National Physical Laboratory

Heat Transfer in Polymers

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19 October 2005

Heat Transfer in Polymers - Summary

- Introduction
- Thermal Conductivity
- Heat Transfer Coefficient
- Industrial Demonstrations: Corus, Zotefoams
- Standards for Thermal Properties Measurement
- Outline of Heat Transfer Project 2005-08
- Summary

- AOB

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Heat transfer is:

- key to polymer processing
- still inadequately understood
- key to increasing throughput - process times dominated by the cooling phase
- significant in affecting product properties, e.g. warpage, inadequate melting, thermal degradation

Aim of the project

- To help companies measure and model heat transfer in polymer processing
- To enable measurement of more accurate data and encourage the use of improved models for numerical simulation software
- This should lead to:
  - Right first time design
  - Reduce cycle times / higher productivity (faster processing)
  - Energy saving
  - Fewer failures in service
  - Reduce waste- e.g. reduce warpage and hot spots during processing

Resulting in reduced costs and improved quality

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Tasks in the DTI Project

- Heat Transfer Coefficient
  - New facility
- Thermal Conductivity
  - Uncertainty analysis
  - Extension of method to new materials
- Simulation
  - To identify the important data
  - To help design equipment
  - Moldflow & NPL's own software
- Industrial Demonstrations
  - Zotefoams
  - Corus
- Dissemination
  - Web site, IAGs, PAA Newsletter articles, trade press articles, measurement notes, scientific paper

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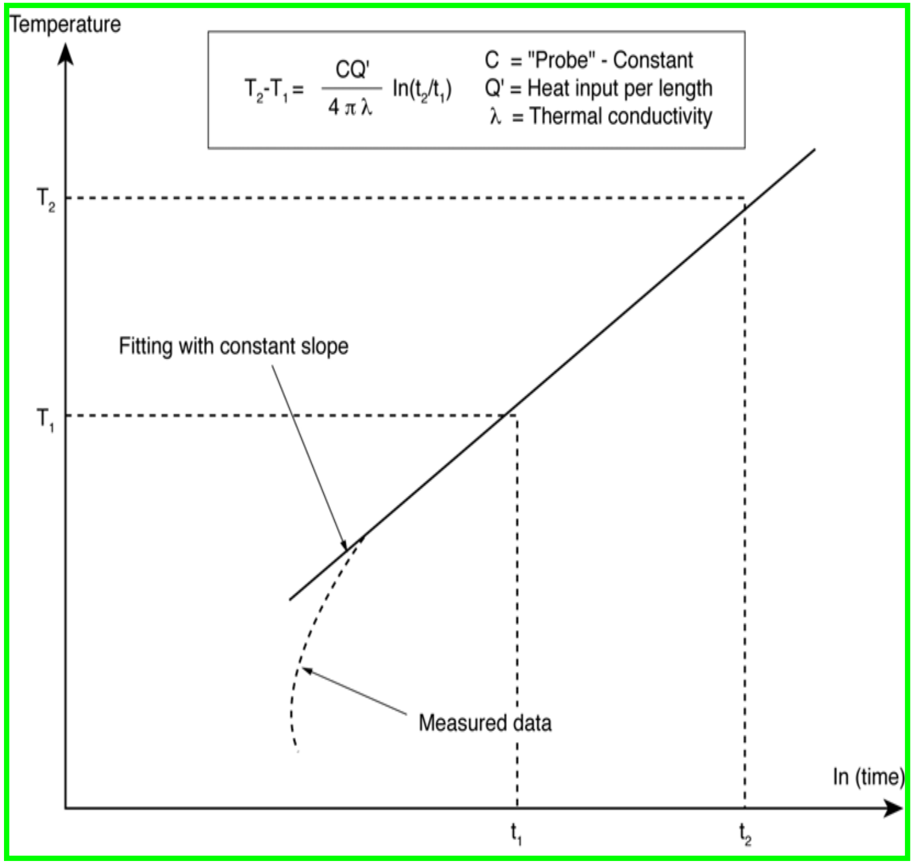
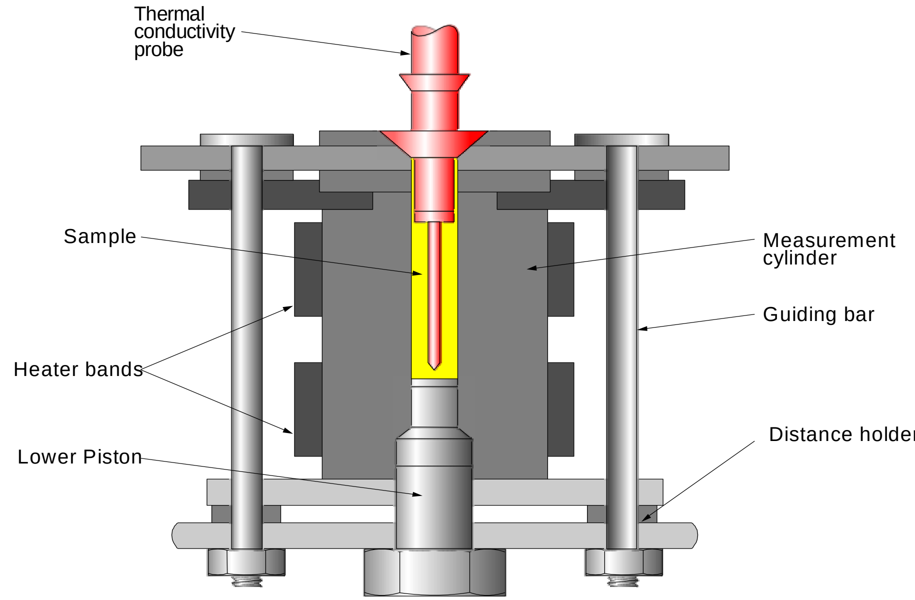
Related Eureka Project: AIMTECH

- An associated Eureka project (AIMTECH) is progressing
- Its aim is to improve productivity of injection moulding
  - Main focus is on the moulds - reduce cycle times by using copper alloy moulds in injection moulding
- NPL Role
  - Measurement of the thermal conductivity of polymer melts (T,P)
  - Understanding the role of the mould/melt interface: Modelling heat transfer and the effect of uncertainties
- Six UK companies involved
- £25k co-funding contribution
- Close fit with the DTI project

Thermal conductivity measurements

Line source probe apparatus





Measures thermal conductivity at industrially relevant processing pressures and temperatures

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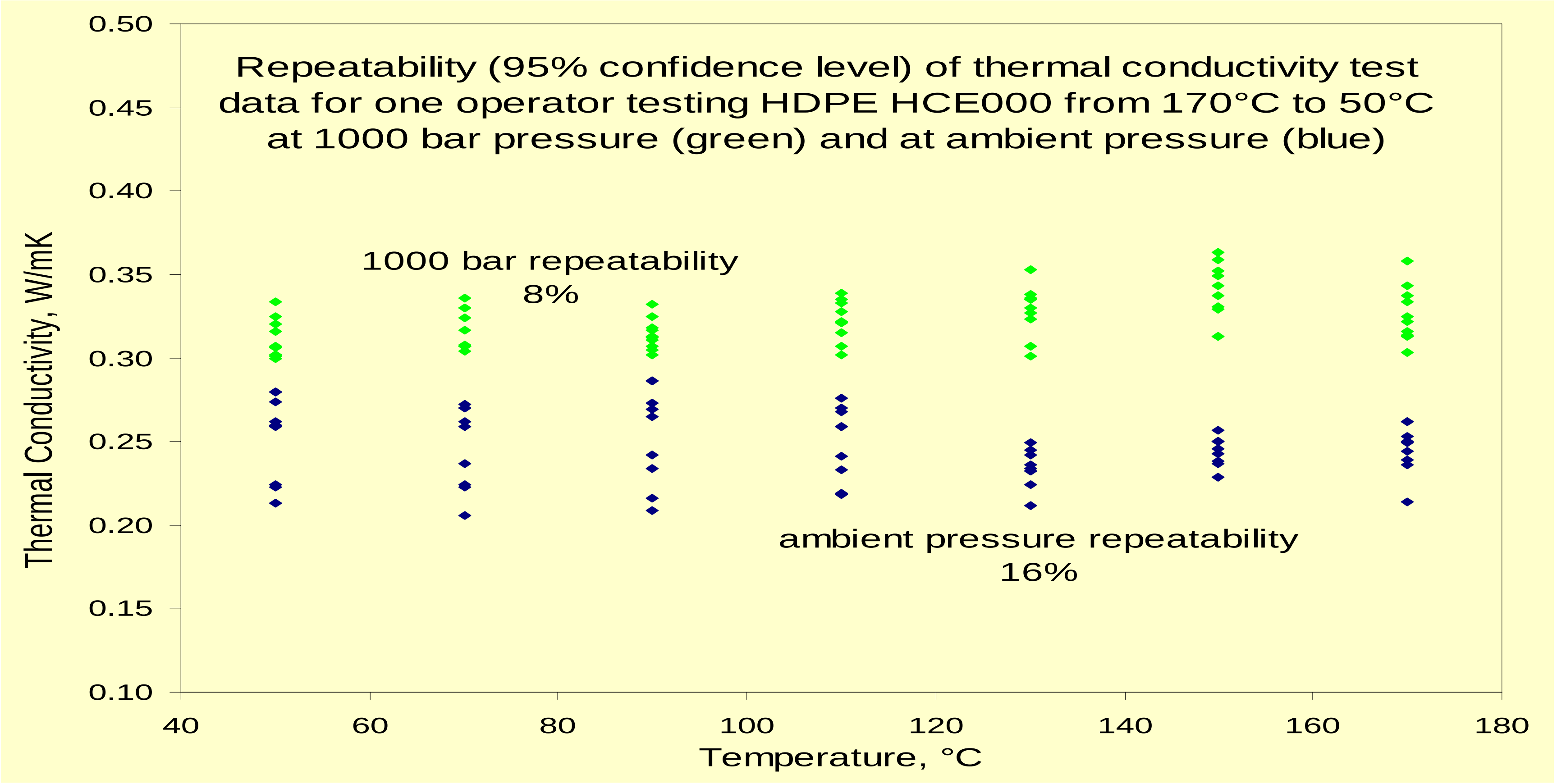
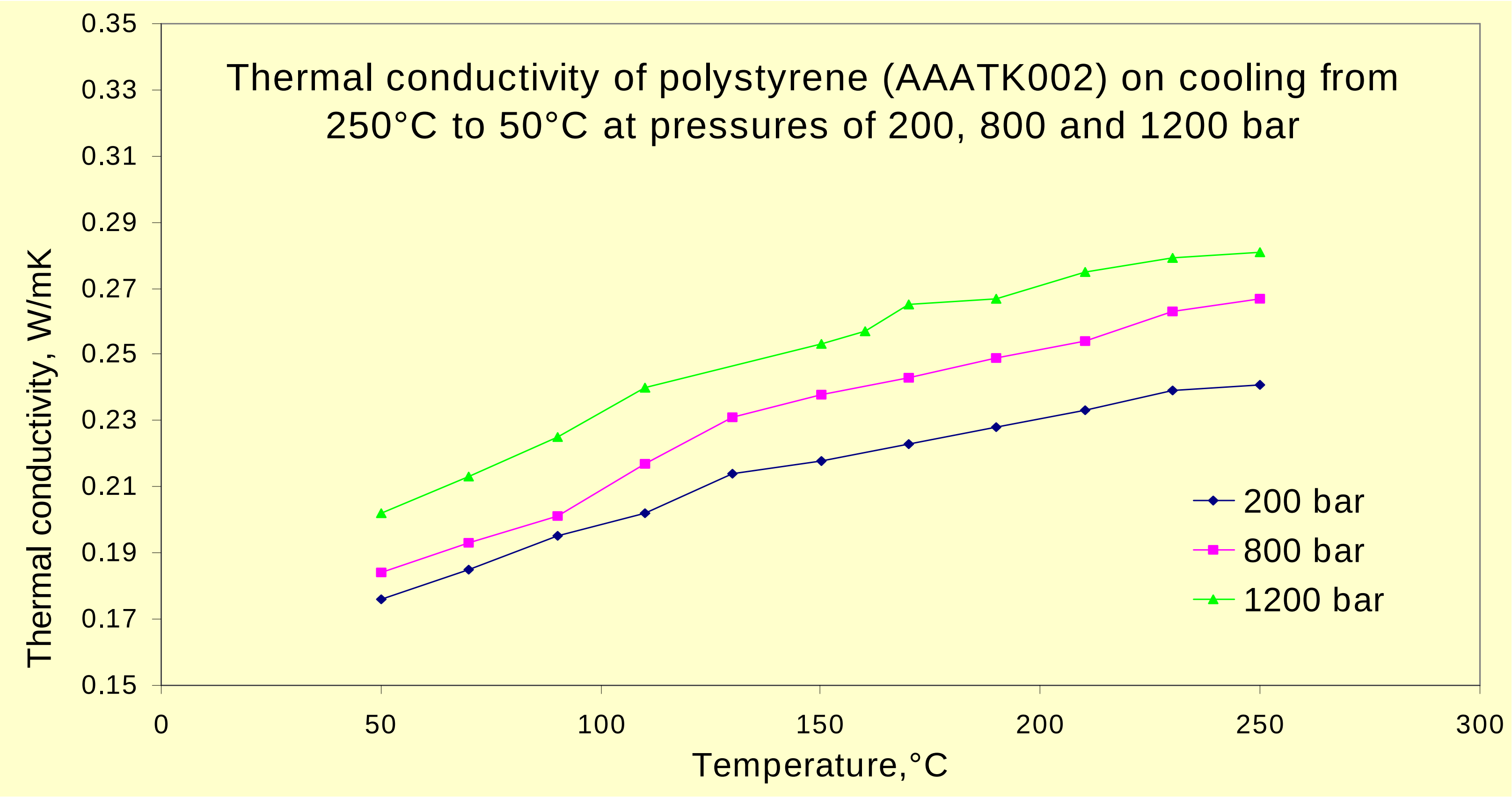
Thermal conductivity

HDPE at atmospheric pressure and 1000 bar



Thermal conductivity

Amorphous polystyrene (PS) under pressure



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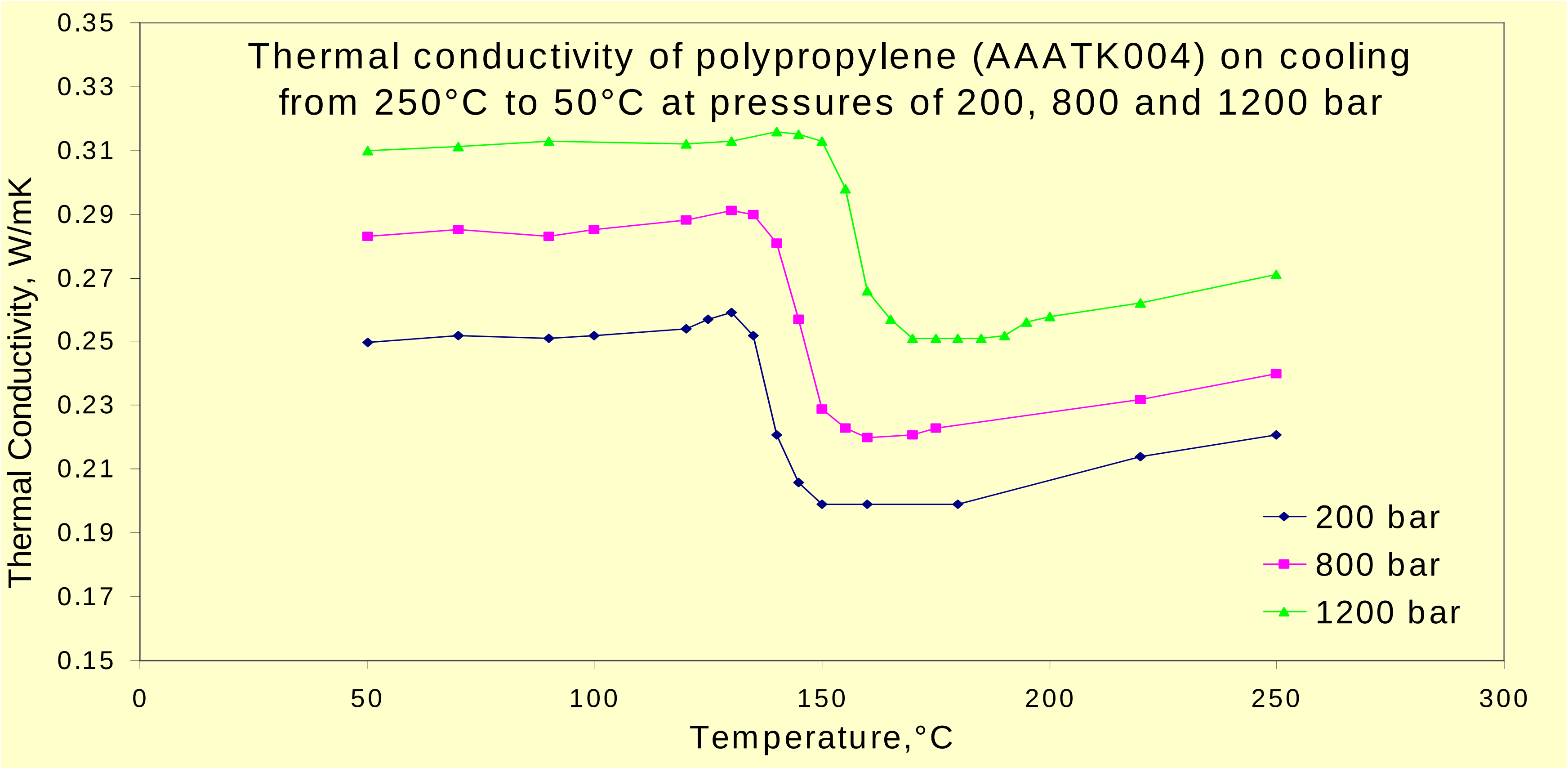
Thermal conductivity

Semi-crystalline polypropylene (PP) under pressure



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Thermal conductivity: implications of results

- Increase in pressure gives increase in thermal conductivity and increase in crystallisation temperature for semi-crystalline polymers

- Thermal conductivity data strongly dependent on both pressure and temperature:
  - 50% over range 50°C to 250°C
  - 43% over range 200 bar to 1200 bar

  Implies single-point thermal conductivity value may be in error by up to 50%

- Important to use pressure and temperature dependent thermal conductivity data for accurate process modelling – will improve, for example, warpage and hot-spot prediction

- More accurate data based on industrial processing conditions - improvements in commercial modelling packages -– possible cost benefits

Paper accepted by
'Polymer Testing'

The Effect of Pressure on the Thermal Conductivity of Polymer Melts

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

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Heat transfer equipment for measurement of thermal conductivity and heat transfer coefficient

Heat Transfer Coefficient

- It is the heat flux per unit area (q) across an interface from one material of temperature T_1 to another material of temperature T_2 :

$$h = q/(T_1 - T_2) \qquad \text{units:} \quad Wm^{-2}K^{-1}$$

- Boundary condition for process simulation

- In injection moulding & compression moulding
  - Polymer to metal
  - Polymer-air-metal (GASM, …)

- In extrusion & film blowing
  - Polymer to fluid (eg air or water)

- This project has built apparatus to measure heat transfer coefficient and will investigate the significance of different interfaces to commercial processing

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Heat Transfer Coefficient (heat transfer across an interface)

Features of apparatus

- Room temperature to 275 °C, pressure to at least 500 bar
- Polymer samples 2 mm to 25 mm thick
- Interchangeable top plate to investigate
  - Different surface finishes
  - Effect of mould release agents
- Option to introduce a gap between polymer & top plate
  - Shrinkage, sink marks
- Instrumented with temperature measurement devices and heat flux sensors

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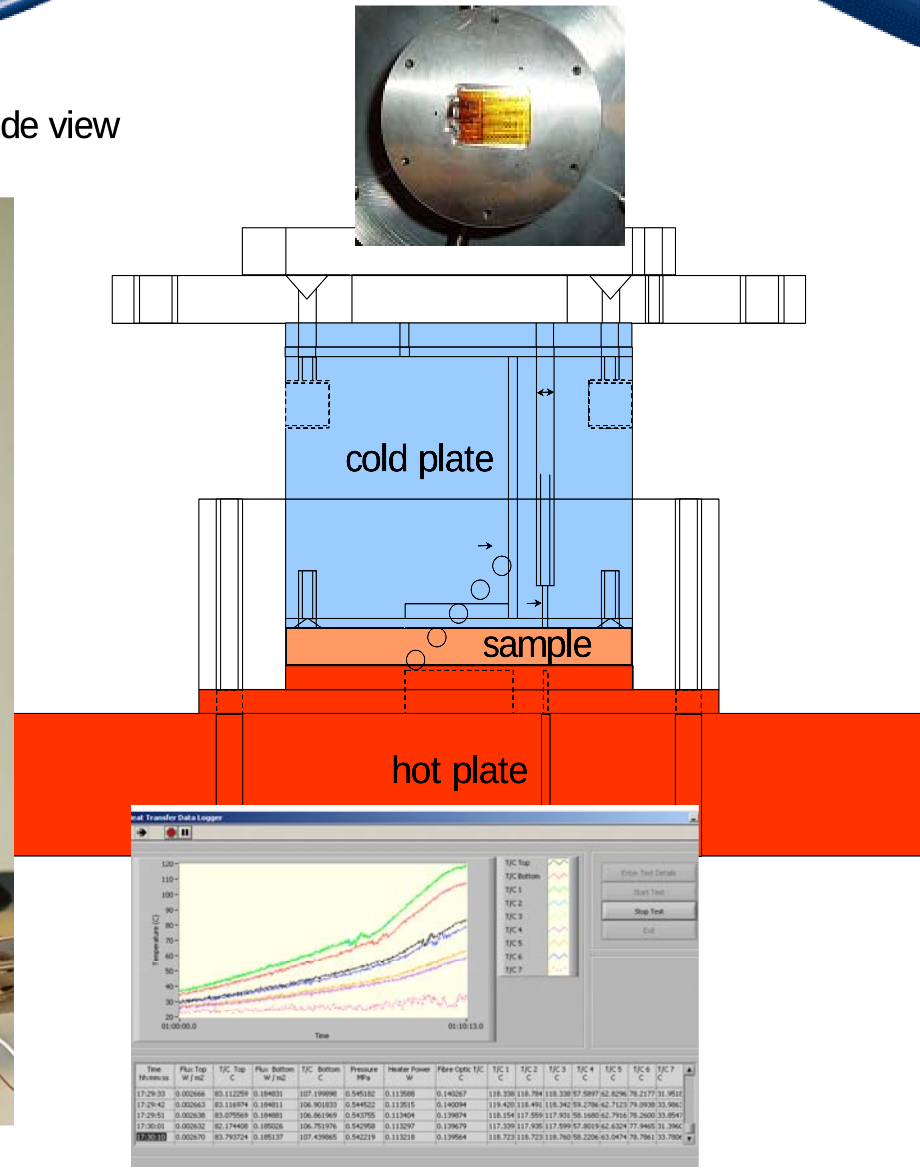
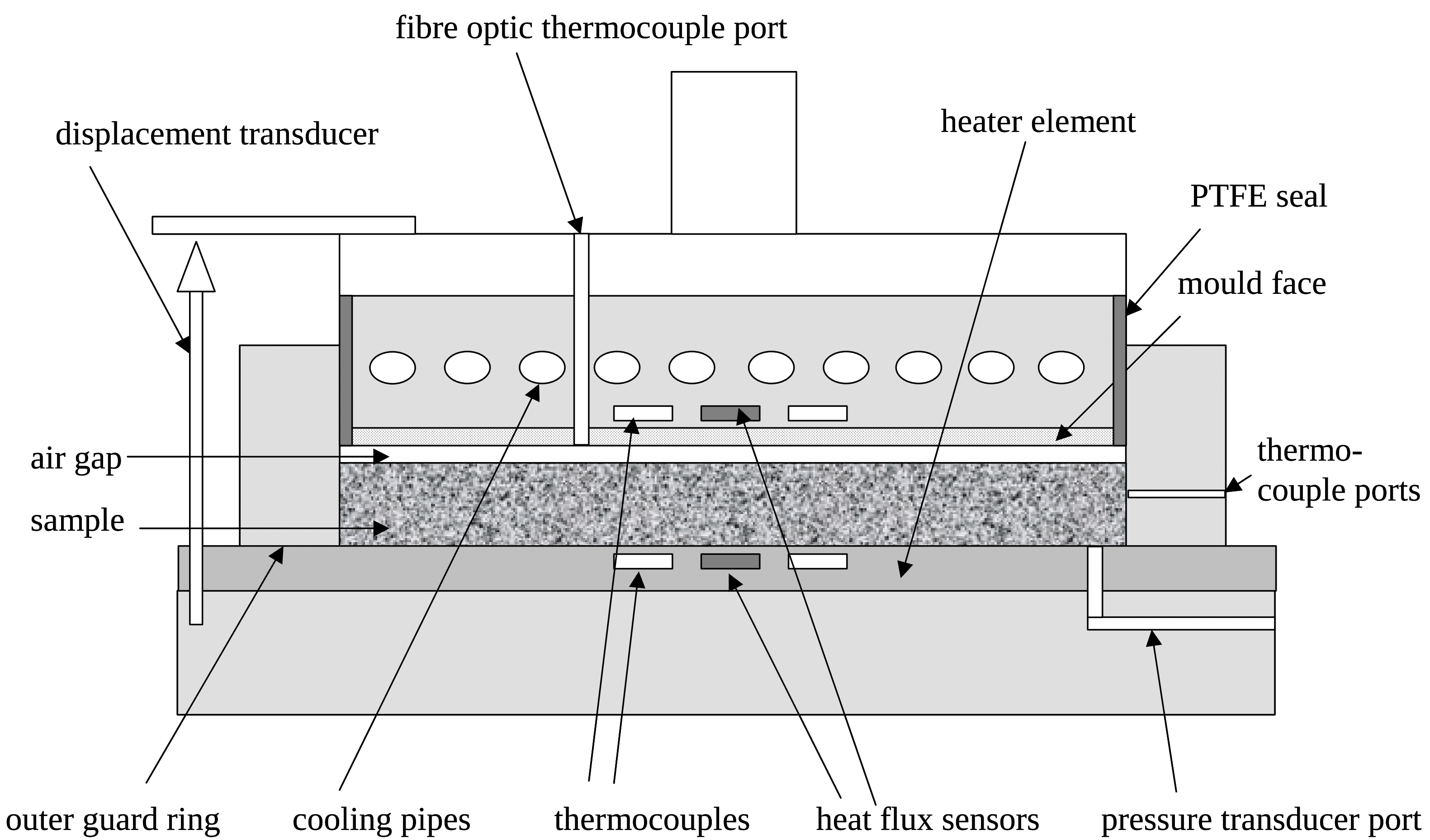
Heat transfer apparatus

Side view



Heat transfer apparatus



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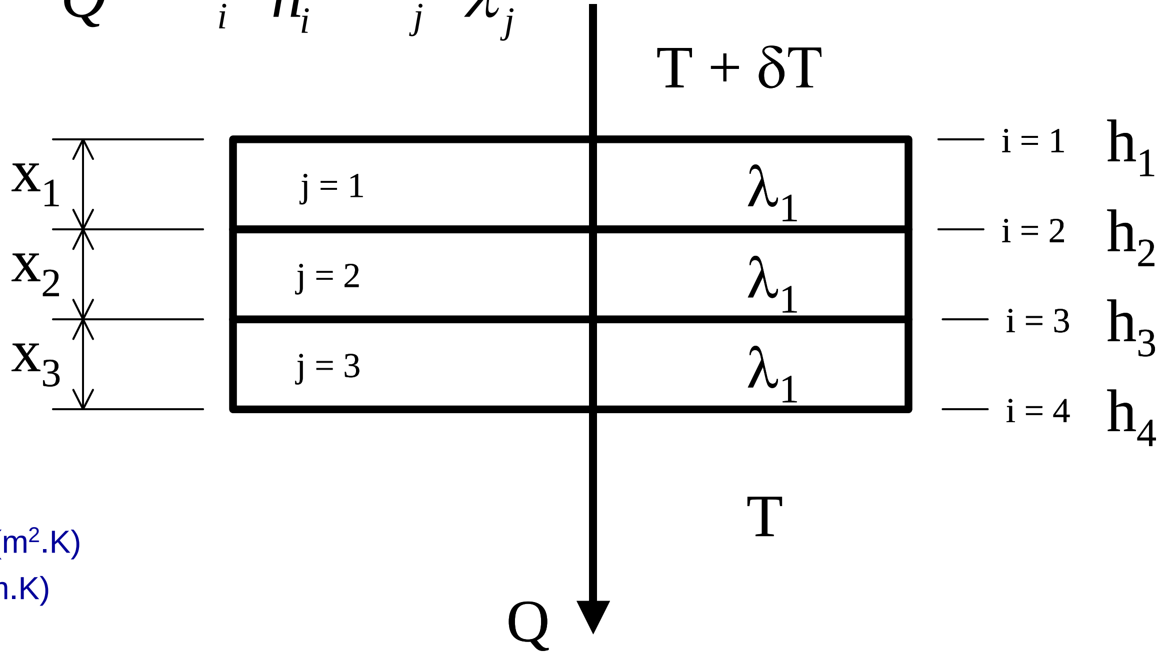
Heat transfer: thermal resistance model

When materials are place in series, their ***thermal resistances R_i (m²K/W)*** are added

$$R = \frac{\delta T}{Q} = \sum_i \frac{1}{h_i} + \sum_j \frac{x_j}{\lambda_j}$$



h – heat transfer coefficient, W/(m^2.K)

λ – thermal conductivity, W/(m.K)

x – thickness, m

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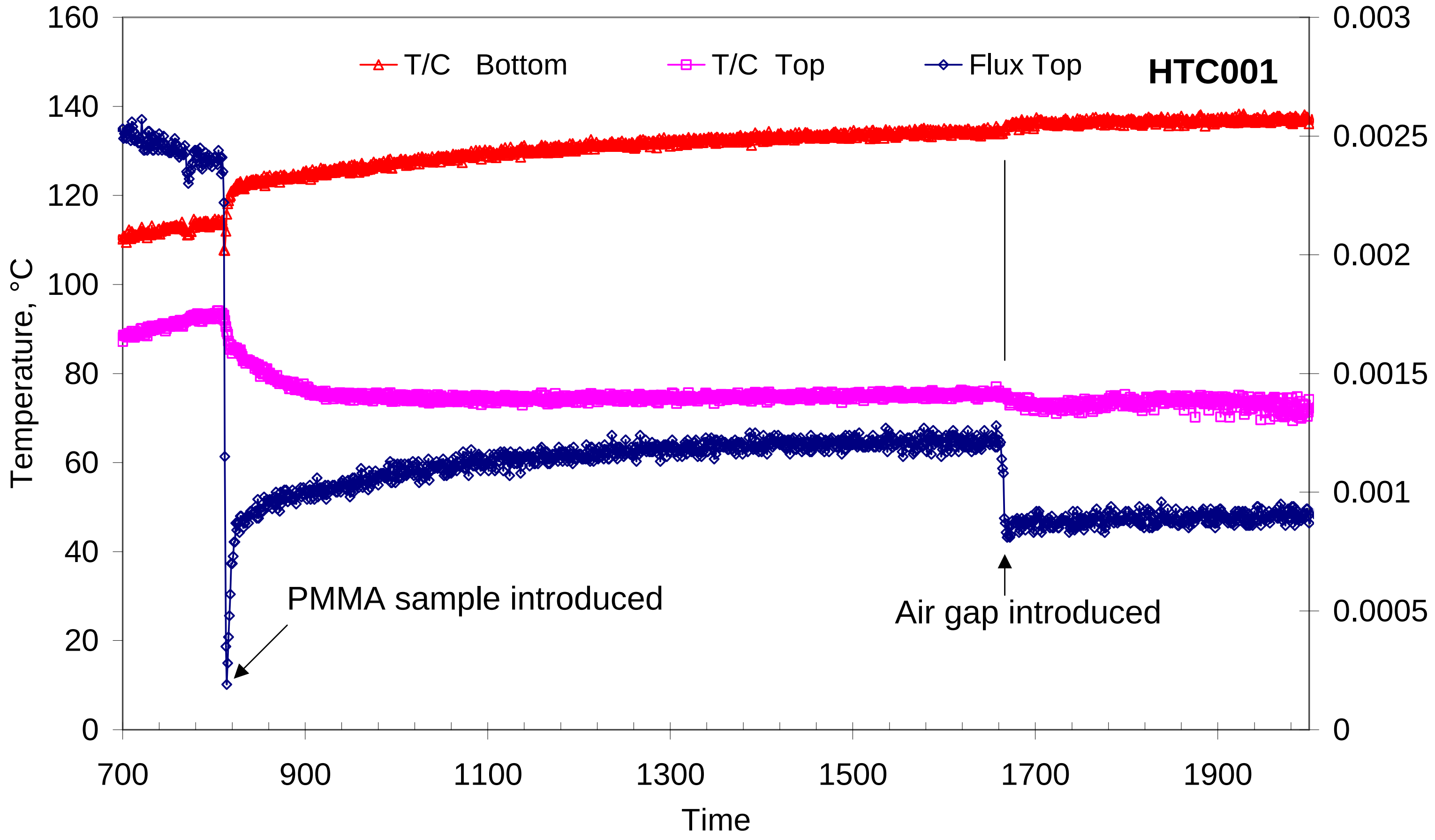
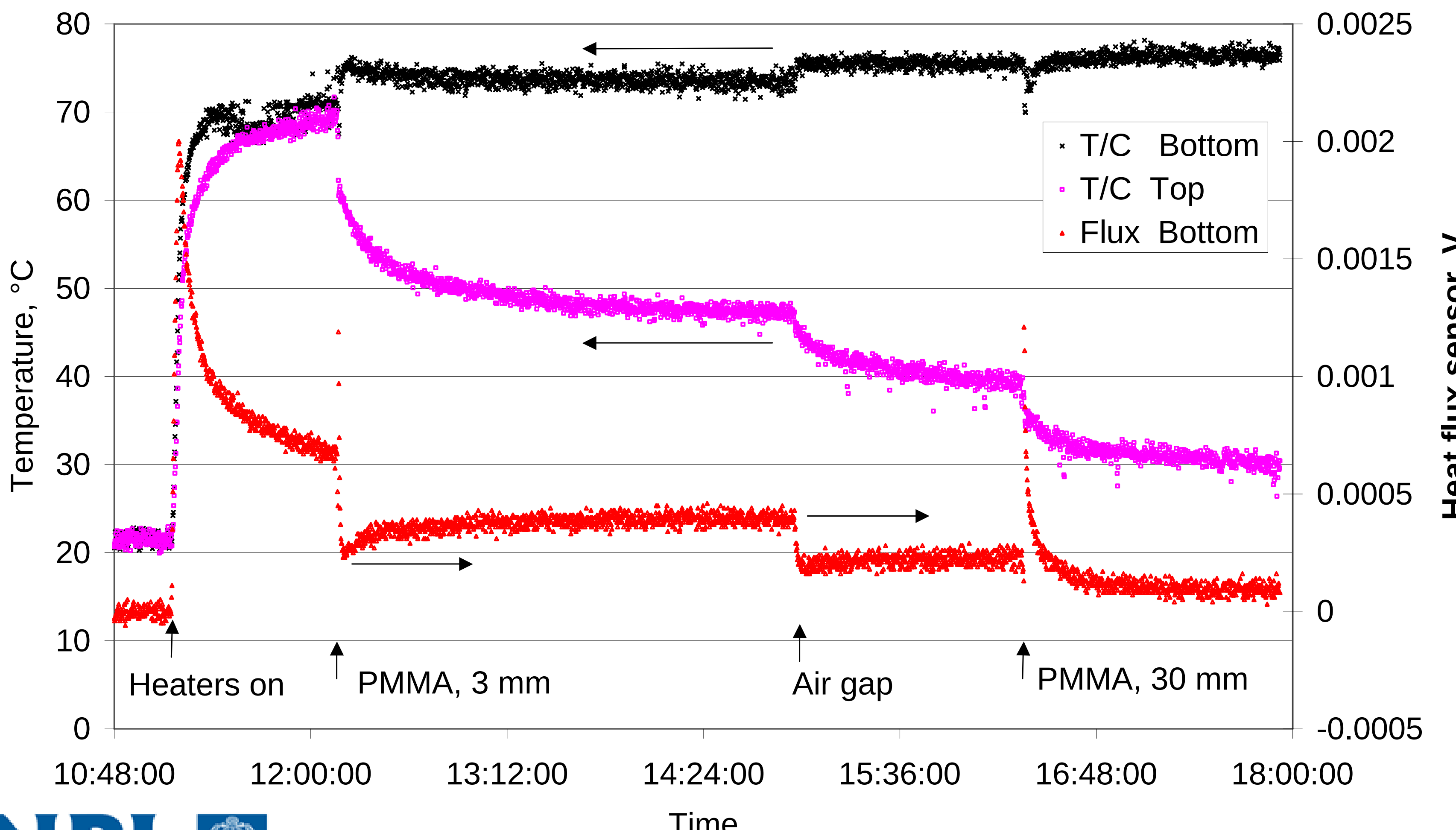
Heat transfer



Heat transfer

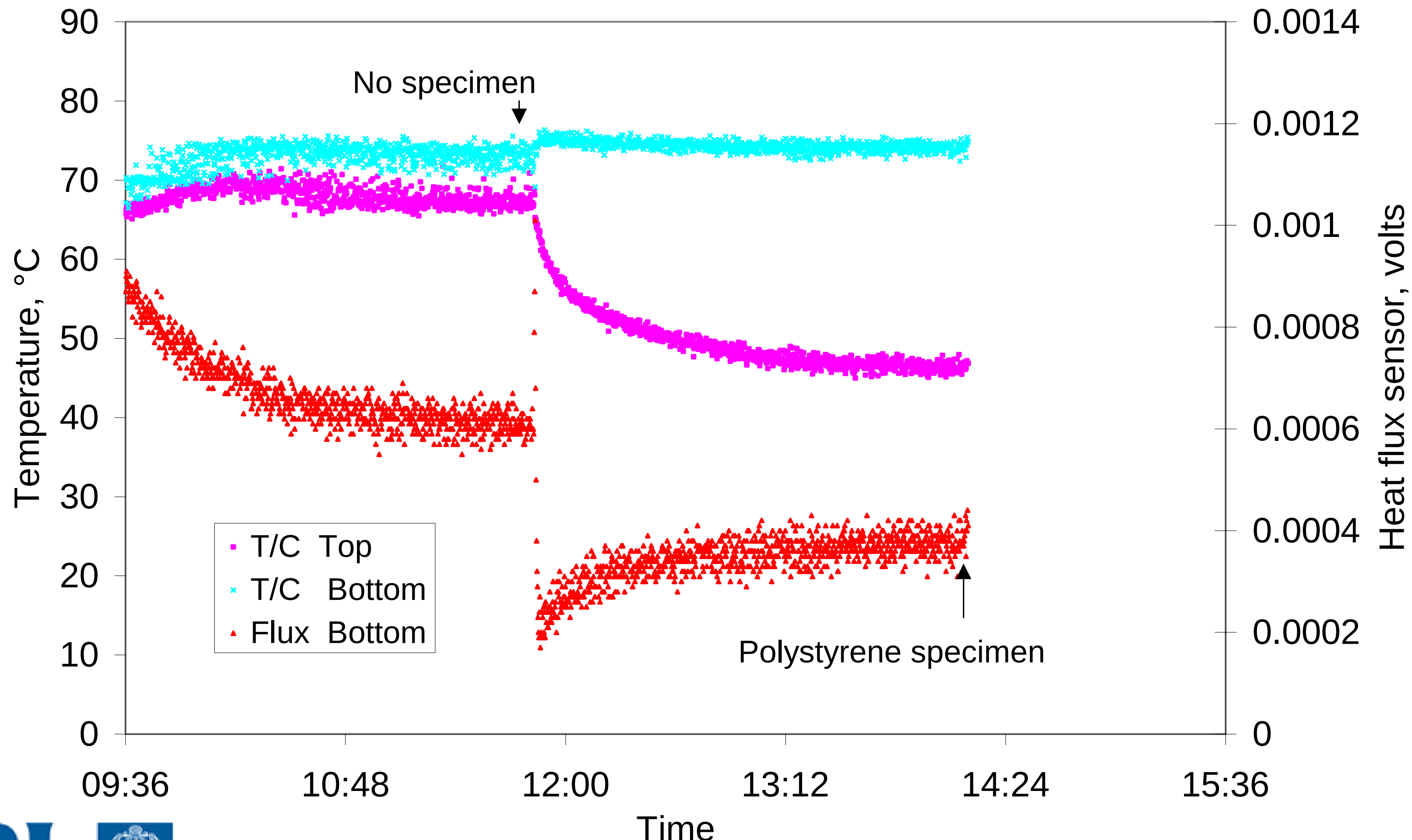


Thermal conductivity measurement

Polystyrene

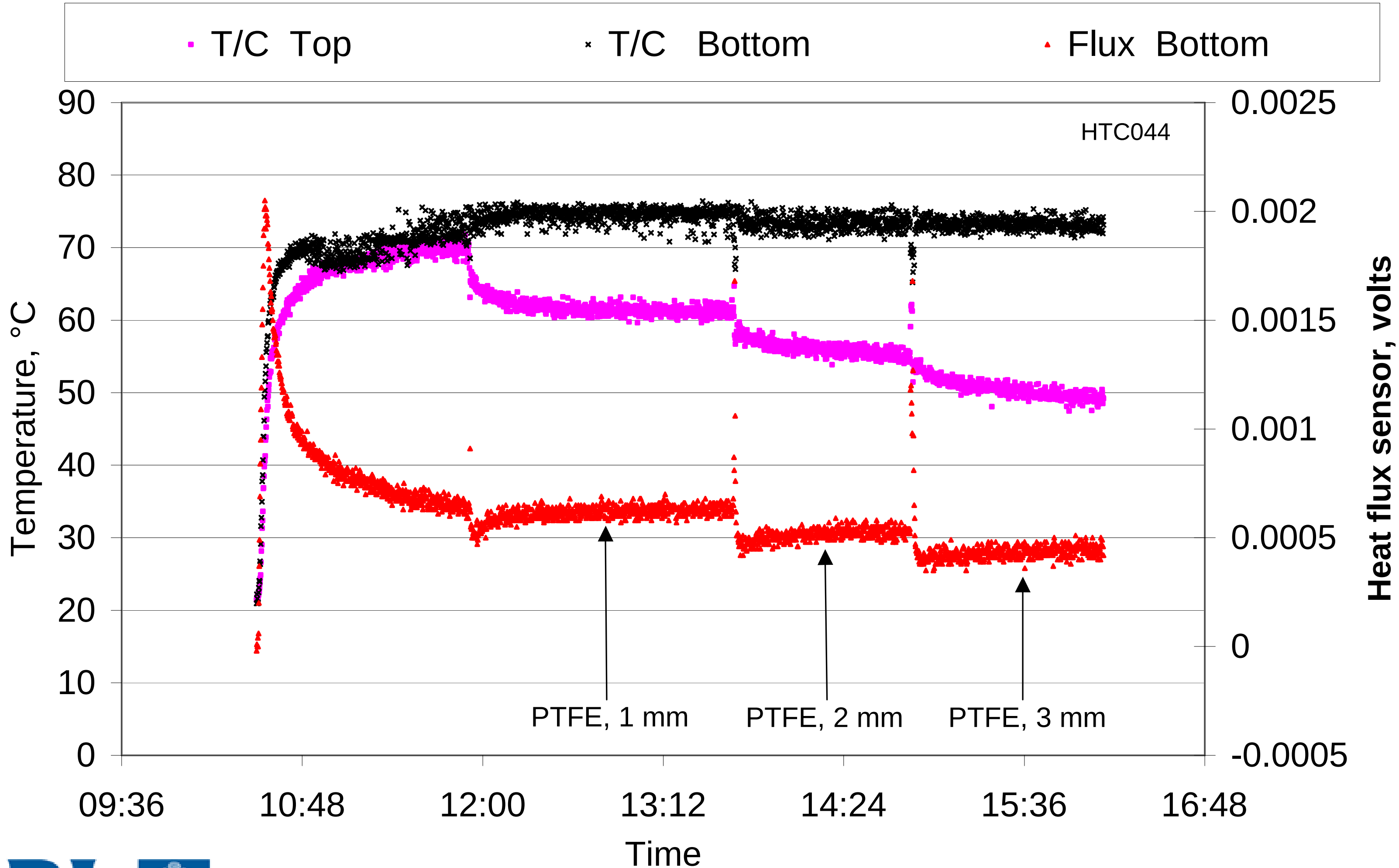
Line source method = 0.185 W/(m.K) at 20 MPa & 70 °C

Heat transfer equipment derived value = 0.170 W/(m.K)



Heat transfer coefficient: effect of specimen thickness

Specimen thickness, mm	1.03	2.15	3.02
Thermal conductivity, W/(m^2.K)	0.25	0.28	0.27



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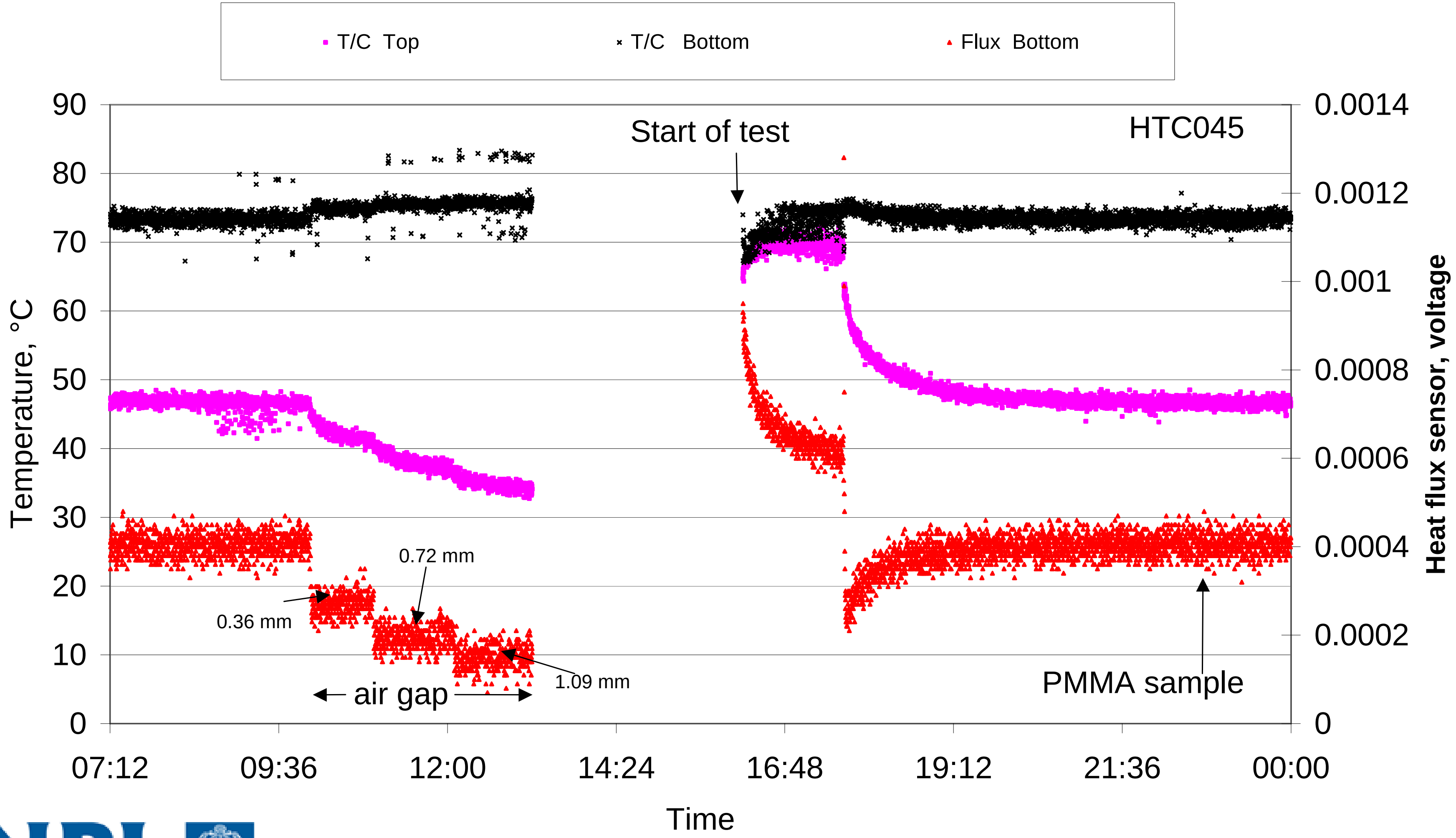
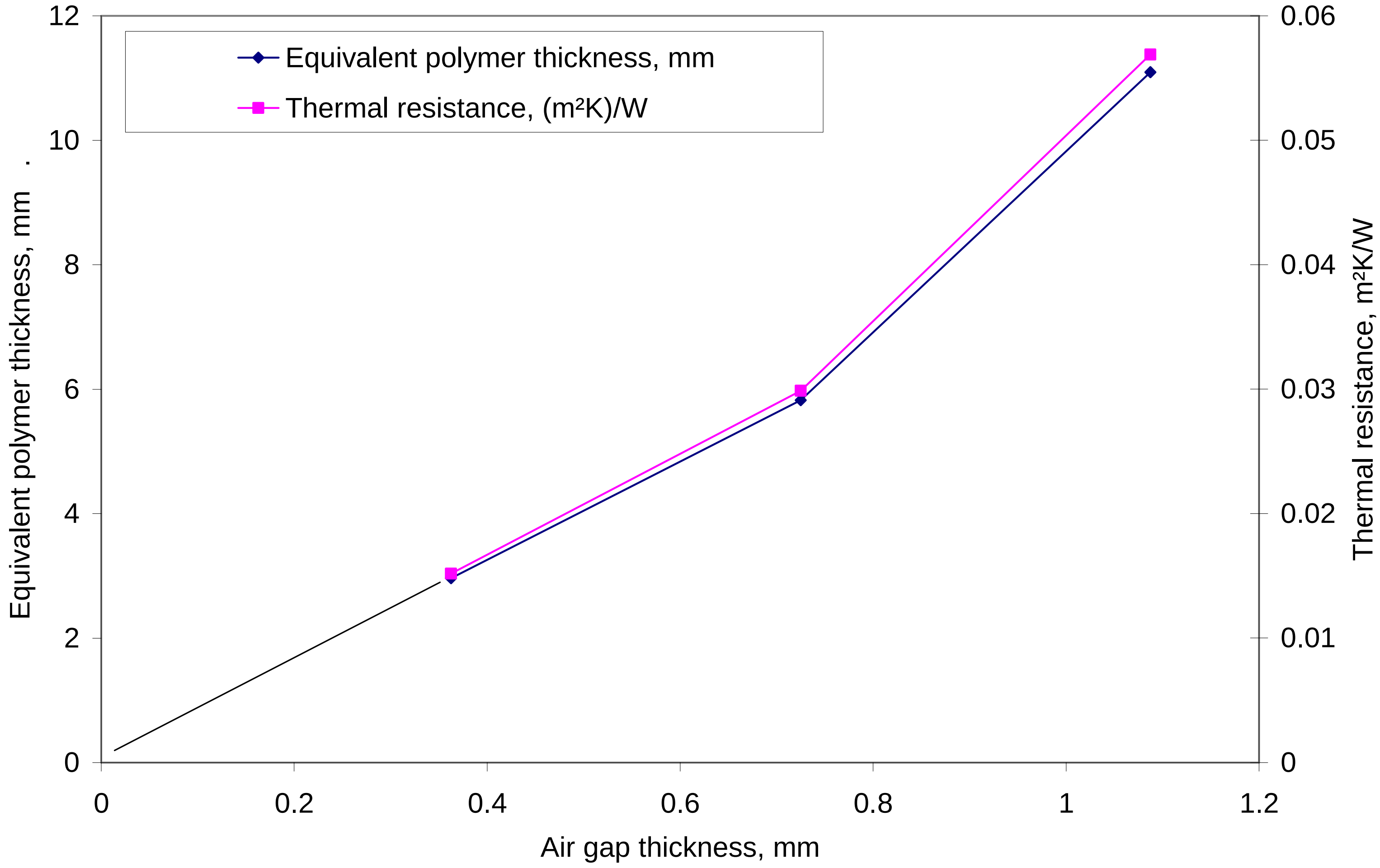
Heat transfer coefficient: effect of air gaps

Air gap, mm	0.363	0.725	1.088
Equivalent polymer thickness, mm	3.0	5.8	11.1
Thermal resistance, (m^2K)/W	0.015	0.030	0.057



Heat transfer coefficient: effect of air gap



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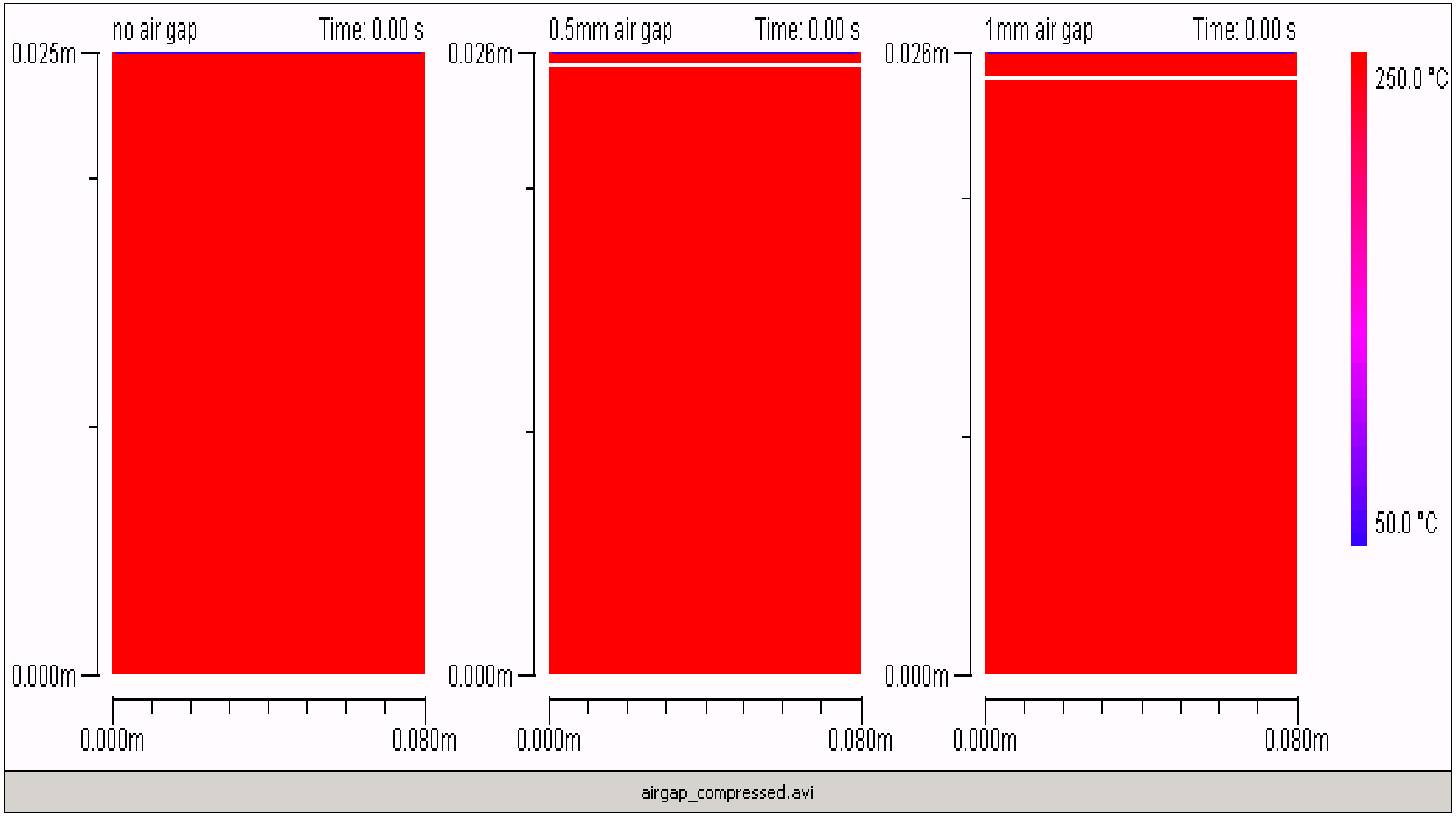
Numerical modelling

- Effect of an air gap
- Effect of vertical thermocouple on distort the temperature field

Air Gap

Mould at 50 °C with air gap of 0, 0.5 & 1 mm



Polymer at 250 °C

Heat Transfer Equipment Summary

- Measurements of thermal conductivity of polymers yielding reasonable values. However, need to
  - address signal noise and improve data analysis procedure
  - further validate instrument through thermal conductivity measurements

- Effect of air gap on thermal resistance quantified

INDUSTRIAL TRIALS
Corus & Zotefoams

Industrial Demonstrations

Aim is to investigate DSC method and demonstrate practical benefits of heat transfer measurements and modelling

- Corus
  - Thermal conductivity of plastisol coated steel before and after solidification
- Zotefoams
  - Heat transfer during cooling of polyolefin foam

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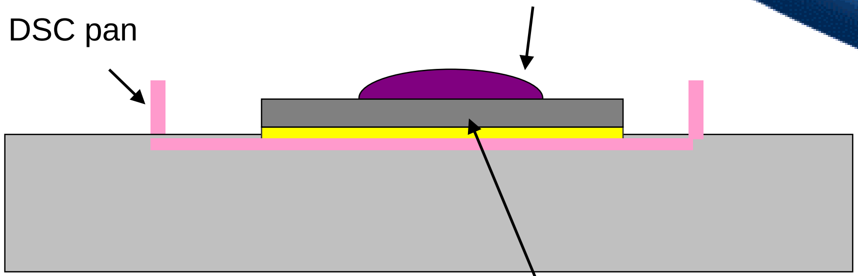
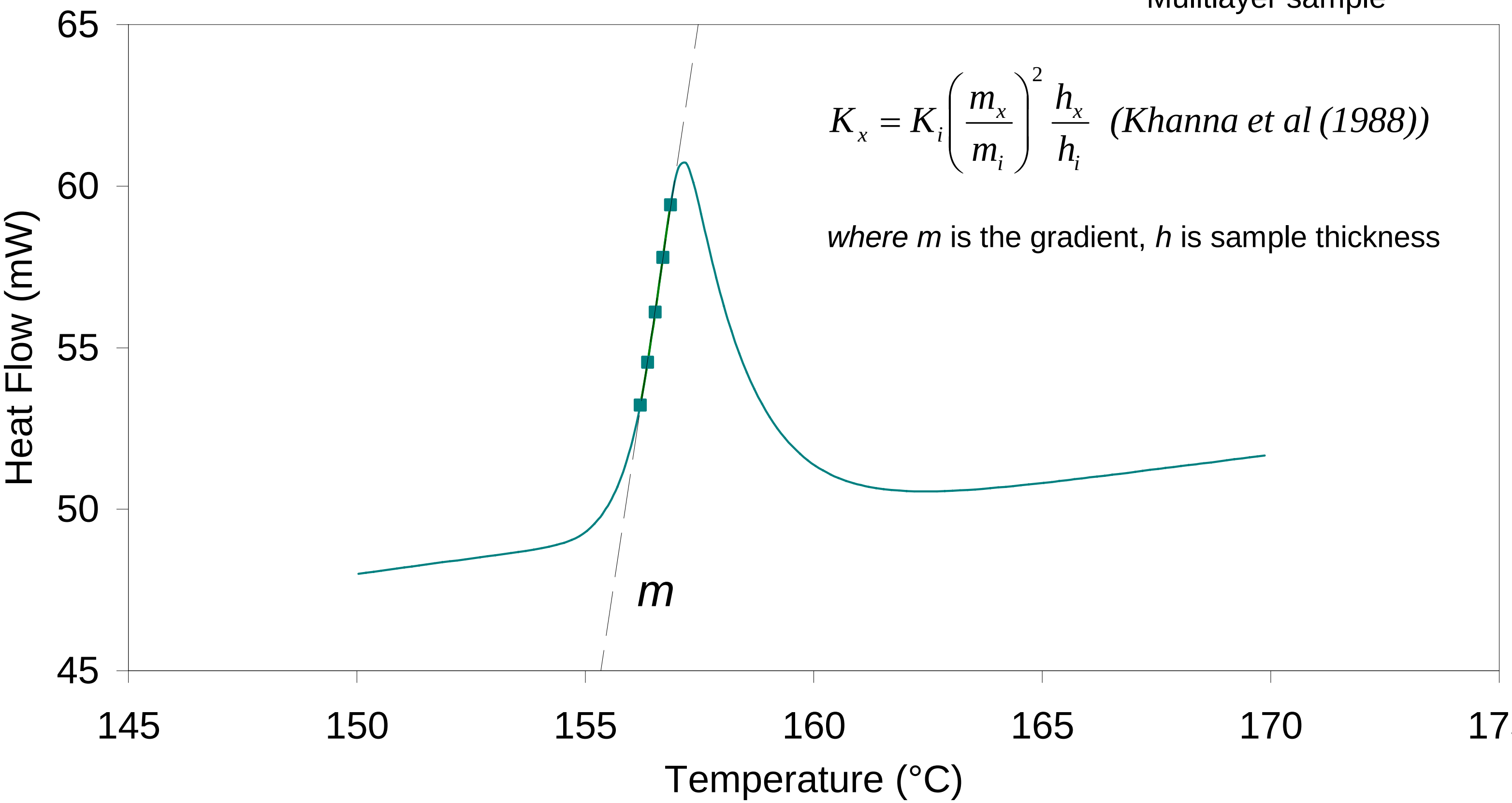
Corus

- Use DSC method to measure thermal conductivity of bilayer Plastisol/steel
- Measure before and after solidification
- Data useful in predicting optimum line speeds
  - Earlier work had shown that the polymer layer was significant in terms of heat transfer

DSC method for thermal conductivity





$$K_x = K_i \left(\frac{m_x}{m_i}\right)^2 \frac{h_x}{h_i}$$ *(Khanna et al (1988))*

where m is the gradient, *h* is sample thickness

DSC method for thermal conductivity

TEST ID	Specimen ID	Specimen gradient mW/°C	Specimen thickness mm	Thermal conductivity value - literature W/mK	Measured thermal conductivity* W/mK	Error
DSC098	PTFE + Indium	2.11	1.026	0.25	0.27	9%
DSC089	PTFE + Indium	2.34	1.027	0.25	0.33	34%
DSC087	PTFE + Indium	2.48	1.030	0.25	0.38	51%
DSC088	PTFE + Indium	2.37	1.030	0.25	0.34	38%
average	PTFE + Indium	2.32	1.03	0.25	**0.33**	33%
95% confidence $					**0.09**	
DSC092	PTFE + Indium	1.77	1.693	0.25	0.32	27%
DSC090	PTFE + Indium	1.75	1.767	0.25	0.32	29%
DSC099	PTFE + Indium	1.61	1.769	0.25	0.27	10%
DSC091	PTFE + Indium	1.77	1.780	0.25	0.33	33%
average	PTFE + Indium	1.73	1.75	0.25	0.31	25%
95% confidence $					**0.05**	
DSC093	ref. PMMA + Indium	1.60	1.208	0.18	0.18	3%
DSC110	ref. PMMA + Indium	1.64	1.208	0.18	0.19	7%
DSC111	ref. PMMA + Indium	1.51	1.208	0.18	0.16	-9%
average	PMMA + Indium	1.58	1.21	0.18	0.18	1%
95% confidence $					**0.01**	

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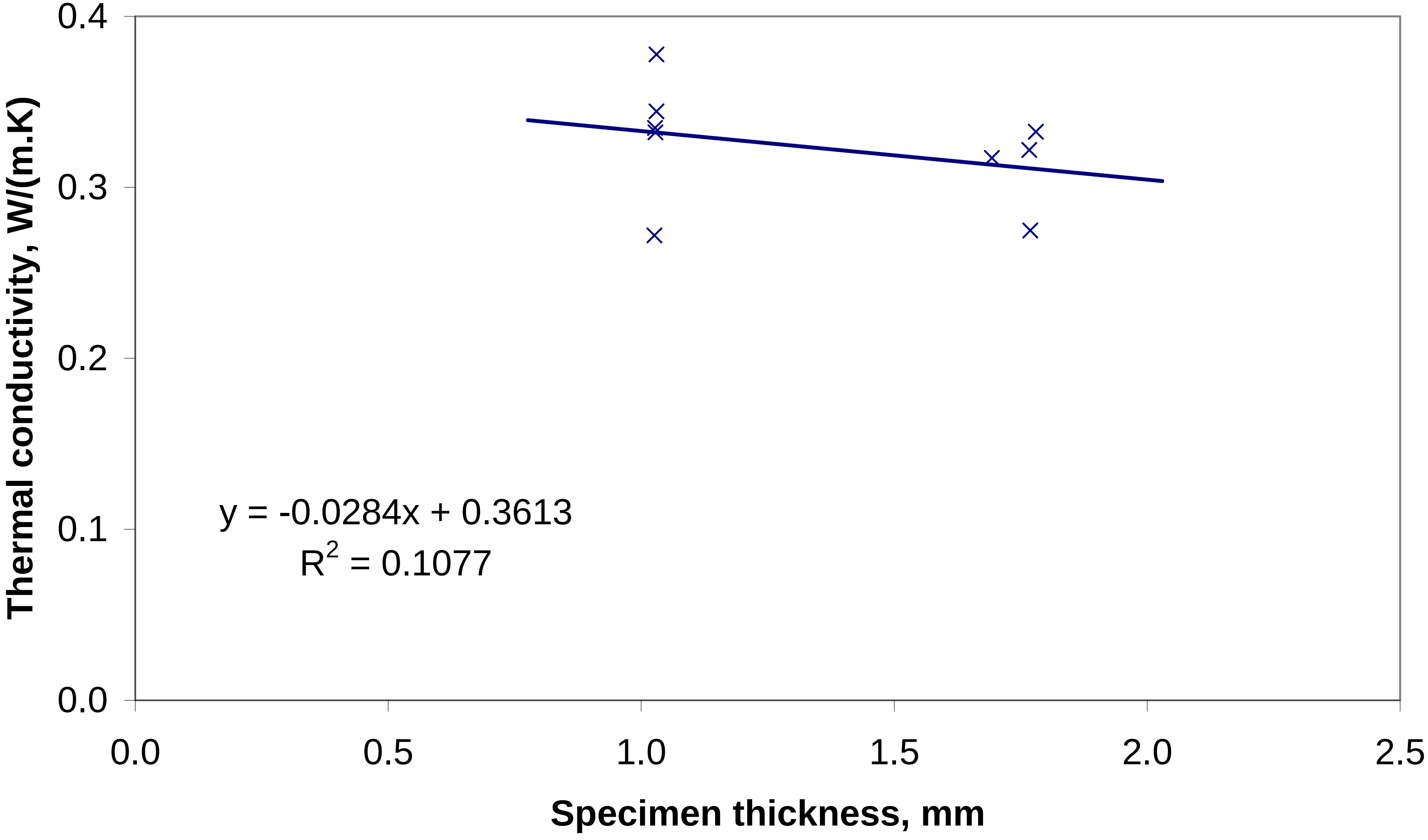
*Calculated assuming thermal conductivity of 0.18 W/(mK) for PMMA reference sample

$ 95% confidence = 2 x standard deviation

DSC method for thermal conductivity



DSC method for thermal conductivity

- Essential to use reference specimen of similar thermal conductivity as specimen to be measured

- Repeatability 95% confidence levels up to approximately 25%

- Essential to carry out several repeat tests to improve on statistical significance of data

- Not appropriate for testing molten/liquid samples (e.g. plastisols) – having constant and known sample thickness

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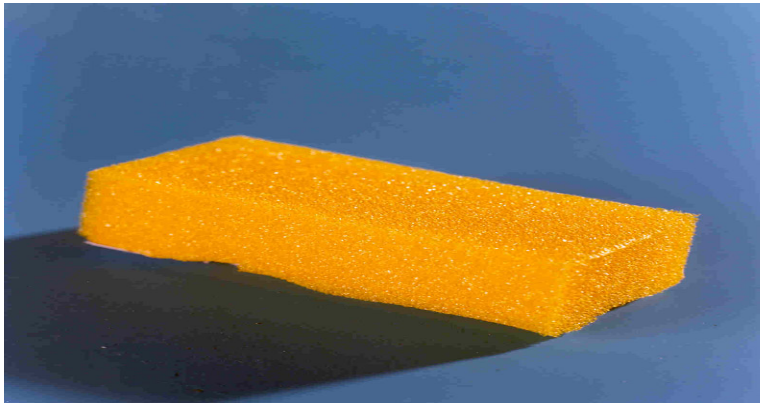
Foams

Problem: waviness in foams – thermal issue

- Model heat transfer
- Measure (T, heat flux) over time
- Measure shrinkage
- Use to predict curvature



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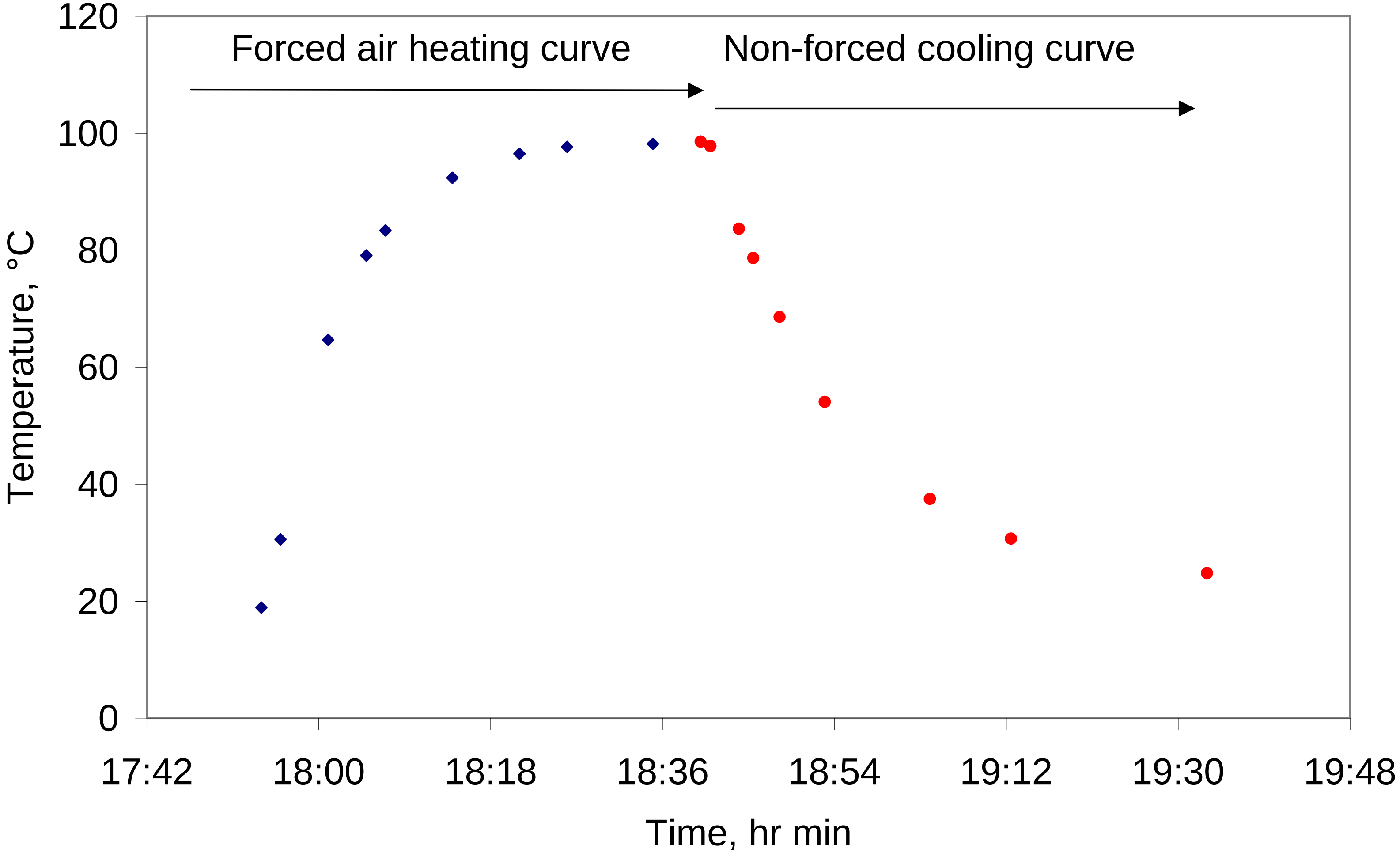
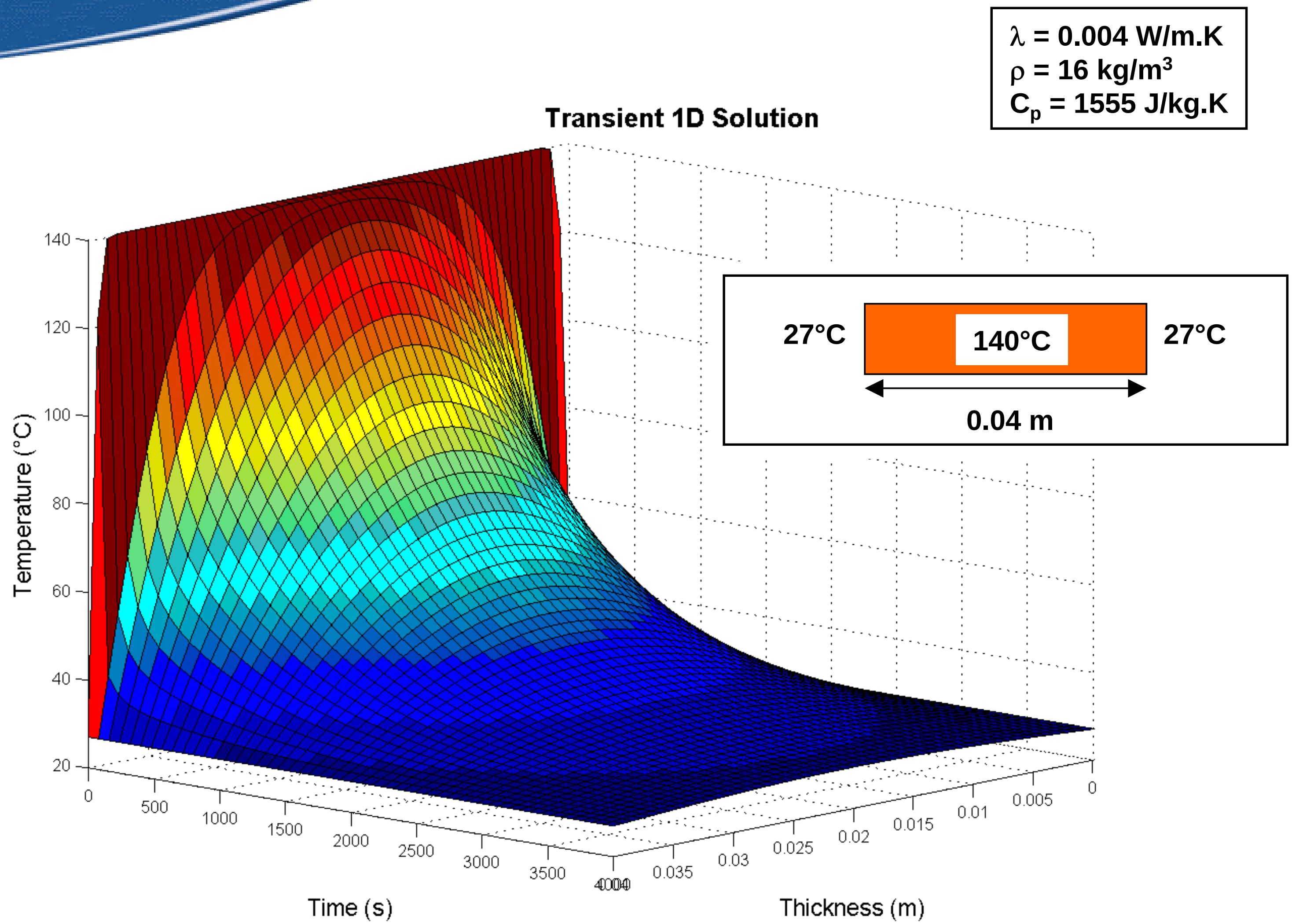
Foams

Problem: waviness in foams – thermal issue

Heating & cooling of foam block



Numerical modelling of cooling of foam block



Foams

Initial observations and results suggest:

- uneven cooling of foam sheet
- resulting in variation in shrinkage with position
- causing waviness of sheets

Industrial trial to be performed 20 – 21 October 2005

Numerical modelling trials ongoing

Standards for Thermal Properties Measurement of Plastics

Differential scanning calorimetry standards

ISO TC61 SC5 WG8 Thermal Properties

ISO 11357 Plastics - Differential scanning calorimetry (DSC)

- ISO 11357-1: 1997 Part 1: General principles (being revised)
- ISO 11357-2: 1999 Part 2: Determination of glass transition temperature
- ISO 11357-3: 1999 Part 3: Determination of temperature and enthalpy of melting and crystallization
- ISO 11357-4: 2005 Part 4: Determination of specific heat capacity
- ISO 11357-5: 1999 Part 5: Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion
- ISO 11357-6: 2002 Part 6: Determination of oxidation induction time
- ISO 11357-7: 2002 Part 7: Determination of crystallization kinetics

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Plastics thermal conductivity standards

ISO TC61 SC5 WG8 Thermal Properties

ISO 22007 Plastics –
Determination of thermal conductivity and thermal diffusivity

ISO/NWIP 22007-1 Part 1: General principles

ISO/CD 22007-2 Part 2: Transient plane source hot-disc method (Gustafsson)

ISO/CD 22007-3 Part 3: Temperature wave analysis method

ISO/CD 22007-4 Part 4: Laser flash method

Plastics thermal conductivity standards

Proposal to develop
Line Source Method for Thermal Conductivity
as part of ISO 22007 series

Method currently standardized as:

- ASTM D 5930-01, Test Method for Thermal Conductivity of Plastics by Means of a Transient Line-Source Technique

However this does not make provision for:

- effect of applying pressure to minimize measurement scatter, and
- effect of pressure on thermal conductivity

ISO TC61 SC5 WG8 Thermal Properties.

Plastics thermal conductivity standards

Proposed intercomparison of thermal conductivity methods

To be carried out, in support of standardisation activity, including at least:

- Transient plane source hot-disc method (Gustafsson)
- Temperature wave analysis method
- Laser flash method
- Line source probe

To be led by NPL

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Summary – Heat Transfer

- Heat transfer coefficient apparatus now being used
  - Design assisted by numerical modelling studies
  - Effect of uncertainties investigated (report available)

- Melt thermal conductivity measurements
  - Nano-filled materials
  - Powders/granules
  - Effect of pressure
  - Effect of uncertainties investigated (report available)

- ISO Standards being developed

- New IAG members facility on website

http://www.npl.co.uk/materials/polyproc

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The next 6 months

- Complete commissioning trials on heat transfer coefficient equipment
- Complete industrial demonstrations (Corus & Zotefoams)
- Dissemination of thermal conductivity and heat transfer coefficient measurement work

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Heat Transfer Project
2005-08

H1: Measurement methods for heat transfer properties data for application to polymers

Objectives:

- Development of the method for the measurement of heat transfer properties across surfaces (particular interest has been expressed in the effect of the solid/air interface)
- Industrial case study to demonstrate the value of reliable heat transfer data
- Support development of standards for measurement of thermal properties of plastics, including an intercomparison of thermal conductivity methods that are being proposed for standardisation
- Assessment of uncertainties in heat transfer data and effect on modelling predictions
- Development of a new user-friendly web-enabled modelling facility, to facilitate industrial adoption of the above

Heat Transfer Coefficient - future activity

- Investigate effect of:
  - Air gap between polymer & top plate (simulating shrinkage and sink marks) (Cinpres) and compare with numerical modelling predictions
  - Different surface finishes/mould materials (RAPRA)
  - Effect of mould release agents

Potential areas of relevance for future work to increase understanding of heat transfer:

- Effect of air gaps (EGM)
- Effect of mould materials (RAPRA)
- Gas assisted injection moulding (GAIM)
- Water assisted injection moulding (WAIM)
- Additives, fillers – e.g. effect of nano-particles on thermal conductivity
- Micro-moulding

Potential areas of relevance for future work to increase understanding of heat transfer:

- Effect of nano-particles on heat transfer in polymers
- Effect of dispersion of nano-particles on heat transfer
- Measurement of heat transfer within micro-fluidic systems
- Developing techniques for measuring heat transfer properties of foams
- Curing of fibre/matrix composites and cross-linking of rubbers

Your:

Ideas,
comments,
suggestions,
participation,
contributions, …

to steer the project to maximise the benefits to you.

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Any other business

Tea & coffee available