NATIONAL PHYSICAL LABORATORY

INDUSTRIAL ADVISORY GROUP MEETING ON MEASUREMENT METHODS RELATED TO THE CHARACTERISATION AND PROCESSING PROPERTIES OF POLYMERIC MATERIALS

Minutes of the 11th Industrial Advisory Group (IAG) Meeting held at RAPRA Technology, Shawbury, Shropshire, SY4 4NR Thursday, 4th October 2007

Participants:

Adrian Kelly	Bradford University
Paul Baines	Brand-Rex Ltd
Rex Beechey	Copperplas Int
Don Fleming	Fleming Polymer Testing & Consultancy
Mark Edwards	Gammadot Rheology
Andi Clements	MIRA
Peter Cox	Peter Cox Associates
Alan George	Porpoise Viscometers
Richard Simpson	RAPRA
John Colbert	RAPRA
Jenny Cooper	RAPRA
Andy Hulme	RAPRA
Jan Czerski	Consultant
Martin Rides	NPL
Angela Dawson	NPL

Apologies for absence were received from:

Brian Powell	Alpha Technology
Mogon Patel	AWE
Paul Morrell	AWE
Roy Carter	Celsum
Dilwyn Jones	Emral Ltd
Barry Hennessey	Ford
Mark Holmes	PERA
Alec Barron	Wartsila UK Ltd

CONTENTS

1. INTRODUCTION AND MINUTES OF THE PREVIOUS MEETING

Richard Simpson welcome all to Smithers RAPRA Technology Ltd.

Peter Cox (Peter Cox Associates) welcomed all participants to the Industrial Advisory Group (IAG) Meeting on Industrial Advisory Group Meeting on Measurement Methods Related to the Characterisation and Processing Properties of Polymeric Materials, funded by the DTI NMSD Materials research programmes.

Martin Rides commented that the delegates meeting pack held copies of all but one of the presentations. All of the presentations would be available on the web shortly after the meeting from the link.

http://resource.npl.co.uk/materials/polyproc/iag/

USERNAME: polyprociag_member PASSWORD: poly30056

The minutes of the 10th IAG meeting held on 18th April 2007 at NPL were accepted as written. They are obtainable from:

http://resource.npl.co.uk/materials/polyproc/iag/april2007/minutes5.doc

Martin Rides reported that all the actions of those Minutes had been completed with the exception of Action 1. He reported that the intercomparison had not yet been carried out due to the desire to run it after the larger international intercomparison being organised by NPL in conjunction with Japan. This Action was continued.

ACTION 1: NPL to initiate an intercomparison on thermal conductivity with Avon Rubber and TARRC. Avon/TARRC to supply materials/samples (Action continued from last meeting).

2. SMITHERS RAPRA, RECENT HISTORY AND CURRENT CAPABILITIES

See presentation - *Introduction_IAG#11.pdf*

Jenny Cooper described Smithers Rapra's history and current capabilities. This was followed by a tour during lunch to visit various test facilities.

3. CHARACTERISATION OF THE FLOW PROPERTIES OF FILLED MATERIALS AND UPDATE ON STANDARDS (H4 & U4)

See presentation - *H4_U4_Rheology_IAG#11.pdf*

Martin Rides described the project H4: Flow properties of filled materials (2005-08) in which measurement methods are being investigated and developed for melt flow rate testing of moisture sensitive materials, characterising fast curing systems (including by extrusion rheometry), gels and the mix quality of materials, and the use of piezoelectric techniques.

Martin Rides 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. The standard was now at CD ballot and is likely to be published in 2009/2010.

Martin Rides reported in detail on the results of an intercomparison in support of development of the melt flow rate standard for moisture sensitive materials, which the NPL had organised, and additional in-depth NPL testing on the materials used in the intercomparison. The results suggest that the

moisture is the most important of the parameters to control to obtain repeatable and reproducible results. The repeatability of MVR and MFR for moisture sensitive materials was estimated to be up to 25% and for reproducibility of up to 250%, after outliers had been removed (95% confidence level), indicating poor practice in drying and/or controlling exposure of the sample to moisture.

Martin Rides reported that for the PET material MVR values of up to approx 1000 cm^3/10 minutes were obtained when the sample had not been dried and commented, following a question, that in this case foaming of the extrudate due to the absorbed moisture had occurred. He commented that in this case the recommended drying time of 4 hours was insufficient and had to be extended to 5 hours to ensure adequate drying of the sample.

Martin Rides reported that in the current revision of the melt flow rate standard ISO1133 an annex was being prepared on the preparation of a consolidated charge. In answer to a question, he commented that this was aimed, in particular, at testing samples where the particles had a high aspect ratio and therefore would not necessarily pack well, e.g. flakes of shredded, recycled polymers. The objective is to produce a near-solid charge of material as free from air bubbles as possible thereby improving the data obtained. It also, potentially, facilitates the introduction of the sample into the melt flow rate instrument's cylinder.

Peter Cox asked whether some intercomparison participants used desiccant dryers? Martin Rides commented that the laboratories used hot air or vacuum dryers. Peter Cox questioned whether drying time and routine also need to be specified to which Martin Rides commented that perhaps a measure of moisture content was preferable as the drying conditions would be dependant on the efficiency of the drying equipment in use. Also, the drying conditions should be specified by the materials standards. It was commented that it was possible to avoid, or at least minimise, uptake of moisture by using a nitrogen blanket. However, it was desirable to produce a standard that could be adopted and implemented at reasonable cost.

Further questions on the repeatability and reproducibility of the method were asked to which Martin Rides commented that he now intended to analyse further the results of the intercomparison to provide precision data on the method. He commented that some of the participants had not carried out testing in compliance with the method and that these data had to be removed from the analysis for that reason. He commented that he did not consider the very tight temperature control to be a significant factor in influencing precision but considered that the drying and subsequent handling of the samples to be the critical factor. In terms of the drying, rather than specifying drying conditions the actual moisture content should be specified.

ACTION 2: NPL to reanalyse MVR/MFR intercomparison data.

In response to a question from Peter Cox, Martin Rides commented that the considered that melt volume flow rate (MVR) was a more fundamental measure of the rheological behaviour of the polymers that the melt mass flow rate (MFR) as the MFR was also dependant on the material's density. Alan George commented that MVR relates much closely to molecular weight. He added that to obtain density one could use the melt flow rate instrument by extruding a known volume of material and weighing it. For conversion of MVR to MFR for PP a universal melt density of 0.758 g/m^3 is quoted in the material specification standard. Martin Rides commented that industry is increasingly utilising MVR.

He then briefly reported on the use of the piezoelectric device for viscosity measurement, with good capability for in-process or in-situ measurement.

Martin Rides reported on the development of a disposable extrusion rheometer that could be used for testing curing systems, and presented results obtained using it including against results obtained by oscillatory rheometry. In particular he reported on its use in a controlled stress mode, compared with the normal extrusion rheometry controlled rate mode. He commented that this provided much clearer

information of the processability of the material under investigation. He finally commented on some rheometry testing of gel materials that exhibited complex behaviour involving slip arising due to the composition of the materials (very high water content).

Andi Clements asked whether batch-to-batch variation had been considered. Martin Rides commented that for the purposes of the intercomparison single batches of each of the materials had been used in order to assess the precision of the method for a given material. Andi Clements commented that MVR and MFR values are often given as a range, with 5% often quoted but for thermoplastic elastomers it can be up to 25%. Martin Rides commented that if the requirement is for $\pm$ 20% then should be able to measure to $\pm$ 2%. This would require a temperature accuracy to approximately $\pm$ 0.4% in the worst case for these materials. John Colbert commented that he knew of specifications for (2.0 $\pm$ 0.5) g/10 minutes so deliveries of 1.7 g/10 mins and 2.3 g/10 mins are both within the specification. Alan George commented that one can get discrepancies between the machine set temperature and the die/fluid temperature. Furthermore, if a draft occurs (i.e. a door is opened) one can observe temperature fluctuations and so he considered a requirement of $\pm$ 0.4 °C as being quite demanding. Martin Rides commented that temperature tolerance of $\pm$ 0.5°C is specified in the current ISO 1133. Andi Clements asked whether a greater spread in MVR values was expected for high MVR materials? Martin Rides commented that for moisture sensitive materials one would anticipate both the repeatability and, in particular, the reproducibility (between laboratories) to be worse due to different practices. For example, if the length of piston travel varies between laboratories then shorter piston displacements will result in shorter residence times and therefore less degradation. Consequently as there are more sensitivities in testing moisture sensitive materials there will be greater variation in results. It is for these reasons that the standard is, and needs to be, limited to high MVR tests and that tighter time limits are imposed. However for stable materials there was not expected to be a relatively greater spread in results for high MVR materials compared with low MVR materials.

4. MATERIALS 2007+ AND 2008+ PROGRAMMES

See presentation - *Materials_2007&08_IAG#11.pdf*

Martin Rides briefly described some of the projects in the current Polymers and Composites portfolio, recently commenced projects (Materials & Thermal Metrology 2007+ NMSD programme) and finally some proposals for the Materials 2008+ programmes. He highlighted three projects in particular on *Dispersion in polymeric nanocomposites / Traceable size & shape measurements on nanoparticles, Micro-scale characterisation of the mechanical properties of polymeric materials,* and *Characterisation of polymeric fluids for micro-scale processing and fabrication.* He welcomed comment on these proposals.

He also commented that the formulation was a rolling process and recommended that ideas were submitted at any time, rather than waiting for a formal request.

Adrian Kelly asked about DIUS funding. Martin Rides suggested that he either contacted Bill Nimmo of NPL or directly with Bill Martin of DIUS.

5. MEASUREMENT METHODS FOR HEAT TRANSFER PROPERTIES DATA FOR APPLICATION TO POLYMERS (H1) AND UPDATE ON STANDARDS (H1)

See presentation - *H1_Thermal_IAG#11.pdf*

Angela Dawson outlined the project then described in detail measurements using the heat transfer coefficient (HTC) apparatus to determine the effect of interfaces, including that of a polymer - mould and polymer - air gap - mould on heat transfer. Results indicated that an air gap had the same thermal

resistance as that of the polymer layer approximately 6 times thicker. She also described the method used for determining values for the heat transfer coefficient across a polymer-metal interface representing that occurring in moulding practice and presented a recently obtained result. Planned work entailed further measurement of the thermal resistance of the polymer-mould interface and modelling to investigate the sensitivities of predictions to uncertainties in the input parameters for thermal conductivity and heat transfer coefficients.

ACTION 3: Angela Dawson to discuss with Andrew Hulme their modelling work on effects of heat transfer on moulding simulations, in the context of the proposed NPL modelling activity.

Martin Rides then briefly described the standardisation activities that were progressing in ISO. He reported results obtained in an intercomparison of thermal conductivity and thermal diffusivity of PMMA samples using a range of transient and equilibrium techniques. He commented that contributions to the standardisation process by industry were welcome.

Finally, Martin Rides commented that ISO 11357-1 on DSC was being revised and again welcomed comment.

6. A RECIPE FOR ENERGY MANAGEMENT

John Colbert (jcolbert@rapra.net) reported on the EU project *RECIPE* (*Reduced Energy Consumption In Plastics Engineering*). Extensive project details are provided at www.eurecipe.com. He presented the results of the survey carried out into energy usage in plastics processing across Europe. He tabled a European Best Practice Guide *Low Energy Plastics Processing* produced as an output of the project (available through the website link above). He then reported on the *Flowfree* project that focuses on the use of supercritical fluid (CO_2) for extrusion of sheet and profile. Finally he reported on the *PEPTFlow* project on the use of positron emission particle tracking for flow visualisation in extruders.

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.

The next IAG meeting will be held on:

Wednesday 12 March 2008 at NPL (Bushy House), Teddington, TW11 0LW

This date has been confirmed since the meeting. An agenda will be circulated one month prior to the meeting. Details of the meeting will be posted on the website:

http://www.npl.co.uk/server.php?show=ConWebDoc.2043

All are invited to attend the meeting (contact: Lydia Solomon, NPL).

8. DOCUMENTS AND PRESENTATIONS AVAILABLE

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

http://resource.npl.co.uk/materials/polyproc/iag/

using the username ***polyprociag_member*** and password ***poly30056***

9. ACTION ITEMS

ACTION 1: NPL to initiate an intercomparison on thermal conductivity with Avon Rubber and TARRC. Avon/TARRC to supply materials/samples (Action continued from last meeting).

ACTION 2: NPL to reanalyse MVR/MFR intercomparison data.

ACTION 3: Angela Dawson to discuss with Andrew Hulme their modelling work on effects of heat transfer on moulding simulations, in the context of the proposed NPL modelling activity.

10. DISTRIBUTION

IAG members and NPL staff (by downloading from the NPL website)

AGENDA

MEASUREMENT METHODS RELATED TO
THE CHARACTERISATION AND PROCESSING PROPERTIES
OF POLYMERIC MATERIALS
Industrial Advisory Group (IAG) Meeting

Thursday 4 October 2007
at RAPRA Technology
Shawbury, Shrewsbury, Shropshire, SY4 4NR

Chairman: Peter Cox, Peter Cox Associates

10-30	Welcome and Introductions	*All*
10-40	Minutes of the previous meeting	*Peter Cox*
10-50	Smithers Rapra, Recent History and Current Capabilities	*Jenny Cooper*
11-20	Characterisation of the flow properties of filled materials and update on standards (H4 & U4)	*Martin Rides/Crispin Allen*
12-00	Materials 2007+ (2007-10) & Materials 2008+ (2008-11) programmes	*Martin Rides*
12-20	**LUNCH + Tour**	
13-40	Measurement methods for heat transfer properties data for application to polymers (H1) and update on standards	*Angela Dawson, Martin Rides*
14-20	European Research Projects on Energy Reduction	*John Colbert*
14-50	Date/venue of next meeting (*Proposed 12 March 2008 at NPL*)	*Peter Cox*
14-55	AOB	*Peter Cox*
15-00	Close	

Tea/Coffee will be available before the meeting (from 10.00 am) and after the meeting at 15.00.

Further discussions with RAPRA and NPL experts will be possible after 15.00.

For details of the venue see RAPRA and how to get there see travel details.