

# Heat Transfer in Polymers

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27 April 2005

# Heat Transfer in Polymers

## - summary

- Introduction
- Heat Transfer Coefficient
- Thermal Conductivity
- Thermal Imaging
- Industrial Demonstrations
- Standards for Thermal Properties
- Summary of current heat transfer project
- Outline of heat transfer project 2005-08
- Future Needs

# Aim of the project

- To help companies measure and model heat transfer in polymer processing
- This should lead to:
  - Right first time design
  - Higher productivity (faster processing)
  - Energy saving
  - Fewer failures in service

Resulting in reduced costs and improved quality

# 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

# 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

# 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  $T_2$  :

$$h = q/(T_1 - T_2)$$

units:  $\text{Wm}^{-2}\text{K}^{-1}$

- 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

# Heat Transfer Coefficient

(heat transfer across an interface)

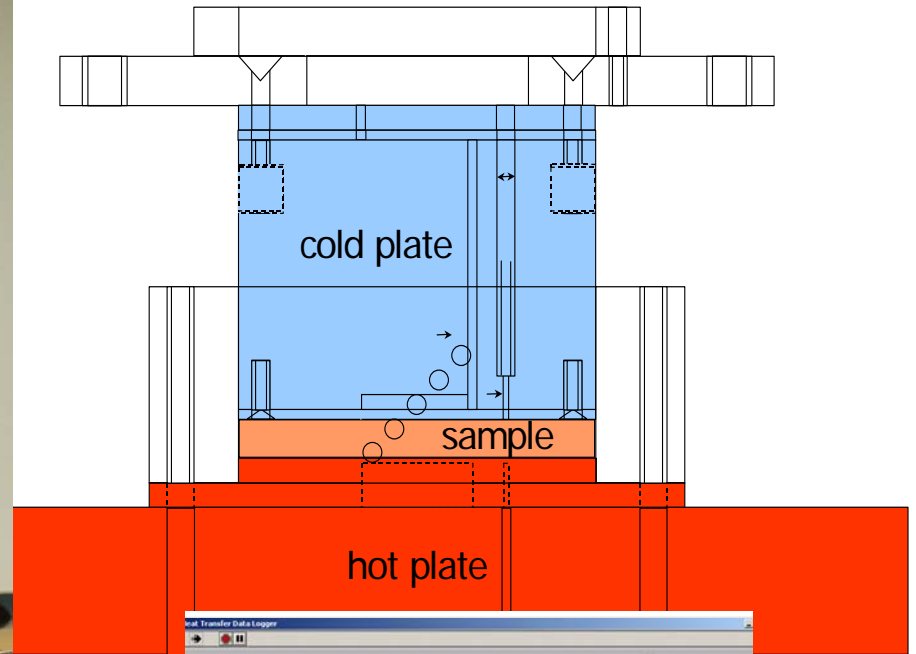
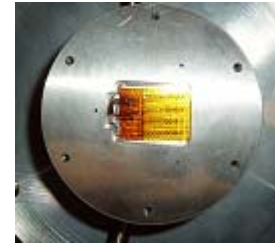
## Features of apparatus

- 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

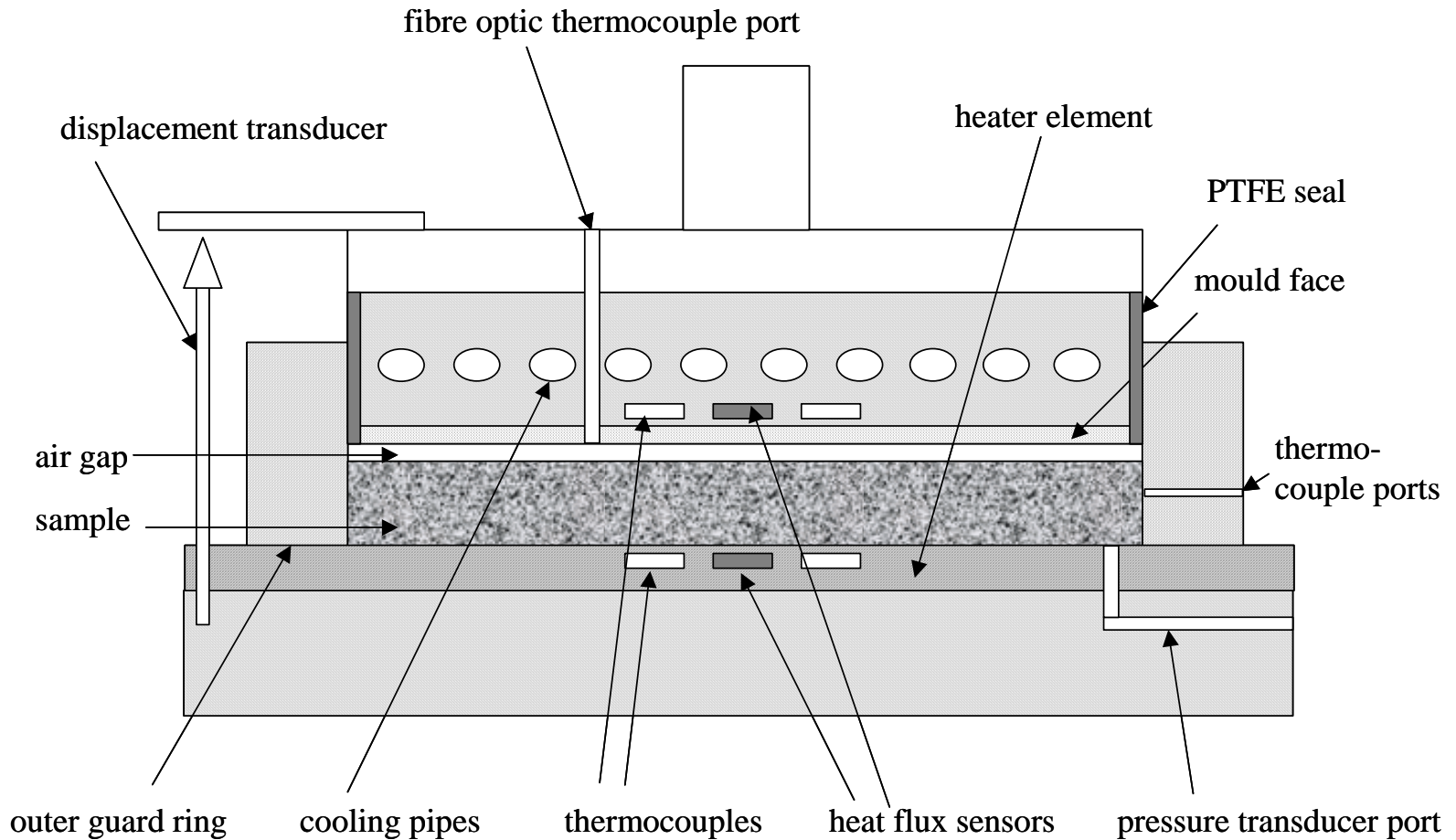


# Heat transfer apparatus

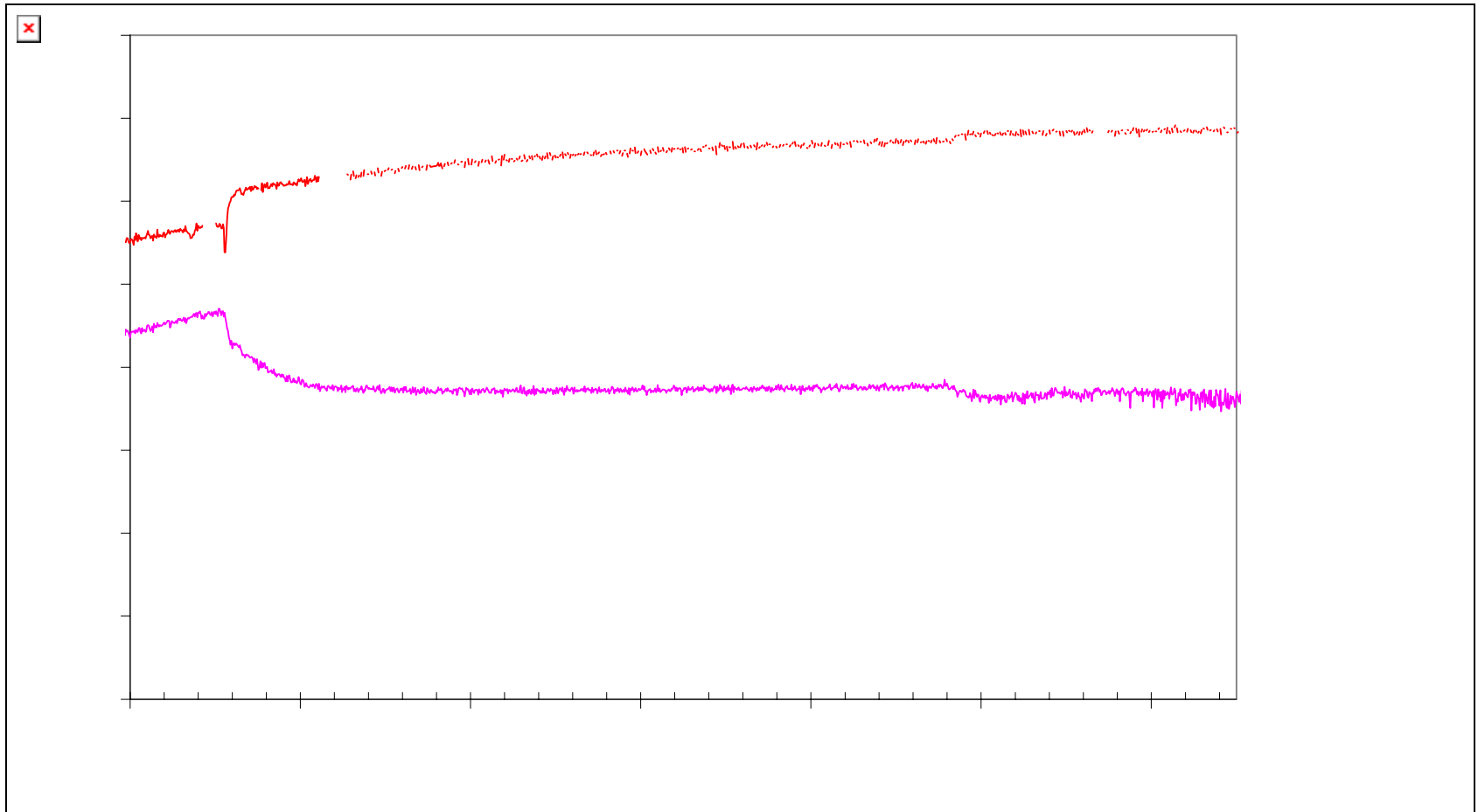
Side view



# Heat transfer apparatus



# Heat transfer coefficient

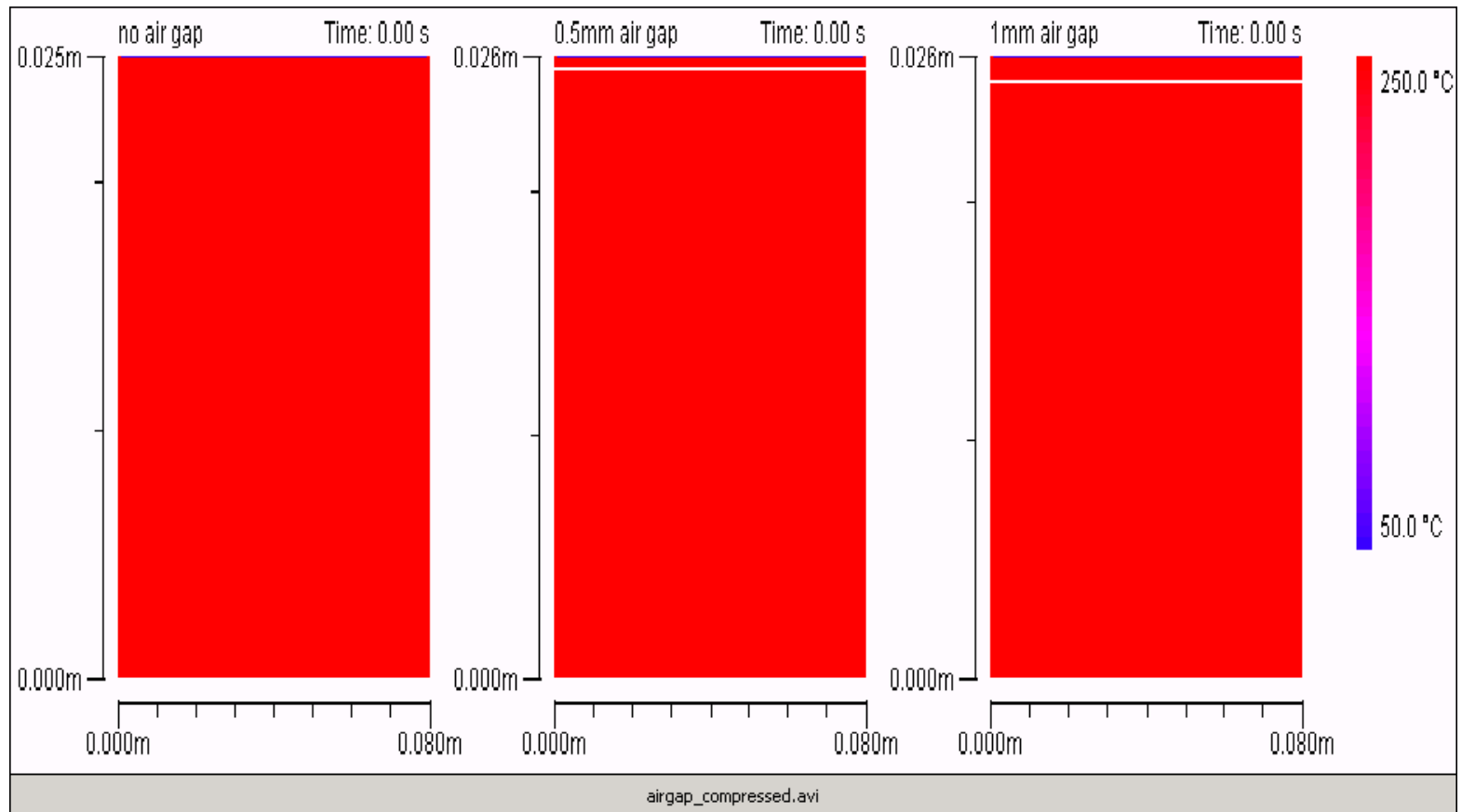


# Modelling of key features

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

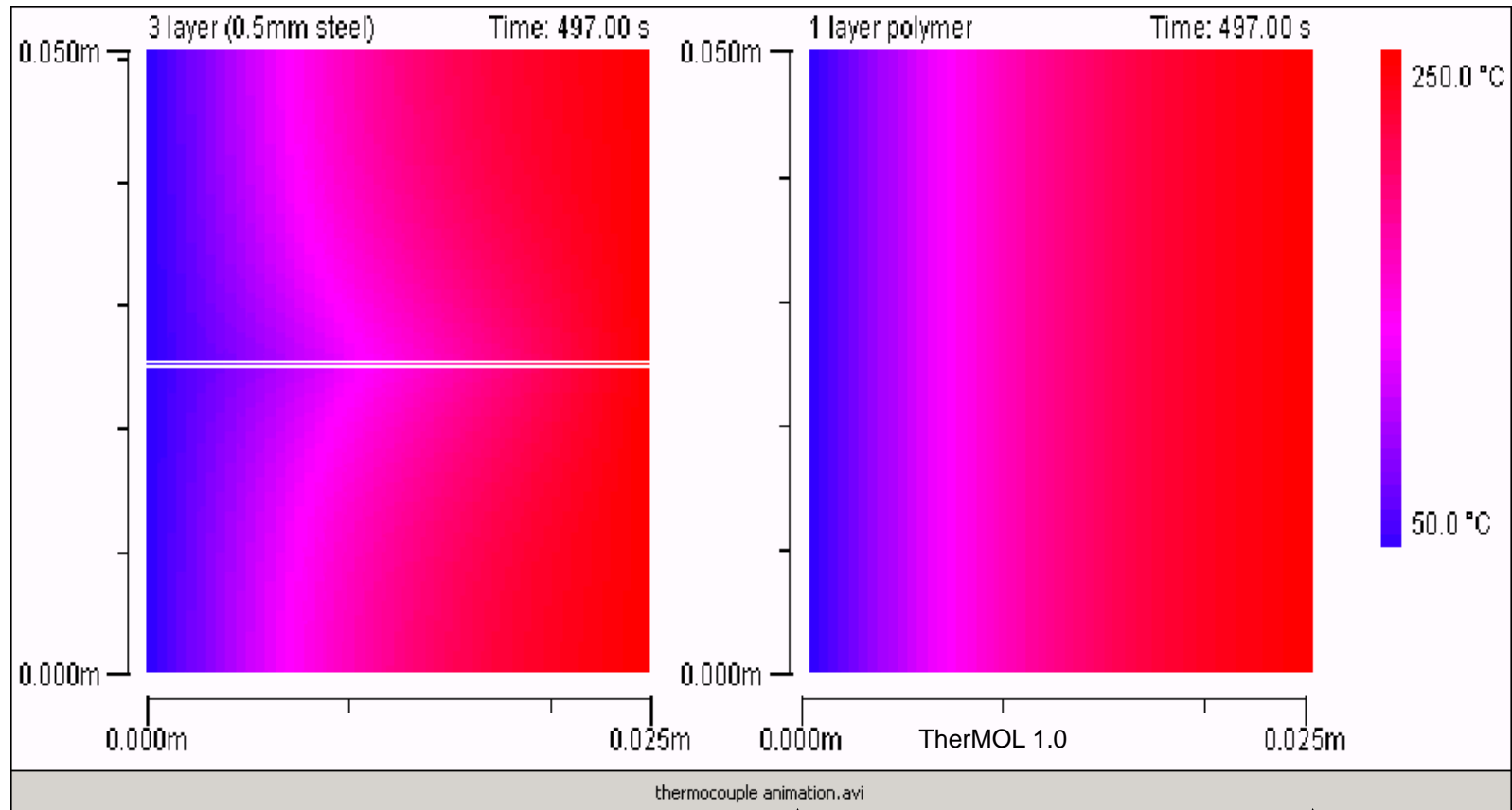
# Air Gap

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



Polymer at 250 °C

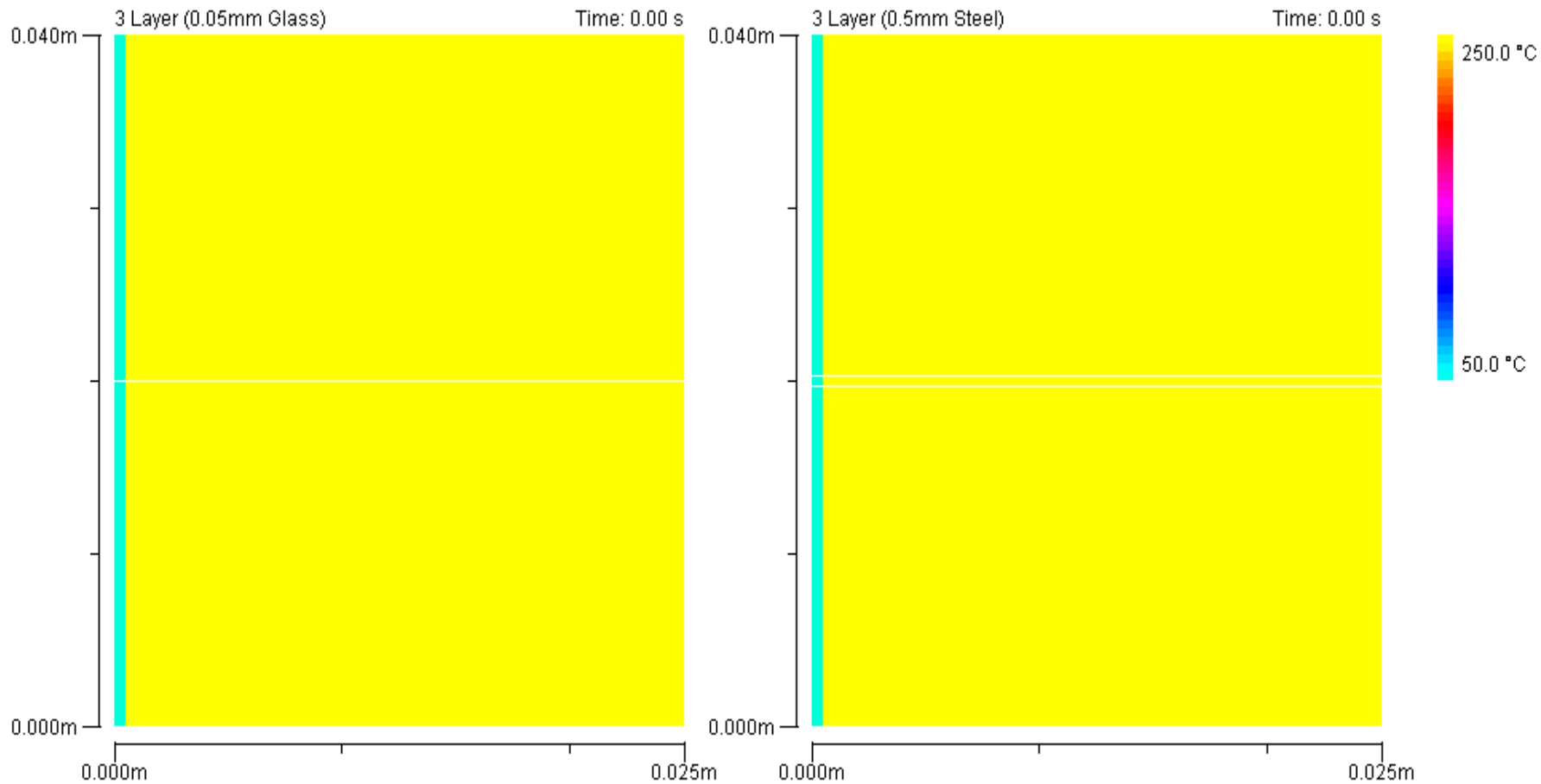
# Effect of a thermocouple



Mould at  
50 °C

Polymer  
at 250 °C

# Simulation of Heat Transfer with Fibre Optic (left) & Thermocouple (right)



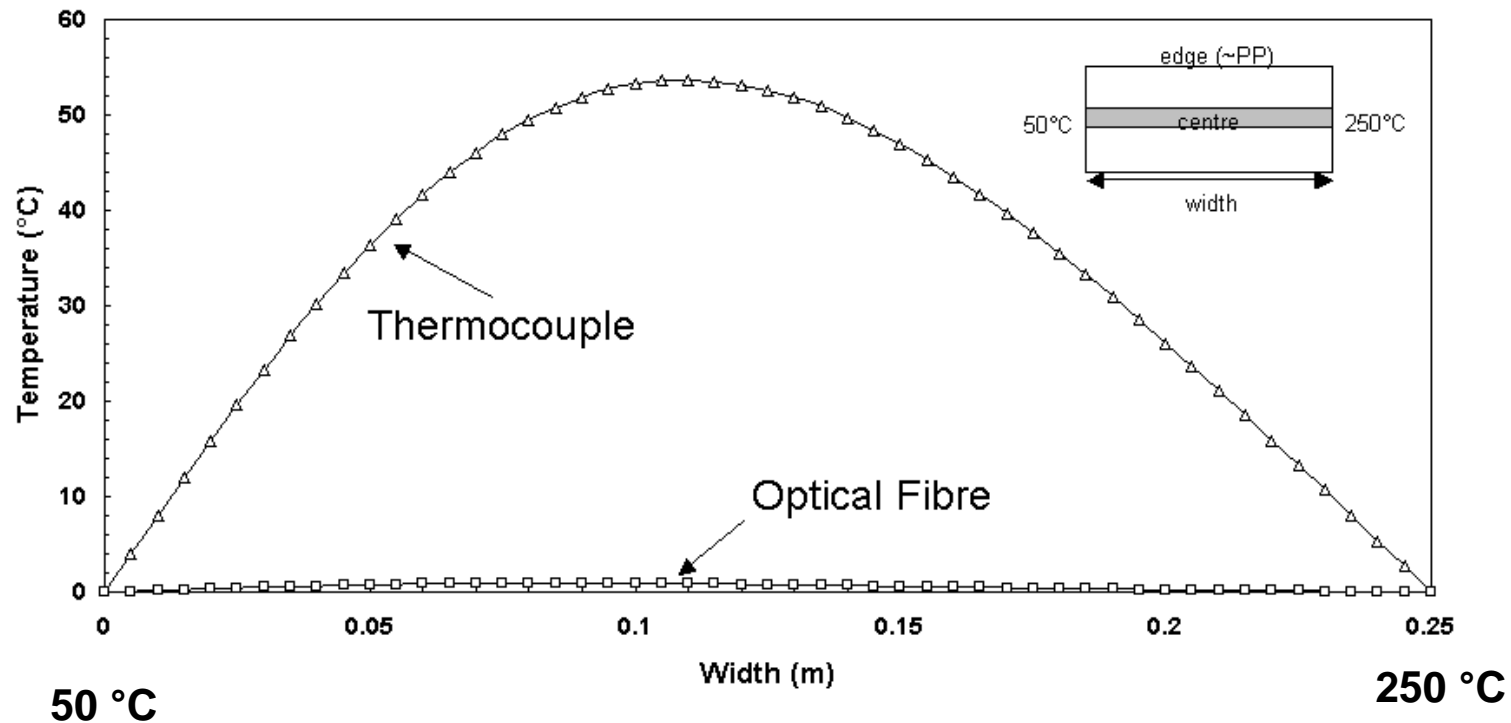
Data from TherMOL 1.0 on 24/02/04 at 15.32

Data from TherMOL 1.0 on 24/02/04 at 21.27



# Comparison of thermocouple & fibre optic

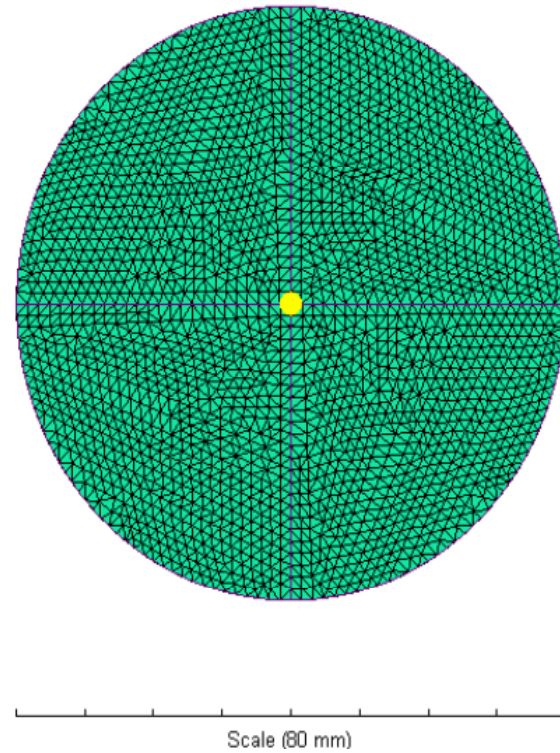
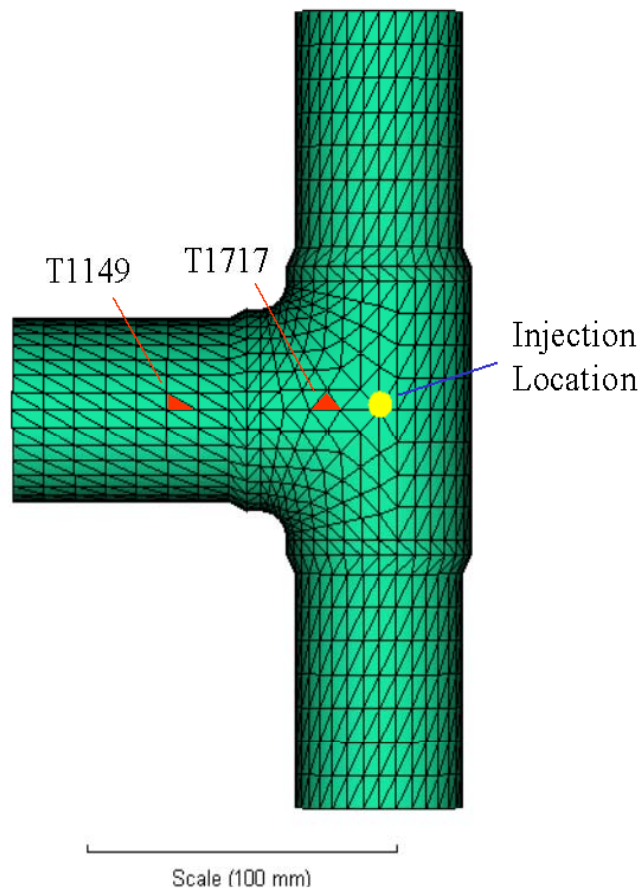
TherMOL prediction of the temperature difference between the sensor (centre) and the edge (PP) for PP & a thermocouple and PP & a optical fibre after 400s





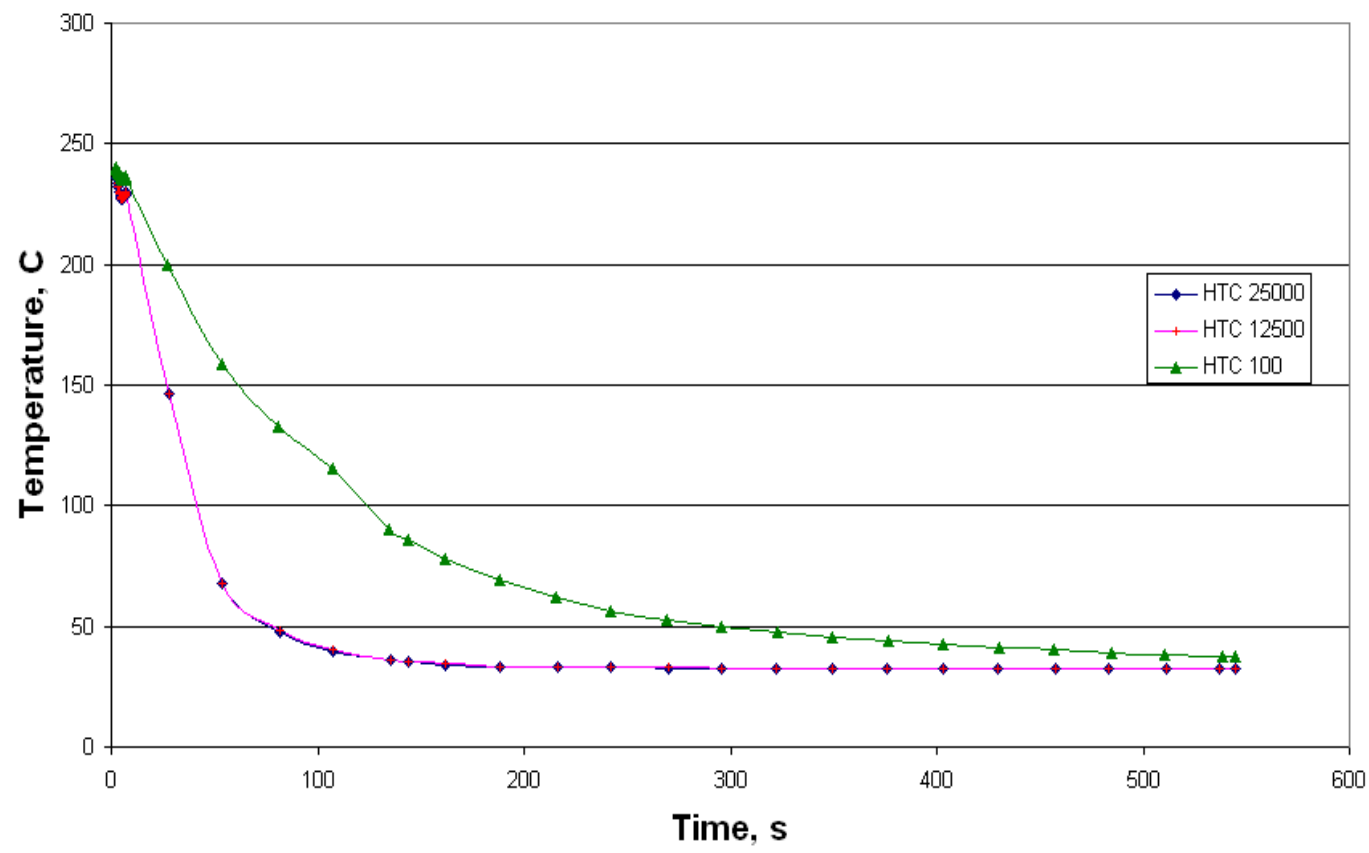
# Heat transfer coefficient effect of uncertainties

# Pipe 'T' piece and 80 mm diameter disc models



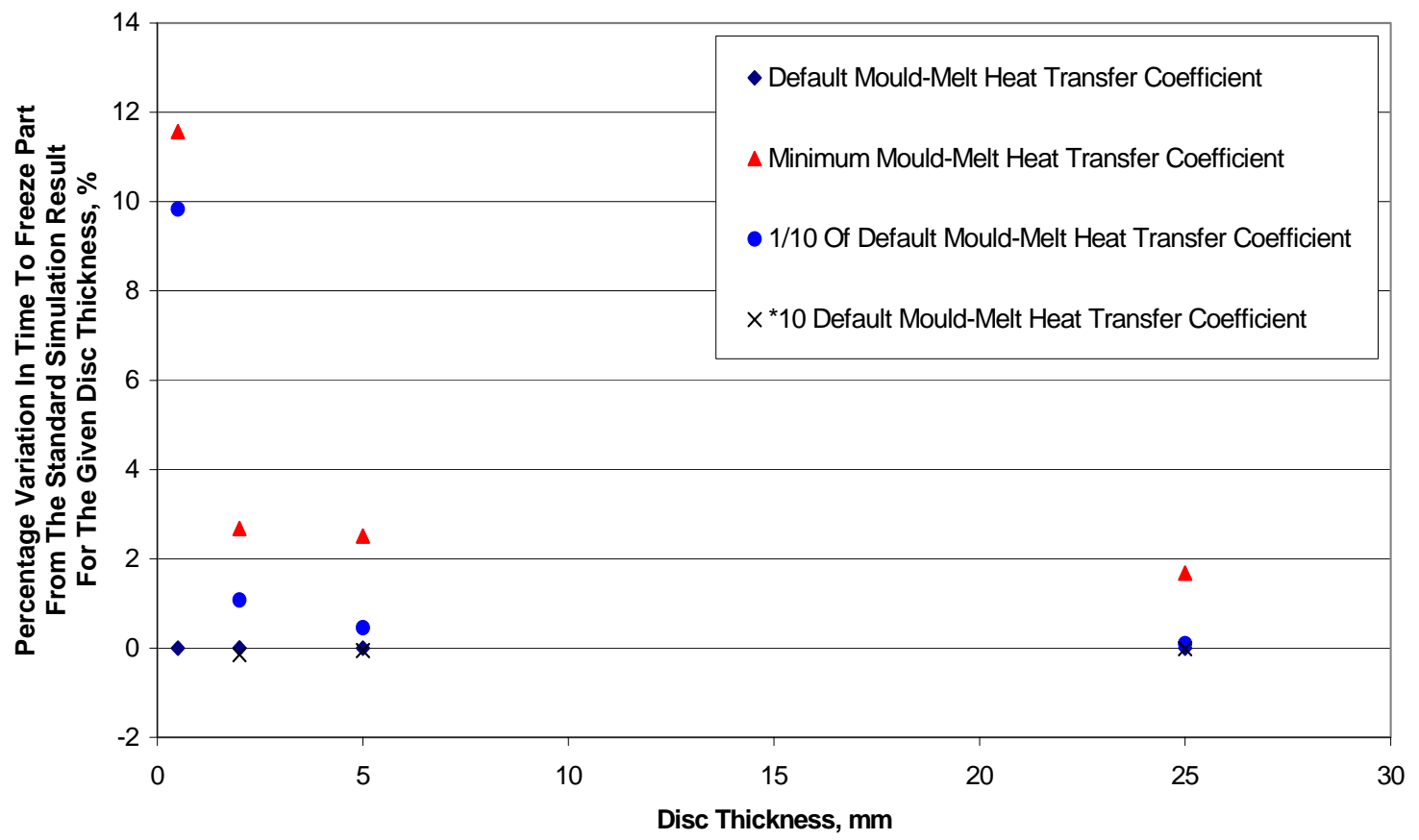
# Effect of uncertainties in HTC

The Change in Temperature Over Time For A Given Location (T1149) on the T-Pipe For Analyses With Different Heat Transfer Coefficient



# Effect of uncertainties in HTC

The Effect Of Mould-Melt Heat Transfer Coefficient Upon Time To Freeze Part For Discs Of Different Thickness



**NPL Report DEPC-MPR 001**

# **The Effect of Uncertainty in Heat Transfer Data on The Simulation of Polymer Processing**

**J. M. Urquhart and C. S. Brown**

[http://libsvr.npl.co.uk/npl\\_web/search.htm](http://libsvr.npl.co.uk/npl_web/search.htm)

# Heat Transfer Coefficient Summary

- Initial testing commenced using HTC equipment
- To investigate effect of:
  - Different surface finishes/mould materials
  - Mould release agents
  - Air gap between polymer & top plate  
(simulating shrinkage and sink marks)

# Thermal conductivity measurements

## Thermal Conductivity Measurements Under Industrial Processing Conditions:

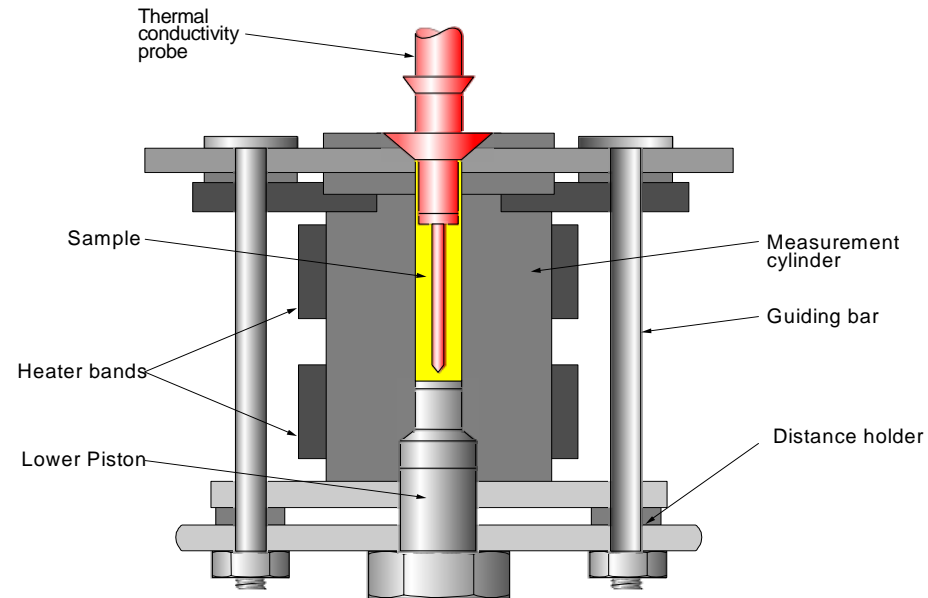
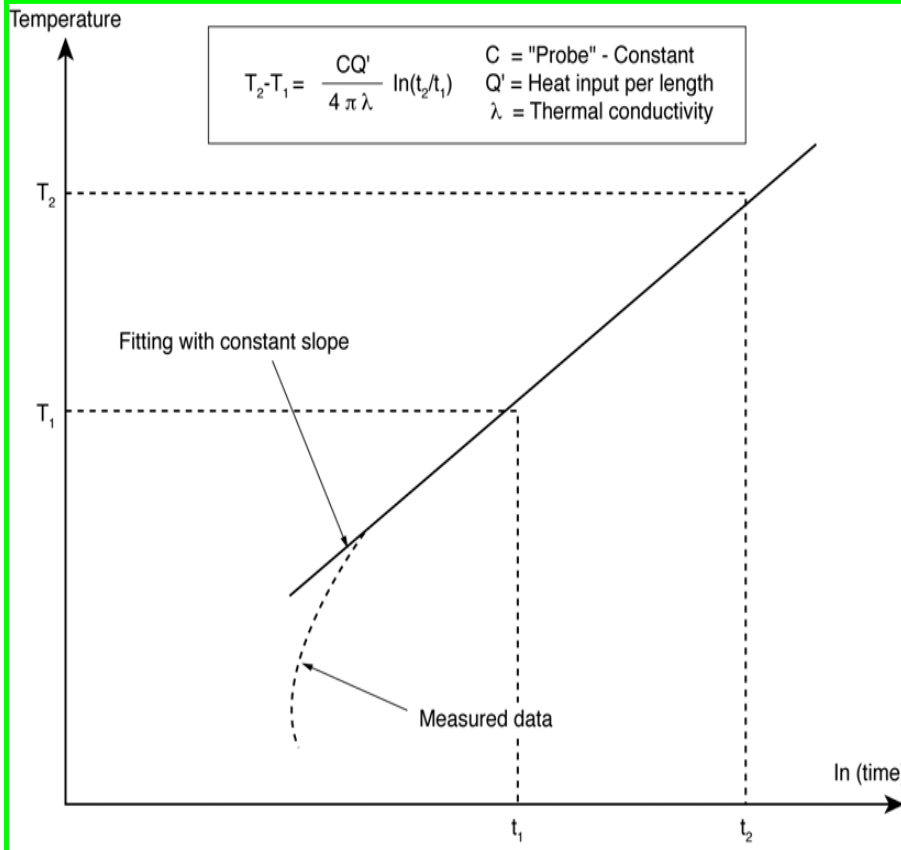
- More accurate data for modelling software
- Reduce warpage and hot spots during injection moulding process – reduce waste
- Reduce cycle times and improve processing efficiency



## Plan of Action:

- Measured thermal conductivity of amorphous and semi-crystalline polymers at injection moulding pressures
- Used experimental techniques to attribute uncertainty to thermal conductivity measurements
- Compared thermal conductivity measurements with known pvT technique

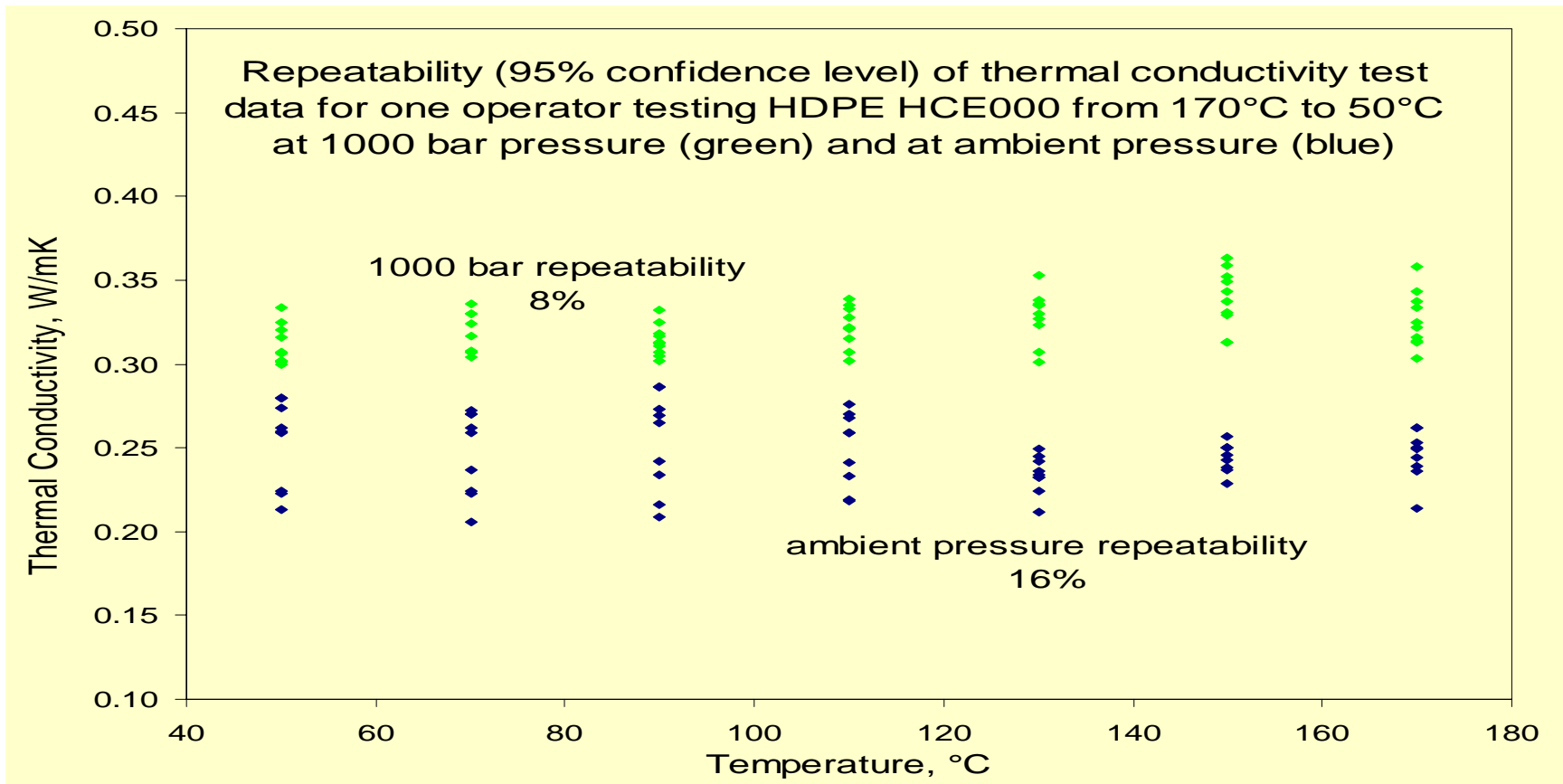
## Line source probe apparatus



Measures thermal conductivity  
at industrial processing  
pressures

# Thermal conductivity repeatability measurements and uncertainty

# Thermal Conductivity of HDPE (Atmospheric and 1000 bar Pressures)



## Uncertainty Budget For NPL Line-Source Thermal Conductivity Probe (Atmospheric Pressure)

	<b>Value ± %</b>	Probability Distribution	<b>Divisor</b>	$C_i$	<b>Uncertainty Contribution ± %</b>	Uncertainty Squared ± %	<b><math>V_i</math> or <math>V_{eff}</math></b>
<b>Type A</b>							
Repeatability	15.6 @ 2 std devs	Normal	2	1	7.815 @ 1 std dev	61.07	89
Reproducibility	13.6 @ 2 std devs	Normal	2	1	6.801 @ 1 std dev	46.25	89
<b>Type B</b>							
Non-uniformity of heat input	0.002	Rectangular	1.73	1	0.00116	1.34E-06	∞
Non-uniformity of temperature	0.0	Rectangular	1.73	1	0.000	0.000	∞
Sample height	0.0	Rectangular	1.73	1	0.000	0.000	∞
Time	0.0	Normal	1	1	0.000	0.000	∞
					<b>Calculation of Uncertainty</b>		
					<b>Sum of squares</b>	107.3 %	
					<b>Square root of sum of squares</b>	10.4 %	
					<b>Multiplication by <math>k=2</math> for 95% confidence level</b>	±20.7%	
						<b>Final Uncertainty Value</b>	

# Thermal conductivity measurements under pressure

## Materials tested:

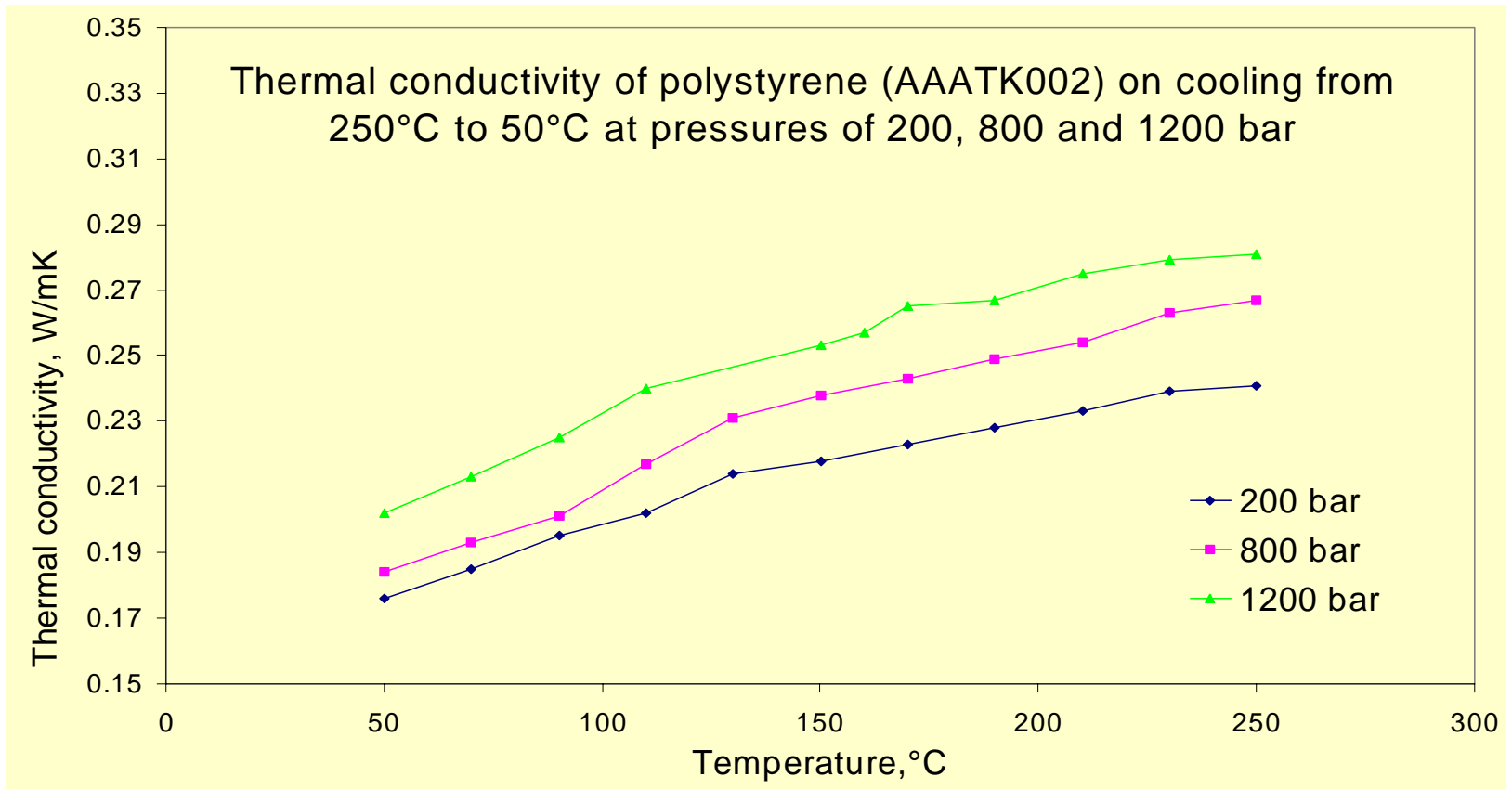
### Amorphous:

- Acrylonitrile-butadiene-styrene
- Polystyrene
- Polycarbonate

### Semi-crystalline:

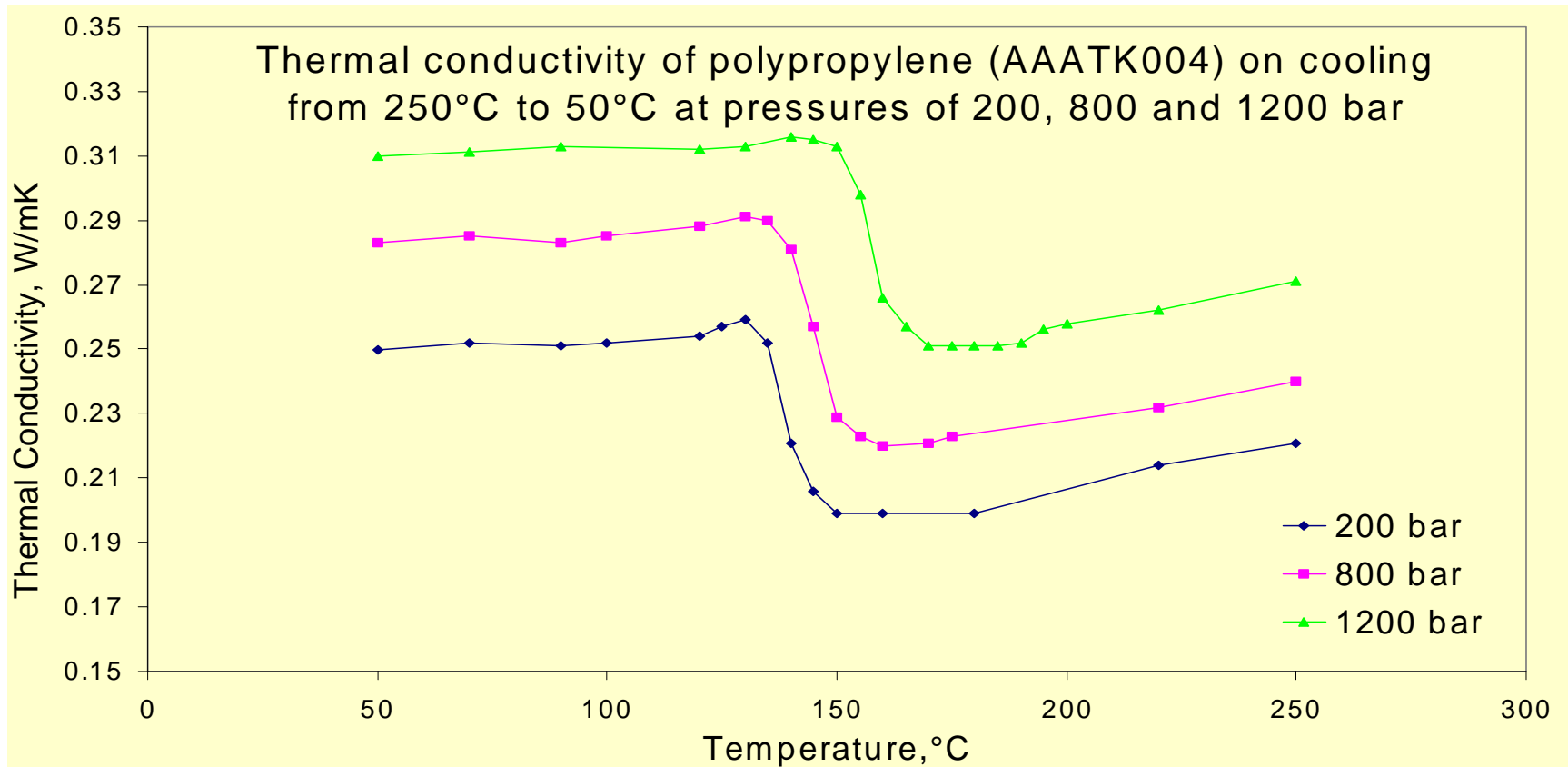
- Polypropylene
- Polystyrene
- Polyethylene(terephthalate)
- Glass filled nylon

## Thermal Conductivity Behaviour of Typical Amorphous Material (PS) Under Pressure



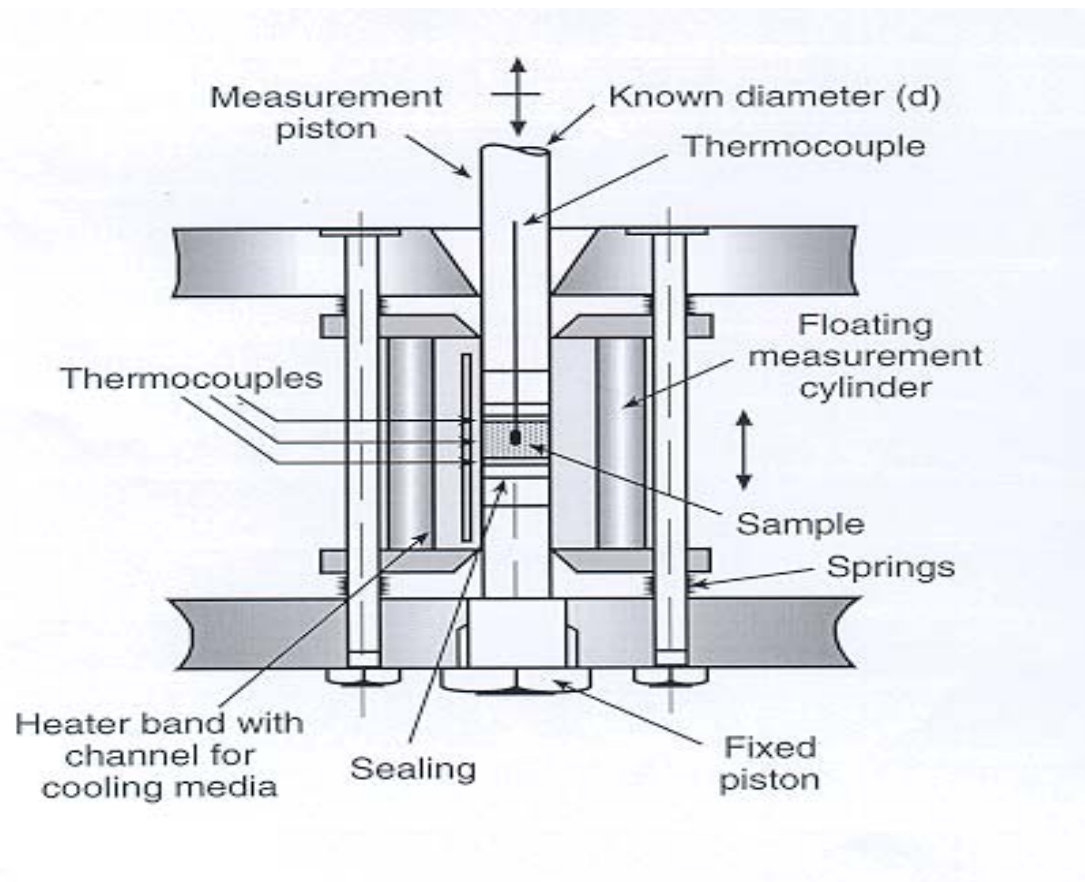


## Thermal Conductivity Behaviour of Typical Semi-crystalline Material (PP) Under Pressure

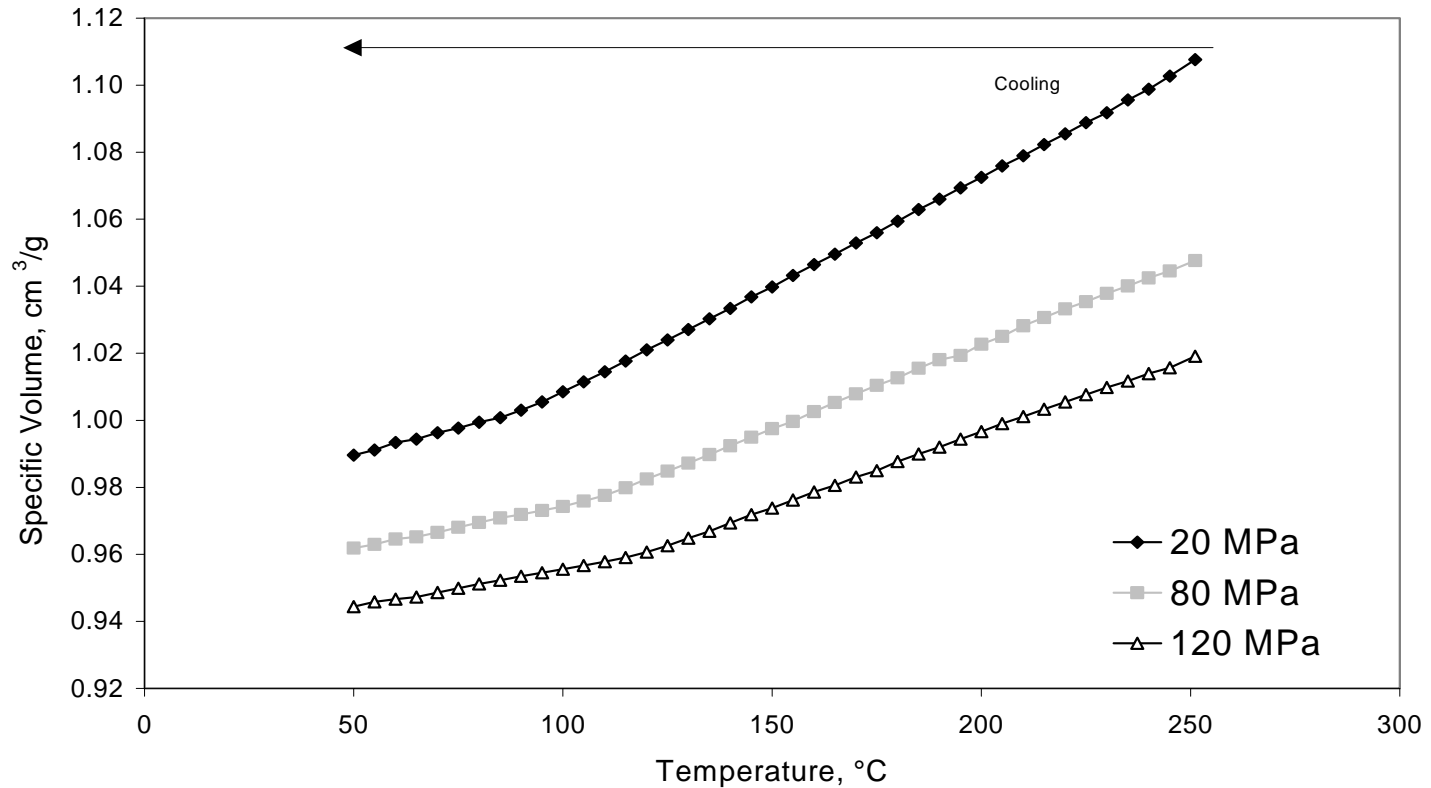


# pVT measurements under pressure

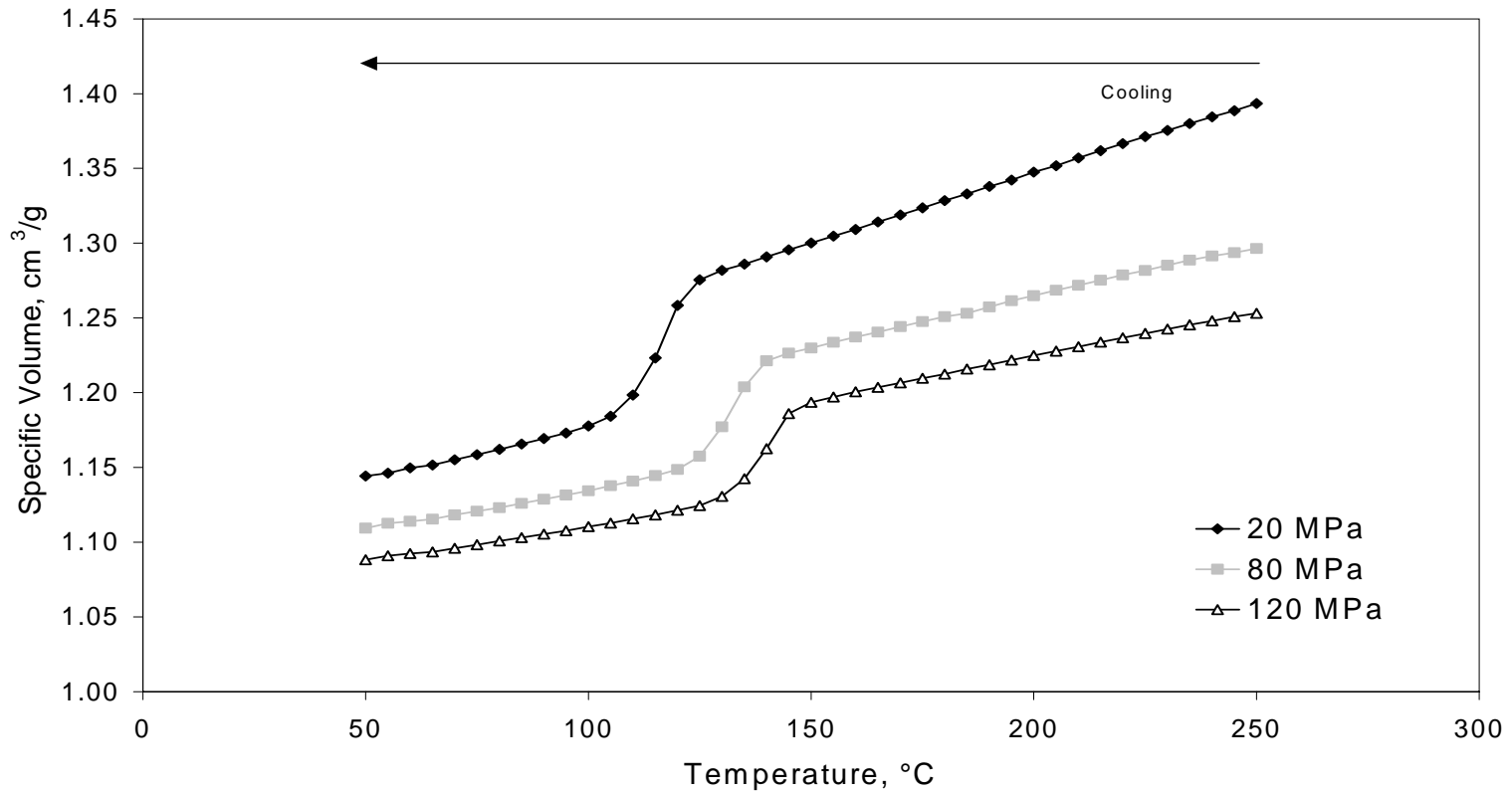
## Schematic of pvT Instrument



## pvT Behaviour of a Typical Amorphous Polymer (PS)



## pvT Behaviour of a Typical Semi-crystalline Polymer (PP)



# Models for specific volume and thermal conductivity

Pressure term with temperature dependence

Temperature term

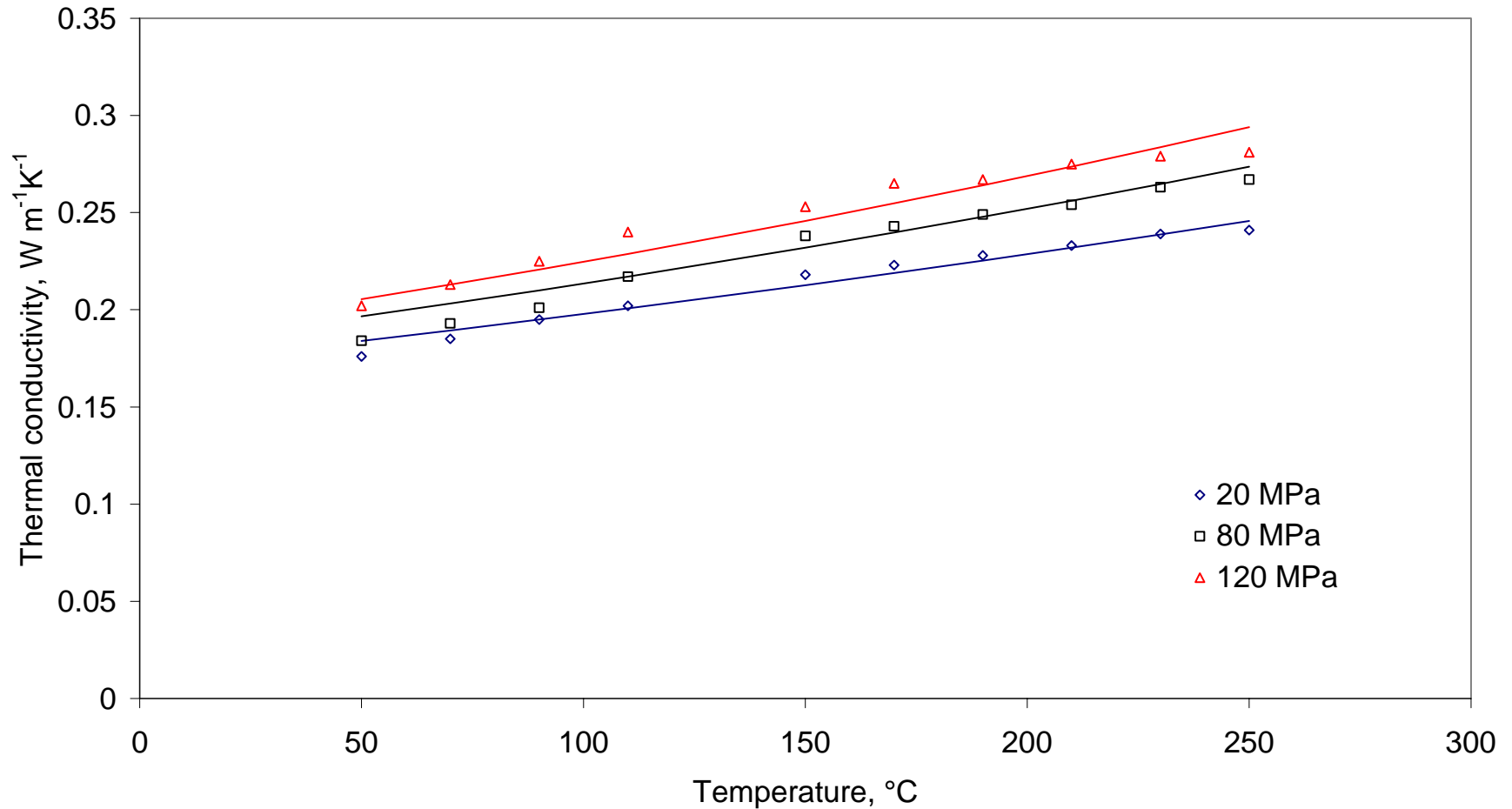
$$v = v_0 [\exp(k(\theta - \theta_o))] [\exp(\ell(p - p_o)(\theta + 273.15))]$$

$$\lambda = \lambda_0 [\exp(k'(\theta - \theta_o))] [\exp(\ell'(p - p_o)(\theta + 273.15))]$$

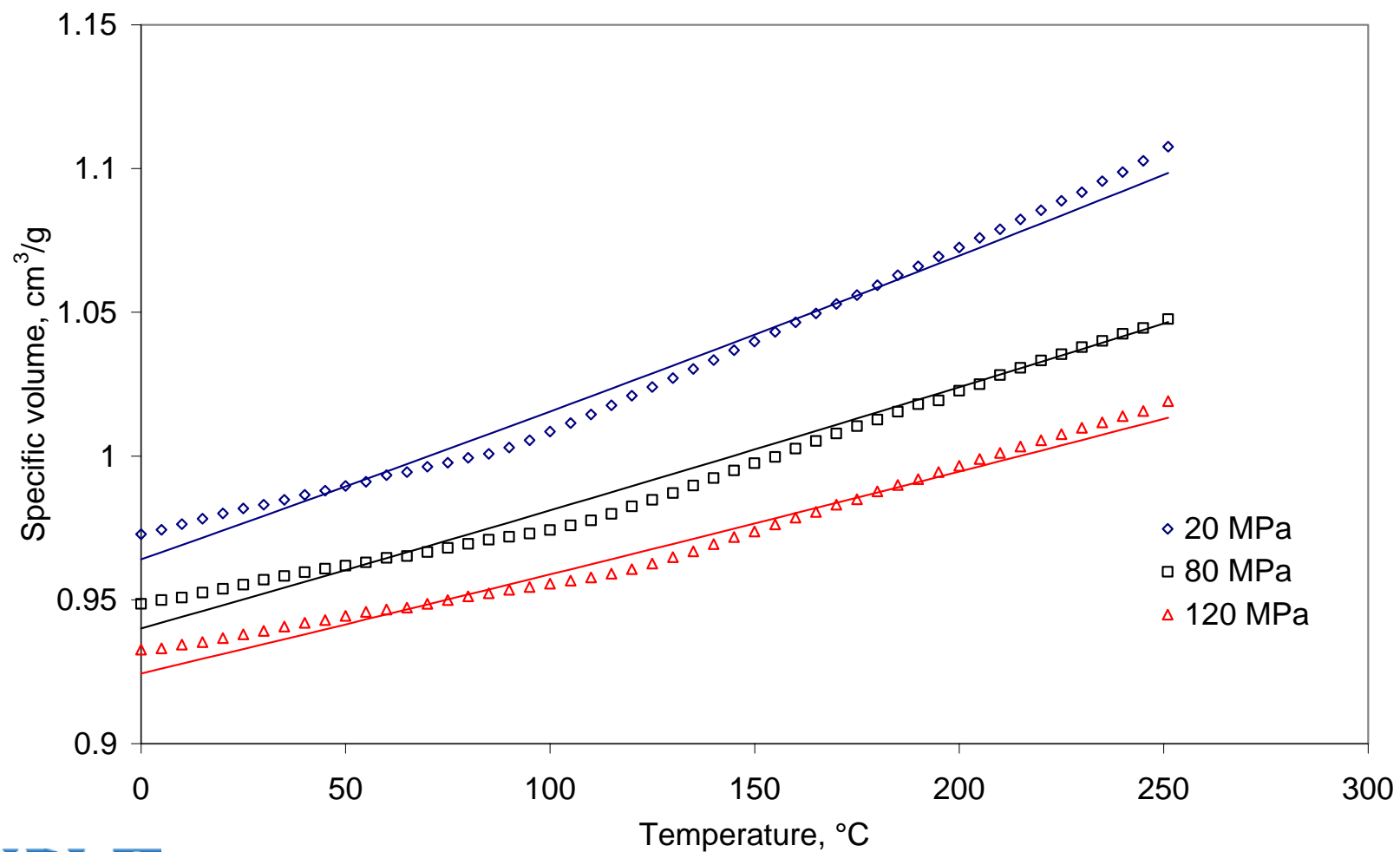
PS					
Thermal conductivity, $\lambda$ $\text{Wm}^{-1}\text{K}^{-1}$	$\lambda_0$ $\text{Wm}^{-1}\text{K}^{-1}$	$k'$	$\ell'$	$\theta_o$ $^{\circ}\text{C}$	$p_o$ MPa
	0.274	0.00165	3.43E-06	250	80
Specific volume, $v$ $\text{cm}^3\text{g}^{-1}$	$v_0$ $\text{cm}^3\text{g}^{-1}$	$k$	$\ell$	$\theta_o$ $^{\circ}\text{C}$	$p_o$ MPa
	1.047	0.000427	-1.54E-06	251.1	80

$$\lambda = v \frac{\lambda_0}{v_0} [\exp((k - k')(\theta - \theta_o))]^{-1} [\exp((\ell - \ell')(p - p_o)(\theta + 273.15))]^{-1}$$

# Thermal conductivity data for polystyrene

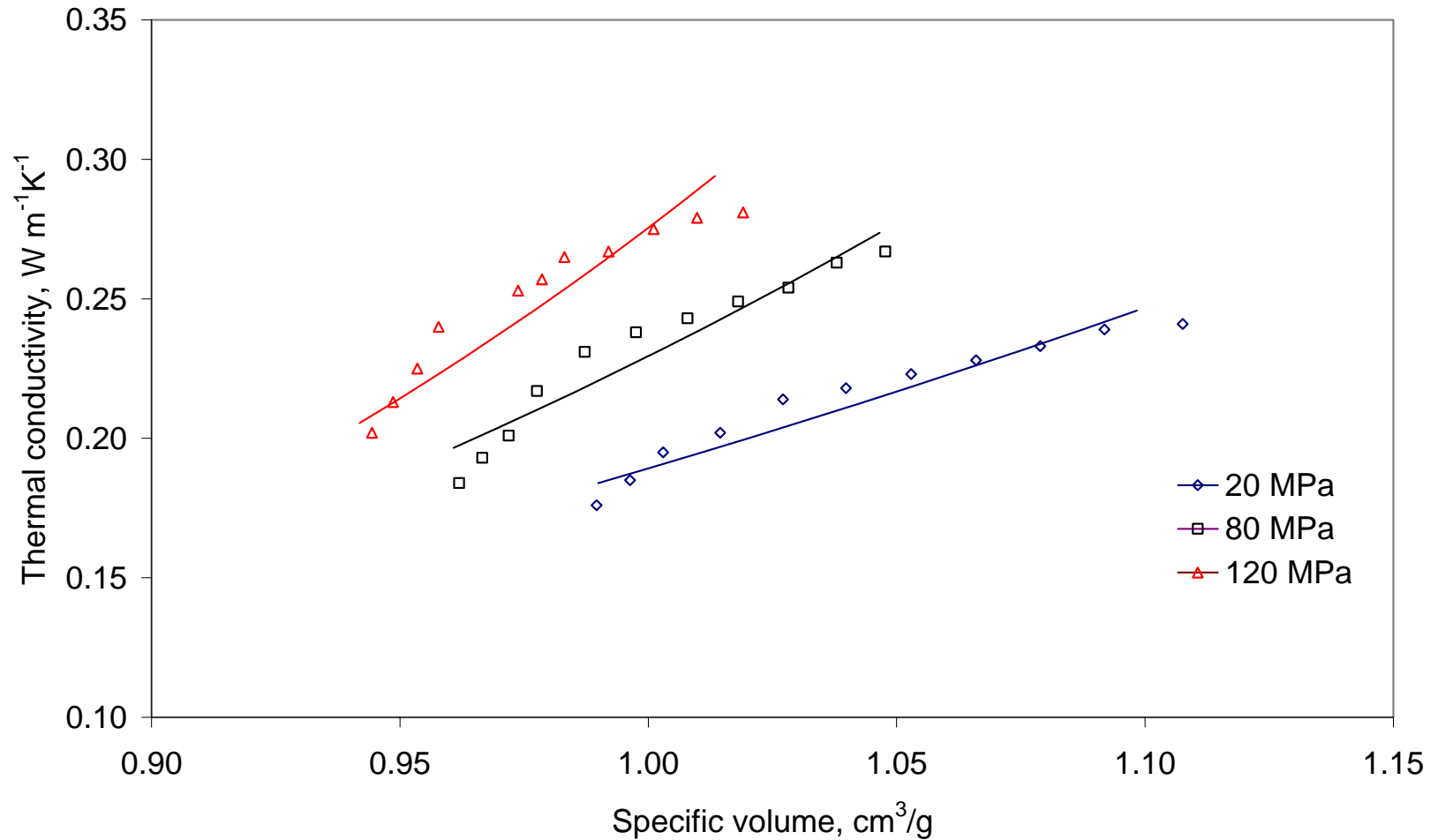


# Specific volume data for polystyrene





# Correlation of thermal conductivity with specific volume data for PS



## Implications of Results

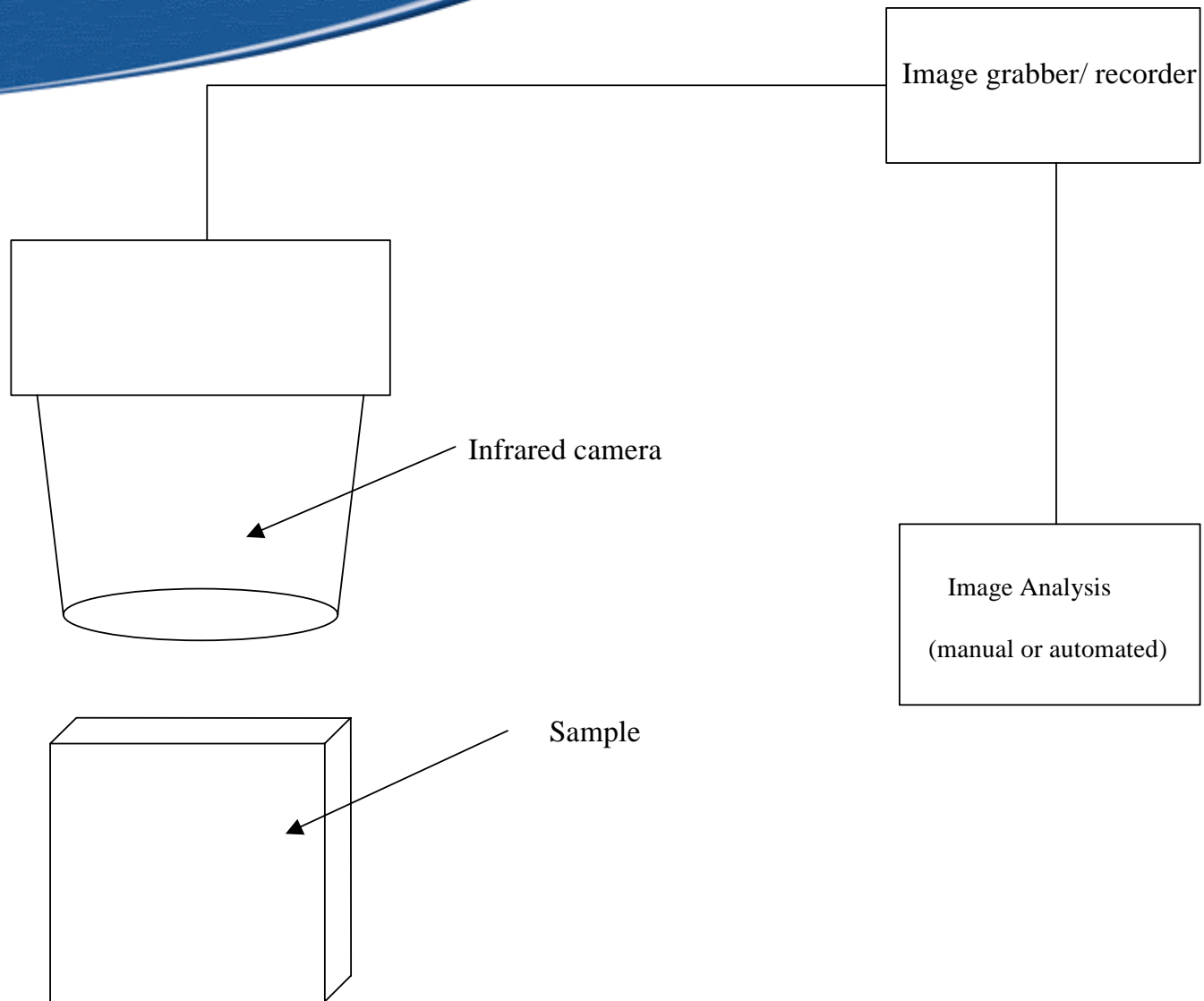
- Increase in pressure gives increase in thermal conductivity - reduction in cycle times – possible cost benefits
- Increase in crystallisation temperature for semi-crystalline polymers with increase in pressure – may reduce time to freeze parts - possible cost benefits
- More accurate data based on industrial processing conditions - improvements in commercial modelling packages - cut scrap rates by improving warpage and hot-spot prediction – possible cost benefits
- Crystallisation temperature for PP occurred over a similar temperature range for thermal conductivity and specific volume results confirming validity of TC tests
- Correlation of specific volume and thermal conductivity values

# Thermal Imaging

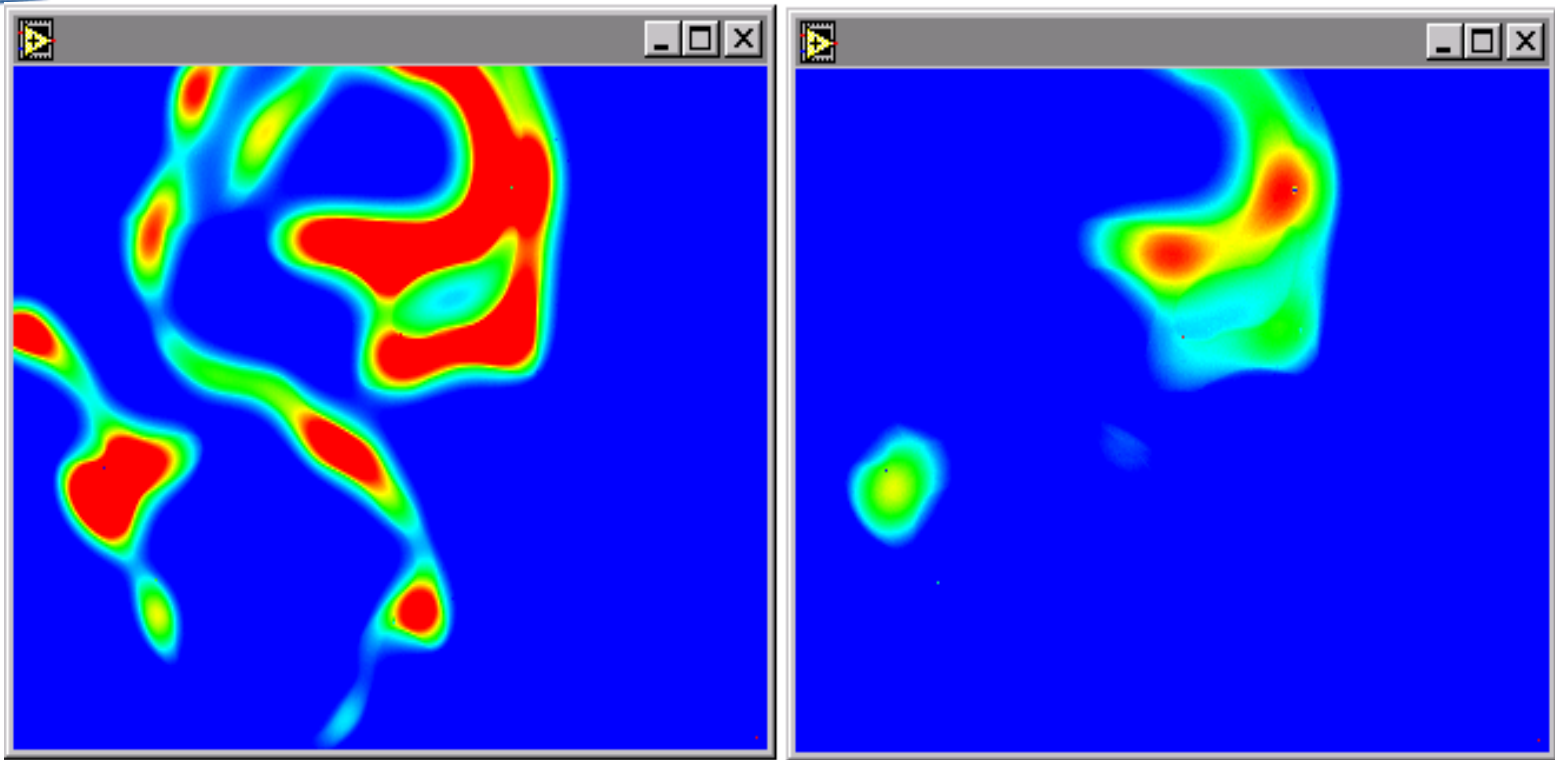
# DEPC IR Camera



# Schematic Diagram of IR Camera Operation



# Cooling of Hot Melt Adhesive Study Using IR Camera

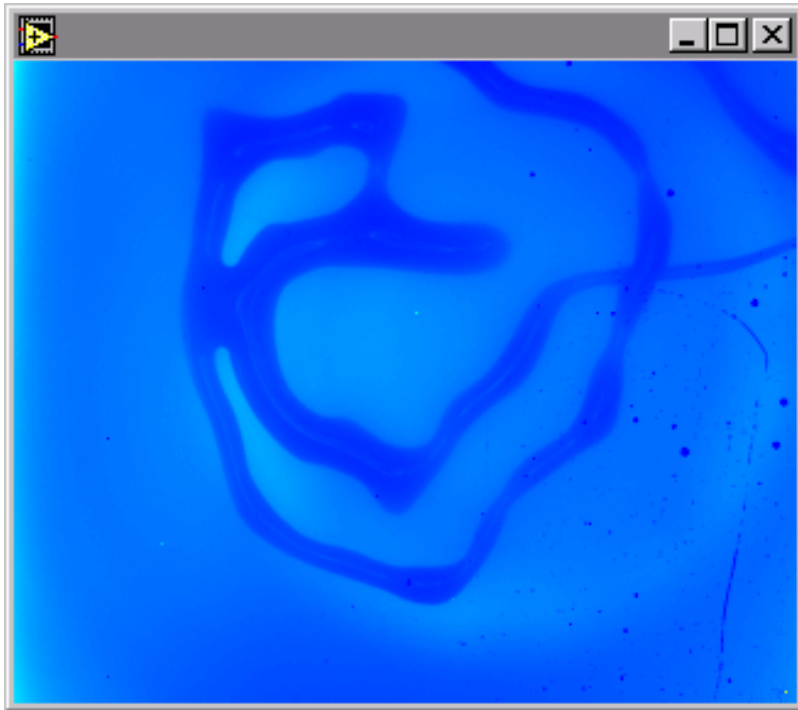


Time after extrusion

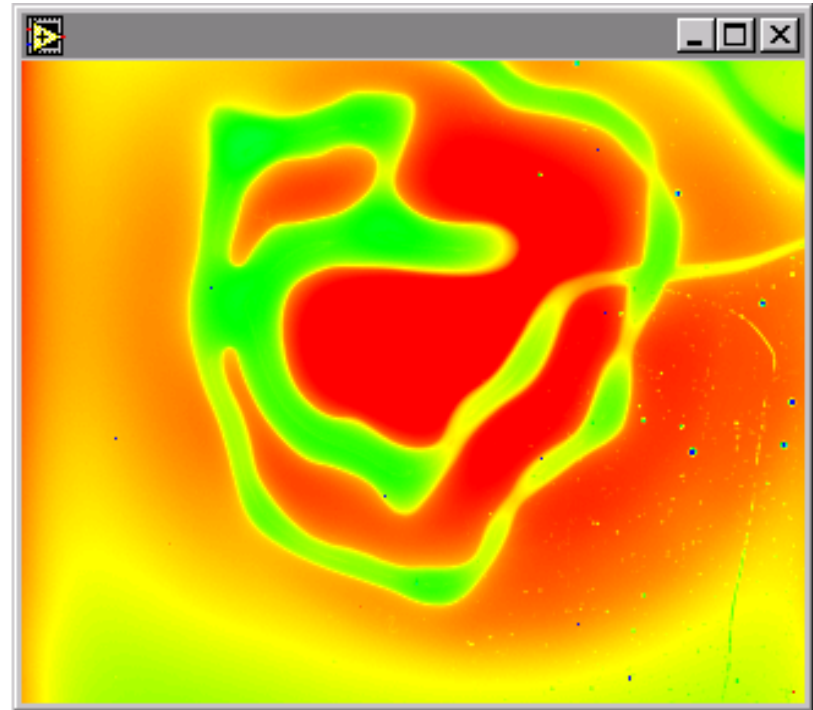
37 seconds

152 seconds

# Heating of Hot Melt Adhesive Study Using IR Camera



0 seconds



130 seconds

Time after start of heating



# Infra Red Camera

- Non contact method
- Produces visual record of thermal changes during heating and cooling of sample
- Visual record can be analysed in quantitative way to produce a time vs. temperature plot of thermal changes
- Can be customised to an individual system
- Easy to operate once it has been set up correctly
- Samples to be tested have to be of similar weight and geometry for comparisons to be made



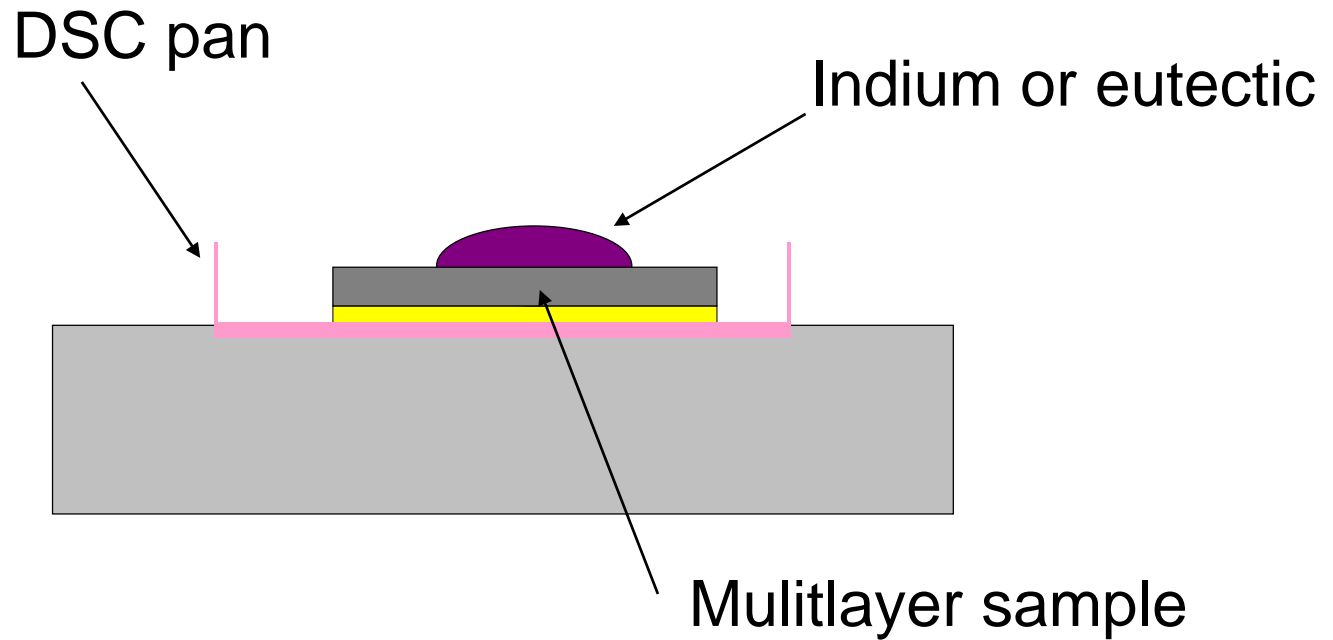
# INDUSTRIAL TRIALS

## Corus & Zotefoams

- Aim is to 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

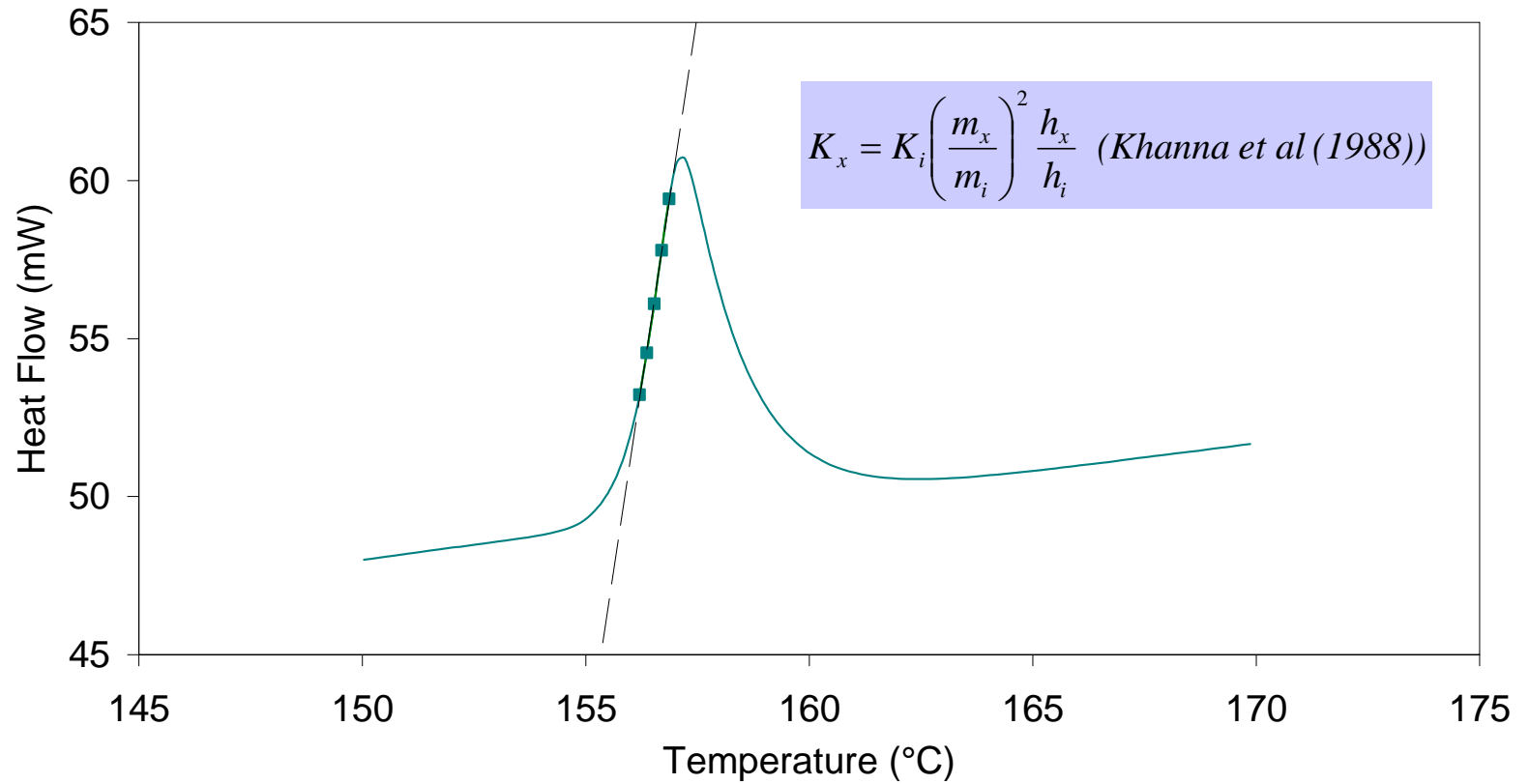
- Use DSC method to measure thermal conductivity of bilayer Plastisol/steel
- Measure before and after solidification
- Data useful in predicting optimum line speeds
  - Earlier work had shown that the polymer layer was significant in terms of heat transfer

# DSC method for thermal conductivity



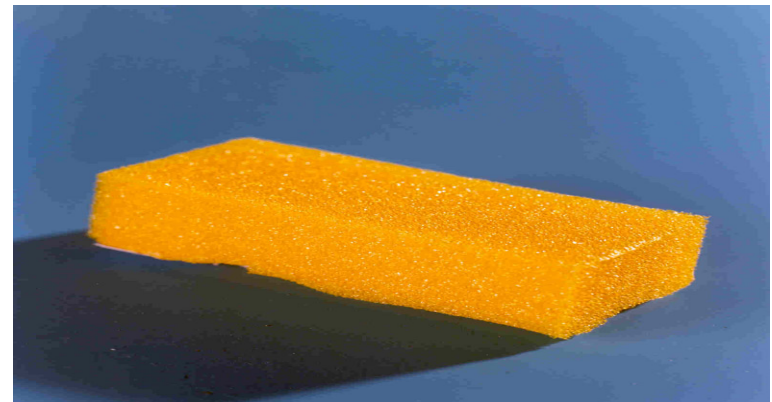
# DSC method for thermal conductivity

Heat Transfer for sapphire



Problem: waviness in foams – thermal issue

- Model heat transfer
- Measure (T, heat flux) over time
- Model/measure shrinkage
- Calculate internal stresses
- Use bending theory to predict curvature



# Standards in Thermal Properties Measurement

### **ISO 11357 Plastics - Differential scanning calorimetry (DSC)**

ISO 11357-1: 1997 Part 1: General principles (now due for revision)

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/FDIS 11357-4 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

***Potential proposal for thermal conductivity measurement by temperature modulated DSC***



### **ISO/AWI 22007 Plastics - Determination of thermal conductivity and thermal diffusivity**

ISO/AWI 22007-1 Part 1: General principles

ISO/AWI 22007-2 Part 2: Gustafsson hot-disc method

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

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

### ***Hot Wire***

- ISO 8894-1:1987 Refractory materials - Determination of thermal conductivity - Part 1: Hot-wire method (cross-array)
- ISO 8894-2:1990 Refractory materials - Determination of thermal conductivity - Part 2: Hot-wire method (parallel)

### ***Line Source***

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

### ***Laser Flash***

- ISO 18755: 2005 Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of thermal diffusivity of monolithic ceramics by laser flash method

### ***Guarded Hot Plate***

- ISO 8302:1991 Thermal insulation - Determination of steady-state thermal resistance and related properties - Guarded hot plate apparatus

### ***Guarded Heat Flux***

- ISO 8301:1991 Thermal insulation - Determination of steady-state thermal resistance and related properties - Heat flow meter apparatus
- ASTM E1530-04 Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by the Guarded Heat Flow Meter Technique

# Heat transfer project concluding summary

# 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
  - 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/npl/cmmt/polyproc>

# The next 6 months

- Complete commissioning and trials on heat transfer coefficient equipment
- Industrial demonstrations (Corus / Zotefoams) to be completed
- Dissemination of thermal conductivity measurement work
  - scientific and conference paper, articles

# 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

## **Your:**

Ideas,  
comments,  
suggestions,  
participation,  
contributions, ...

to steer the project to maximise the  
benefits to you.



# Heat Transfer Future Needs

## 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

## Improved heat transfer could:

- Contribute significantly to reduction in UK energy bill
- Bring indirect benefit to quality of life
- Save money for UK industry

## Areas where future work to increase understanding of heat transfer required:

- Water assisted injection moulding (WAIM)
- Gas assisted injection moulding (GAIM)
- Effect of air gaps, mould materials, supercritical CO<sub>2</sub>, helium
- Micro-moulding
- Additives, fillers effect on decreasing thermal conductivity of insulators
- Developing techniques for measuring heat transfer properties of foam
- Curing of fibre/matrix composites and cross-linking of rubbers

**Further areas where future work to increase understanding of heat transfer required:**

- Effect of nanoparticles on heating and cooling of polymer nanocomposites during processing
- Effect of dispersion of nanoparticles on thermal conductivity and heat transfer coefficient of nanofluids
- Measurement of heat transfer within microfluidic systems to improve data available for modelling
- Investigation of heat transfer during processing of foods for packagers and processors
- Development of techniques for increasing heating/cooling rates for food
- Measurement of surface heat transfer coefficient and external heat transfer medium (water, air) for range of foods

Your suggestions/comments?

**AOB:**