die and capillary die shear viscosity measurements had been obtained, demonstrating the suitability of the slit die flow cell for measurement. Through seeding the flow with particles the local velocities can be measured, using a camera. Results of such velocity measurements were presented, with the objective of provide additional data to validate the method for measuring slip velocities using different die diameters.

By reliable characterisation of slip flow and incorporation of it into modelling then predictions of pressures to fill and temperature fields in injection moulding would be more accurate. This would enable improvements in design predictions leading to likely improvements in product properties (e.g. reduced distortion) and reduced time-to-markets. By exploiting slip, for example through designing materials to slip, it is likely that reductions in the process energy required to fill moulds or extrude through dies could be achieved.

There was discussion on results previously presented on rubber samples (carbon black filled EPDM) provided by Avon Rubber. An issue to clarify is why if we get slip at low rates is it not then observed at high rates. Was a phase separation being observed? Martin Rides said that further testing of those materials could be carried out but it was agreed that fresh samples should be used as the existing samples were now likely to be affected by their age.

Martin Rides then provided an update on progress on rheometry standards, in particular on the development of ISO 1133 MFR/MVR to incorporate moisture sensitive materials as a Part 2 to the standard. Martin Rides said that for testing moisture sensitive materials there are a number of issues such as drying time, temperature and testing under nitrogen that must be considered. He stated that one of the driving forces behind the moisture sensitive materials development was to address the issue of characterising PET recyclate, as well as virgin materials. To address the measurement of moisture sensitive materials more stringent temperature tolerances are considered necessary by the Netherlands. However, Martin Rides had expressed concern that the proposed temperature tolerances would not be achievable by all melt flow rate instruments. A method for preparing compacted charges for melt flow rate testing, also under development in ISO, was presented. It was discussed whether or not there was a need for a rotational rheometry standard: support was expressed for the development of standards for rotational rheometry (a published standard for oscillatory rheometry, drafted by NPL, already exists ISO 6721-10). Martin Rides encouraged all to contribute to the development of standards.

The next six months direction of the project was then outlined, including the investigation of slip using the flow visualisation cell and the intercomparison of slip velocity measurements by capillary rheometry. The effect of slip flow on processing is to be modelled, and standards activity will be progressed.

6. HEAT TRANSFER IN POLYMERS (Project MPP 7.1)

Martin Rides gave an outline of the importance of heat transfer to polymer processing, and outlined the tasks in the DTI project and the major objectives.

A description of the line source thermal conductivity apparatus was given along results on the methods repeatability. Martin Rides discussed the importance of having thermal conductivity data as functions of both temperature and pressure, and stated that Moldflow modelling software now had the facility to input multiple thermal conductivity values for a material. However, the Campus database does not contain thermal conductivity values. Examples of thermal conductivity data for amorphous and semi-crystalline polymers and the benefits of thermal conductivity data obtained at elevated temperature and pressure have been outlined in a paper accepted for publication in ‘Polymer Testing’. A point was made that thermal conductivity values obtained for fire modelling are based on room temperature measurements. Therefore thermal conductivity data obtained at elevated temperatures would be of interest to fire simulation modellers.

Martin Rides then described the heat transfer coefficient (HTC) apparatus and its application for measuring both thermal conductivity and heat transfer coefficient values. The technical specification and the physical set up of the HTC apparatus were explained, and the heat transfer coefficient defined. The method of testing and analysis used to determine the thermal conductivities of materials and heat transfer coefficients is based on a thermal resistance model.

Martin Rides presented thermal conductivity data obtained at 20MPa pressure and 70°C using the line source probe technique and data obtained at ambient pressure and 70°C using the heat transfer coefficient technique. There was good agreement between the two sets of data, allowing for pressure differences. The line source probe and heat transfer coefficient apparatus will be further compared in order to gain increased confidence in, and understanding of the two methods and discrepancies observed in the literature for behaviour above the glass transition temperature. Different thicknesses of PTFE sheet has a small effect on thermal conductivity values, but this variation was considered to be within the instrument’s current repeatability. The effect of incorporating an air gap in the HTC on the measured thermal resistance was presented. The thermal resistance of an air gap was found to be equivalent to a thickness of polymer approximately 10 times greater than that of the air gap. A video clip modelling the behaviour of the air gap was shown. Martin Rides commented that a meeting had been arranged with the head of the Moldflow test laboratory with a view to incorporating the outputs of the project into the Moldflow software.

The case study at Corus was described. It was suggested that maybe it would be possible to work on developing a standard for measurement of thermal conductivity using DSC as a technique. Martin Rides replied that there is some concern over the reproducibility of the technique but that if there is sufficient interest in developing a standard in this area then future work could possibly be carried out to do so. Martin Rides said a report would be written on the thermal conductivity/DSC technique.

Martin Rides then outlined the case study at Zotefoams where foam sheets are distorting, thereby forming wave profiles. It is considered that this is due to internal stresses due to the cooling process. Discussions of appropriate case studies for the follow on project will be held off-line.

The work on standards was summarised. Currently several thermal conductivity standards are under development and the need for a further one specific to polymers using the line source probe method is to be investigated by Martin Rides. If required a proposal will be made at the next ISO meeting. It was stated that an intercomparison of thermal conductivity methods will be undertaken in the following project. This will examine repeatability and reproducibility of the methods, and allow the user to be directed to the most appropriate test method. PMMA was recommended as a reference material by Ian Robinson as it is not injection moulded and so is subject to minimal internal stresses.

Looking at areas of future work it was asked how Moldflow software handles an air gap. A comment was made that the air gap may in fact be a vacuum. Martin Rides said a vacuum gap will be investigated, if that is possible, and discussed with Moldflow.

Interest was expressed in the measurement of the heat transfer coefficient across the solid/air interface formed during film blowing where there is moving air.

No objections were raised to experimental work on heat transfer coefficient measurements using different metal surface finishes and mould materials being discontinued.

A general comment was made that anything discovered in the group is made immediately available to everyone else in the IAG. In response, the projects are funded by the DTI for the benefit of UK industry. UK companies get a lead-time from participating in the work and have the added advantages that they can steer the work of the project and supply materials for testing, thus providing greater benefit to them. Specific details of materials are generally not made publicly available, and companies do not have to reveal to the rest of the IAG details that are confidential.

7. AOB & DATE OF NEXT IAG MEETING

There was no other business to discuss.

Peter Cox thanked everyone for attending this IAG meeting and invited all to attend the next one (details below) before closing the meeting. The next IAG meeting will be held on:

Wednesday 29 March 2006 at a location to be confirmed.

An agenda will be circulated one month prior to the meeting. All are invited.

8. DOCUMENTS AND PRESENTATIONS AVAILABLE

The presentations made and the documents distributed at the meeting are available on the website:

www.npl.co.uk/npl/cmmt/polyproc

using the following username and password

USERNAME: polyprociag_member PASSWORD: poly30056

9. ACTION ITEMS

9.1 NPL to contact the National Space Centre as a possible venue for a future meeting.

9.2 NPL to re-run FEA of MPP7.9 component part with more realistic friction values.

10. DISTRIBUTION

To all invited IAG members.

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AGENDA

7th Industrial Advisory Group (IAG) Meeting

Wednesday 19 October 2005
at the National Physical Laboratory,
Teddington, TW11 0LW
(Module 16)

10-30	Introductions	*Peter Cox, Chairman*
10-40	Minutes of the previous meeting	*Peter Cox, Chairman*
10-50	Characterisation Programme Formulation	*Bruce Duncan*
11-10	Date/venue of next meeting	*Peter Cox, Chairman*

11-15

Theme 1:
Modelling the Behaviour of Plastics for Design Under Impact
Greg Dean, Louise Crocker

Theme 2:
Flow Properties of Filled Materials & Rheology Standards Update
Martin Rides

12-30 **LUNCH**

13-45 Application of Computational Techniques During the Development of Packaging and Medical Devices — *Darren Hodson, AstraZeneca*

14-15

Theme 1:
Measurement of Softness
Paul Tomlins

Theme 2:
Heat Transfer in Polymers
Angela Dawson, Martin Rides

15-30 Close

Tea/Coffee will be available before the meeting (from 10.00 am) and after the meeting at 3.30 pm.

Further discussions with NPL experts and laboratory visits will be available after 3.30 pm.