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Melts during Flow**

**S Chakravorty, C R G Allen
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ABSTRACT

The purpose of this report is to assess various temperature measurement methods that are capable of operating during polymer melt flow.

Temperature measurement and control are important to industry as they can have a profound effect on productivity and product quality.

The attributes of six methods reported in the literature have been considered briefly. Three methods have been studied in more detail. An auto-traversing thermocouple was found to give good repeatability ($\pm 1^{\circ}\text{-}2^{\circ}\text{C}$) and agreed well with a temperature measurement device (2nd method) calibrated at NPL (to within $1^{\circ}\text{-}2^{\circ}\text{C}$). It was, however, found to have a response time of 2-3 sec which is too long for some applications.

An infra-red probe, which has a much faster response time (~ 0.01 sec), was also investigated. It was found to require careful calibration, installation and use and was not found suitable for very thin sections (~ 1 mm). It is recommended for use in injection moulding applications with certain reservations.

This report is part of Project PMP4 (MTS programme on Processability). Within this project, temperature measurement is task 3.1.

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Approved on behalf of Chief Executive, NPL, by Dr M K Hossain,
Head, Division of Materials Metrology

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1 INTRODUCTION

Accurate and reliable measurement of a molten polymer's temperature is essential in most polymer processing operations because of its influence on production rates, material consumption, appearance and mechanical properties of finished parts. This is especially true for the injection moulding and extrusion processes, where the temperature profile is one of the most important criteria for the successful production of consistent parts.

In many cases, to obtain maximum throughput, polymers are often processed at temperatures just below the temperatures at which degradation or discolouration can occur. Hence, correct temperature measurement is essential to ensure that the polymer is not overheated or degraded in the processing equipment. In addition, the viscosity of all polymer melts is influenced by the temperature of the melt. Consistent quality and output are dependent upon the consistency of the melt's viscosity, especially in tight tolerance operations. Accurate melt temperature measurement with confidence and its control should, therefore, be a prime concern to all processors.

In practice, in-line determination of a polymer melt temperature and its distribution is difficult (1). The temperature variations, axially and radially, during processing can be significant. Also, the accurate measurement and the control of the temperature become more difficult to attain due to a number of errors associated with in-line measurement. The temperature gradient between the melt and the die wall is often large due to temperature gradients in the processing equipment, conduction effects and shear heating, especially for more viscous polymer melts at high flow rates. The situation is further complicated by the fact that most polymer melts have relatively low thermal diffusivity values. In addition, most conventional methods of measuring melt temperature cause a disturbance to the melt flow field. This can affect the accuracy of the temperature determination and cause problems related to melt flow stagnation, such as localised degradation. The ideal method for melt temperature measurement, therefore, must not only reflect the absolute local temperature, but it must also have as little effect on the polymer flow as possible.

It is probable that most industrial melt temperature measurements are subject to significant error and that often the error is large enough to influence quality, technical judgements and conclusions. The economic loss to the plastics industry attributable to errors in melt temperature measurement is undoubtedly large.

The benefits of precise melt-temperature control are many. For example, the determination of the melt temperature profile in blown film extrusion enables the user to reduce peak profile temperature during processing by adjusting temperature-zone settings. This leads to an increase in throughput. Similar benefits are achievable in sheet extrusion, where temperature-profile control can minimise gauge variation.

The objectives of this research are:

- 1) to assess briefly a number of temperature measurement methods currently available.
- 2) to review any novel techniques that have been reported.
- 3) to recommend at least one reliable and accurate temperature measurement method, which is likely to be widely taken up in production by the plastics industry based on the relevant experimental work.

2 LITERATURE SURVEY

The temperature and pressure of polymer melts are considered to be the most critical parameters in plastics processing. Small variations in temperature and pressure have a large effect on flow and may upset the production process. Accurate melt temperature measurement, that is independent of the temperature of the processing equipment is required to obtain optimum physical properties with minimum variation. This requirement is present in both extrusion and injection moulding as well as in the compounding of thermoplastics and thermosets (2-9).

Two fundamental characteristics of polymer melts would necessitate the demand for accurate and reliable temperature measurement:

- a) Polymers can be degraded if exposed to a time-temperature history outside their normal operating windows.
- b) Polymer flow characteristics may vary from batch to batch or within a batch, depending on precise time-temperature history.

Melt temperature measurements are normally affected by a number of errors and consequently the accuracy and reliability of any particular method depends very much on minimising possible sources of errors.

A conduction error caused by the existence of boundary layers (which act as insulators) in the melt flow, is a principal problem in melt temperature measurement. The flush or wall mounted thermocouples are largely affected by this error. In the case of the immersed sensors, there is an additional conduction error caused by conduction of heat along the probe to the cooler exposed flange, establishing a temperature gradient along the probe. This heat is replaced by transfer of heat from the polymer to the probe. This type of error is frequently observed and can be as great as 20 °C (1).

A second type of error that is usually significant is the shear (friction or viscous) heating of melt flowing past a wall. Mechanical work converts to heat when forced through a conduit and raises the temperature of the melt. Since the shear rates are highest near the wall, the polymer near the wall will have the highest temperature rise.

As the melt flow is typically laminar with no mechanism for radial mixing and the thermal conductivity of the melt is low, the net effect is a radial temperature gradient. This is a third source of error, between conduit wall and the central core of the melt stream. Methods of measurement of radial temperature profiles has been proposed by many authors (eg 10-12).

An additional factor contributing to the shear heating error, is the presence of a temperature probe causing drag in the melt flow. This can increase the temperature of melt flowing near the probe above the bulk temperature and cause further positive errors in the melt temperature measurement.

2.1 TEMPERATURE MEASUREMENT TECHNIQUES

The following measurement methods were considered:

a) Infrared with Fibre Optic Sensor

This is basically a non-intrusive instrument with high sensitivity and fast response time (10 millisecc) (13-17). However, the accuracy largely depends upon proper calibration, installation and characterisation of the IR absorption spectrum of the material being

measured. For materials that are opaque to IR, the instrument gives the temperature from near the surface. For highly transparent materials it is possible that the majority of the signal can come from the back wall, rather than from the polymer melt. Appendix-1 gives the details of the instrument used in this study.

b) Auto-traversing Thermocouple

The instrument is sturdy with programable operation and is capable of measuring through thickness temperature up to a depth of 38 mm (4,18,19). It is cheaper than IR instruments, moderately accurate with a relatively slow response time of 2-3 secs and could be non-intrusive when flush mounted. The details of the instrument are given in Appendix-2.

c) 'Cheese cutter' Thermocouple

This is a cheap and accurate method of determining temperature by placing a bare thermocouple at the centre of the die exit. The molten polymer is forced past the thermocouple under pressure. A type 'T' thermocouple is used with an accuracy of $\pm 1^\circ\text{C}$ and with a relatively fast response time (1-2 secs). The method is, however, not suitable for practical use in the process line, but could be used to form a base line for determining accuracy and validating other instruments.

d) Ultrasonics

This is a non-intrusive novel method with potential for much wider on-line use (20-24). The drawbacks are that it is a pressure sensitive technique with response time limited by the signal processing time and the level of accuracy required.

e) Refractive Index

The method is novel and currently undergoing many technical and scientific developments (25-31). The method, in its concept, is non-intrusive and capable of recording average temperature across the section. It is, however, pressure sensitive and needs appropriate calibration and validation before the data could be accepted. The technique is applicable only to transparent material.

f) Temperature Sensitive Tracers

This is another novel area of temperature measurement (applicable primarily to extrusion), where six emerging technologies can be identified (32-41).

- 1) Metal particles with a range of melting points.
- 2) Metal particles (eg beta bronze) that undergo recrystallisation.
- 3) Metal particles displaying temperature dependent magnetic properties.
- 4) Diacetylene monomers changing colour on polymerisation.
- 5) Metal powders with thermochromic coatings.
- 6) Solid state polymer reaction changing dielectric constant.

Before any form of tracers could find commercial use, the following criteria should be met:

- an irreversible change on exposure to the desired time/temperature history
- a shear independent change
- an option to analyse the change ex-situ
- a change that does not alter what is being measured
- inexpensive
- non-toxic

It is concluded from above, that item 1) ie metal particles with a range of sizes, shapes and melting points and item 5) ie thermochromic coated metal powders have the potential to meet most of the above criteria and perhaps find commercial use in the future. However, in general only the peak temperature can be identified by tracers within an accuracy of about ± 10 °C, with little information on exposure time. At best, temperature sensitive tracers will indicate that some critical temperature has been exceeded. Off-line analysis is inconvenient and time consuming particularly if it involves SEM or light microscopy. The technique is slow, complex and imprecise and unless automatised in some form, is unlikely to be taken up by the processing industry.

Of the six methods described above the first three (a)-(c) have been selected for more detailed assessment and are reported in the following sections. It is also planned to carry out a brief experimental assessment of the potential of the ultrasonic technique (d) to measure the temperature of flowing polymer melts.

3 EXPERIMENTAL PROCEDURE

A medium density polyethylene (MDPE) material supplied by BICC (Batch No NCPE 6032), NPL code 'HAD000' was chosen for this work.

The temperature measurement was carried out in a special Davenport extrusion rheometer barrel with a stainless steel slit die flow cell attached by means of an adapter head (Fig 1). This adapter is insulated although unheated. A screw driven Instron (Model No 1115) machine operated the piston of the barrel at speeds of 2 - 50 cm/min. The rheometer barrel had a 19 mm internal diameter with 280 mm length and the temperature was controlled to $200^\circ \pm 1$ °C by a Eurotherm temperature controller unit with a feedback system. The actual temperature of the melt within the barrel was checked by inserting a PRT (Platinum Resistance Thermometer) probe into the melt approximately 10 mm above the slit die entry plane. Each charge in the barrel had been held at the test temperature for 20 minutes to attain equilibrium.

The slit die flow cell attachment was independently heated by placing cartridge heaters along the length and controlled at $200^\circ \pm 1.0$ °C by a Eurotherm unit with a feedback system.

The slit die was 100 mm long x 25 mm wide with the provision to vary the channel thickness between 1-6 mm (in this work) or higher. The piston speed in most cases was 50 cm/min, giving a flow rate of $2200 \text{ mm}^3/\text{sec}$ in the die. This produced a shear rate of 5 sec^{-1} in the 6 mm thick die and a shear rate of 30 sec^{-1} in the 1 mm thick die respectively.

A limited number of tests were carried out with a very slow piston speed of 2 cm/min, giving a flow rate of $90 \text{ mm}^3/\text{sec}$ and a shear rate of 1.3 sec^{-1} in the 1 mm plate die. Both the IR probe and the auto-traversing thermocouple were unable to detect any temperature change near the entry point of the die. However, the "cheese cutter" thermocouple detected a temperature rise of 2 °C (maximum) at the exit of the die due to viscous heating.

All thermocouples and PRT probes employed in measuring temperature were calibrated at NPL with reference to the primary standard. The Autoprobe 1A auto-traversing

thermocouple and infrared (Model MTX922) probe, supplied by Dynisco, were factory calibrated.

The Autoprobe with a type 'J' thermocouple and the IR probe had measurement accuracies of ± 2 °C. The IR probe was not recommended by the manufacturers for measurement of melts with thicknesses <2 mm. The "cheese cutter" thermocouple was calibrated at NPL with known uncertainty.

The results from the IR probe and the Autoprobe were validated in this work by comparing the data generated by the "cheese cutter" thermocouple operated under near identical conditions.

Initial checks on the temperature of the slit die were made by a 'cement-on' thermocouple, supplied by Omega. The "cheese cutter" thermocouple was used in all test runs at the exit of the slit die (Fig 1). Both the Autoprobe thermocouple and the IR probe were employed to measure temperature at the point T1 near the entry, and at the point T5 near the exit of the slit die. The Autoprobe was also used to measure the through thickness distribution of temperature in the 6 mm slit die by positioning it at 0, 1.5 and 3 mm distant from the die wall. For the 1 mm slit die, the positions were 0, 0.3 and 0.5 mm respectively. The IR probe was used without air purge at the 0 mm position in all cases.

4 RESULTS AND DISCUSSION

4.1 INITIAL SET-UP

The PRT probes, measuring barrel wall and die wall temperatures, showed good stability at $200^{\circ} \pm 1$ °C in all cases. The temperature change during a test was insignificant in both barrel and die walls. Every extrusion tests were identical at the given shear rate and lasted for ~15 secs.

The OMEGA 'cement-on' thermocouple placed on the die wall, read ~4 °C below (ie 196.3 °C) the set temperature of 200 °C under static conditions. It is however, economic and easy to use, and therefore recommended in those cases, where a high degree of accuracy is not a prerequisite. It is unsuitable for measuring temperature during flow.

The melt temperature of the charge in the barrel was controlled at $195.5^{\circ} \pm 0.3$ °C in all cases and the volume of the charge forced through the barrel during each run, was identical. However, the adapter head area between the barrel and the slit-die was considered a "cold spot" inspite of insulation lagging in that area. It was estimated, that the melt temperature at the entry point to the slit die was reduced by ~2°-3 °C. It was also estimated that the actual temperature of the inner wall of the slit-die at the beginning of each extrusion test varied from $\sim 193.5^{\circ} \pm 1$ °C at the entry, to $\sim 197^{\circ} \pm 1$ °C at the midpoint, and $\sim 194^{\circ} \pm 2$ °C at the exit.

4.2 PERFORMANCE ASSESSMENT

The emphasis of this work was placed on evaluating the performance of the Autoprobe and the IR probe for accuracy and reliability in temperature measurement under near identical conditions with reference to the NPL calibrated "cheese cutter" thermocouple of known uncertainty (± 1 °C).

The Autoprobe and the IR probe were tested initially for their relative accuracy by placing them in the 6 mm thick hot die with no material and showed similar temperature recordings to within ± 2 °C in the enclosed air gap.

The temperature recordings by all methods under the static condition of the polymer melt can be determined from any of the Figs (2-9 and 12-16) by referring to the corresponding reading on the temperature axis at zero time in the time/temperature plots.

The repeatability of all three methods was established and all showed excellent results (Figs 2-6). The repeatability of the temperature recordings in each case varied between $\pm 1^{\circ}\text{-}2^{\circ}\text{ }^{\circ}\text{C}$.

The readings during steady flow from the Autoprobe agreed to within $1^{\circ}\text{-}2^{\circ}\text{ }^{\circ}\text{C}$ of the "cheese cutter" readings at the similar positions (Figs 5,7,8). The Autoprobe manufacturer's specification on accuracy was $\pm 2^{\circ}\text{ }^{\circ}\text{C}$.

Temperature rises due to viscous heating in the capillary rheometer were determined to be $\sim 8^{\circ}\text{ }^{\circ}\text{C}$ at the exit and $\sim 4^{\circ}\text{ }^{\circ}\text{C}$ at the near entry point in the case of the 30 sec^{-1} shear rate (Figs 5,6,9). For low shear rate (5 sec^{-1}), the viscous heating effects were found to be between $\sim 2^{\circ}\text{-}4^{\circ}\text{ }^{\circ}\text{C}$ at both entry and exit points in the die (Figs 12-16).

The through thickness distribution of melt temperature during flow was measured by the programable Autoprobe instrument, which showed a temperature variation of up to $2^{\circ}\text{ }^{\circ}\text{C}$ (Figs 5,6,12,13). For the low shear rate (5 sec^{-1}), the temperature increased from the wall to the centre of the melt flow. However, at high shear rate (30 sec^{-1}), due to large shear heating effects near the wall, the trend in temperature distribution in the melt remained unclear (Figs 5,6).

The IR probe was found to be unsuitable in its present specification to measure temperature successfully in 1 mm thick dies. The IR wavelength range of $1.6\text{-}2.2\text{ }\mu\text{m}$ of the instrument was found to be inappropriate for a melt thickness of 1 mm, since the metal wall instead of the polymer would be measured. Elsewhere a wavelength of $3.4\text{ }\mu\text{m}$ for an IR probe has been recommended (42). For thick samples ($>6\text{ mm}$), the IR probe measured the polymer temperature almost instantaneously (response time 10 millisecc) (see Figs 12 and 13) as compared to the Autoprobe, which, with a large thermal mass, had a response time of 2-3 secs. The IR probe was also found to be highly sensitive.

It can be seen from Figs 12 and 13, that for the entire part of the extrusion tests, the IR probe read consistently $\sim 4^{\circ}\text{-}5^{\circ}\text{ }^{\circ}\text{C}$ higher than the well characterised autoprobe readings at 0 mm positions. This difference can be attributed to the lack of proper calibration, installation, positioning or the absence of air purging through the probe (in cases where the barrel and the die temperatures are similar, an incorrect measure of temperature rise of 2-3% from the actual melt temperature can be expected). The manufacturer's limit on accuracy was also stated to be $\pm 2^{\circ}\text{ }^{\circ}\text{C}$; hence, as observed, the cumulative effect was a higher temperature recording.

The IR probe is therefore recommended, in particular, for processes requiring fast response time, ie injection moulding; but extreme caution is necessary regarding its installation and calibration with respect to material, emissivity, melt thickness, spectral range, probe positioning and air purging.

The Autoprobe auto-traversing thermocouple, on the other hand, is a relatively economic, reliable and fairly accurate instrument. The Autoprobe can be operated in a non-intrusive manner with a response time of 2-3 secs. However, a type 'T' thermocouple instead of 'J' is recommended for better accuracy ($\pm 1^{\circ}\text{ }^{\circ}\text{C}$). It is regarded therefore, to be useful for continuous processes, such as extrusion (modification to the equipment to withstand pressure greater than 55.2 MPa (8000 psi), is also recommended).

4.3 MODELLING

A limited amount of work on predictions of temperature rises in similar experimental conditions by the Fillcalc V and Moldflow modelling packages is shown in Figs 10 and 11 (for a 1 mm thick plate) and in Figs 17 and 18 (for a 6 mm thick plate). The Moldflow predictions were based on temperature monitoring points, 90° off-axis on the flow cell from the experimentally measured points and hence, can only be used for an approximate comparison.

It was found from this initial comparison that these two modelling packages were not inconsistent with the experimental results. It should be noted however, that these were preliminary results and that both these models have significant potential to produce better results. Further work is planned to examine the significance of heating effects using these numerical models.

5 CONCLUSIONS

- The repeatability of temperature measurements using an infrared probe, an Autoprobe auto-traversing thermocouple and a "cheese cutter" thermocouple was found to be excellent (within $\pm 1^\circ - 2^\circ \text{C}$).
- The measured temperatures of the Autoprobe agreed well with the "cheese cutter"; with an accuracy generally $1^\circ - 2^\circ \text{C}$ below that of the "cheese cutter", which has an uncertainty of $\pm 1^\circ \text{C}$ (Autoprobe manufacturer's accuracy specification is $\pm 2^\circ \text{C}$).
- The response time of the infrared equipment (manufacturer's accuracy specification $\pm 2^\circ \text{C}$) was found to be almost instantaneous (10 millisecc) as compared with the Autoprobe (2 - 3 sec). The IR probe was found to be highly sensitive and is most suited to measurement of rapid temperature changes.
- Temperature rises due to viscous heating in the capillary rheometer were between $\sim 2^\circ$ and 8°C for shear rate values of 5 to 30 sec^{-1} .
- The through thickness distribution of melt temperature was found to be small even at high strain rates (less than 2°C).
- The Autoprobe was found to be reliable and fairly accurate and is therefore recommended for continuous processes, such as extrusion. It can be used in a non-intrusive manner when flush mounted.
- The infrared probe is recommended, in particular, for processes requiring a fast response time, such as injection moulding. However, extreme care must be taken during installation and calibration with reference to emissivity, material, melt thickness, spectral range, probe positioning and air purging.

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Cross-section of Capillary Rheometer and Die

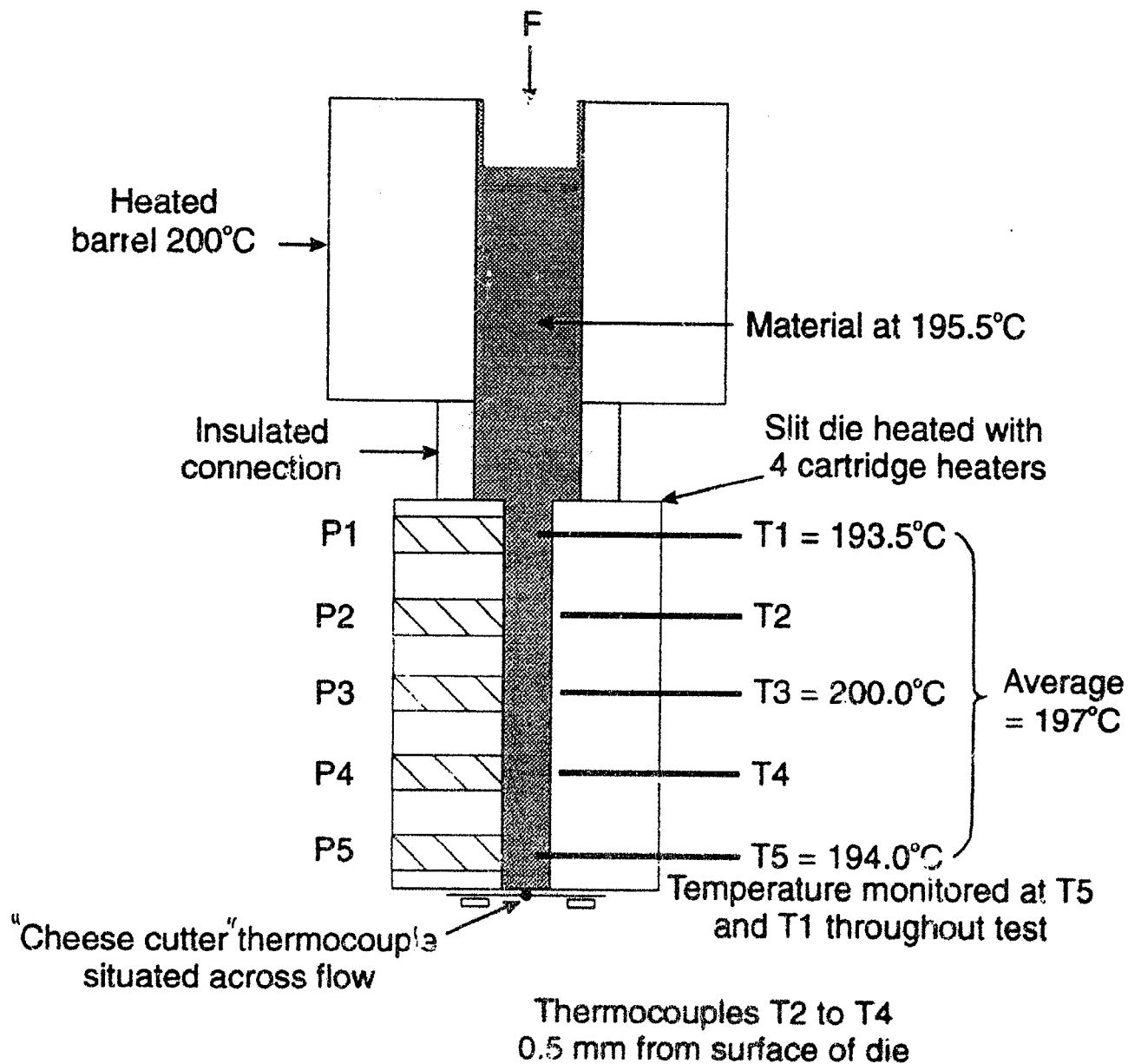


Figure -1

**Temperature Measurement at Exit by CheeseCutter for Tests at T5, Spacer Plate 1mm, Piston
Speed 50 cm/min, MDPE (HAD000)**

Melt Temperature 195.3 ± 0.1 deg C

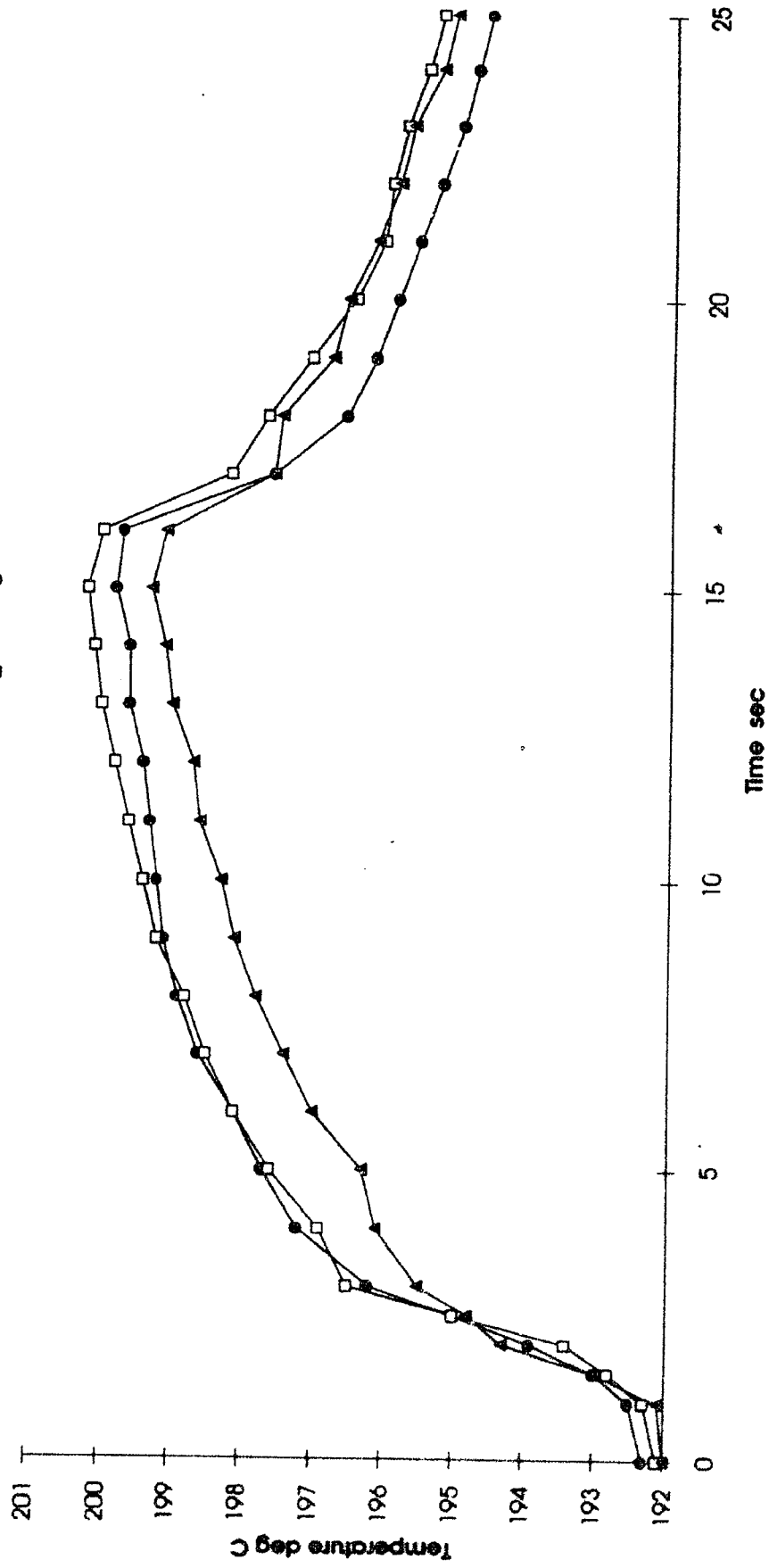


Figure - 2A

**Temperature Measurement by Cheesecutter for Tests at T5, Spacer Plate 6mm, Piston Speed
50cm/r.in, MDPE (HAD000)**

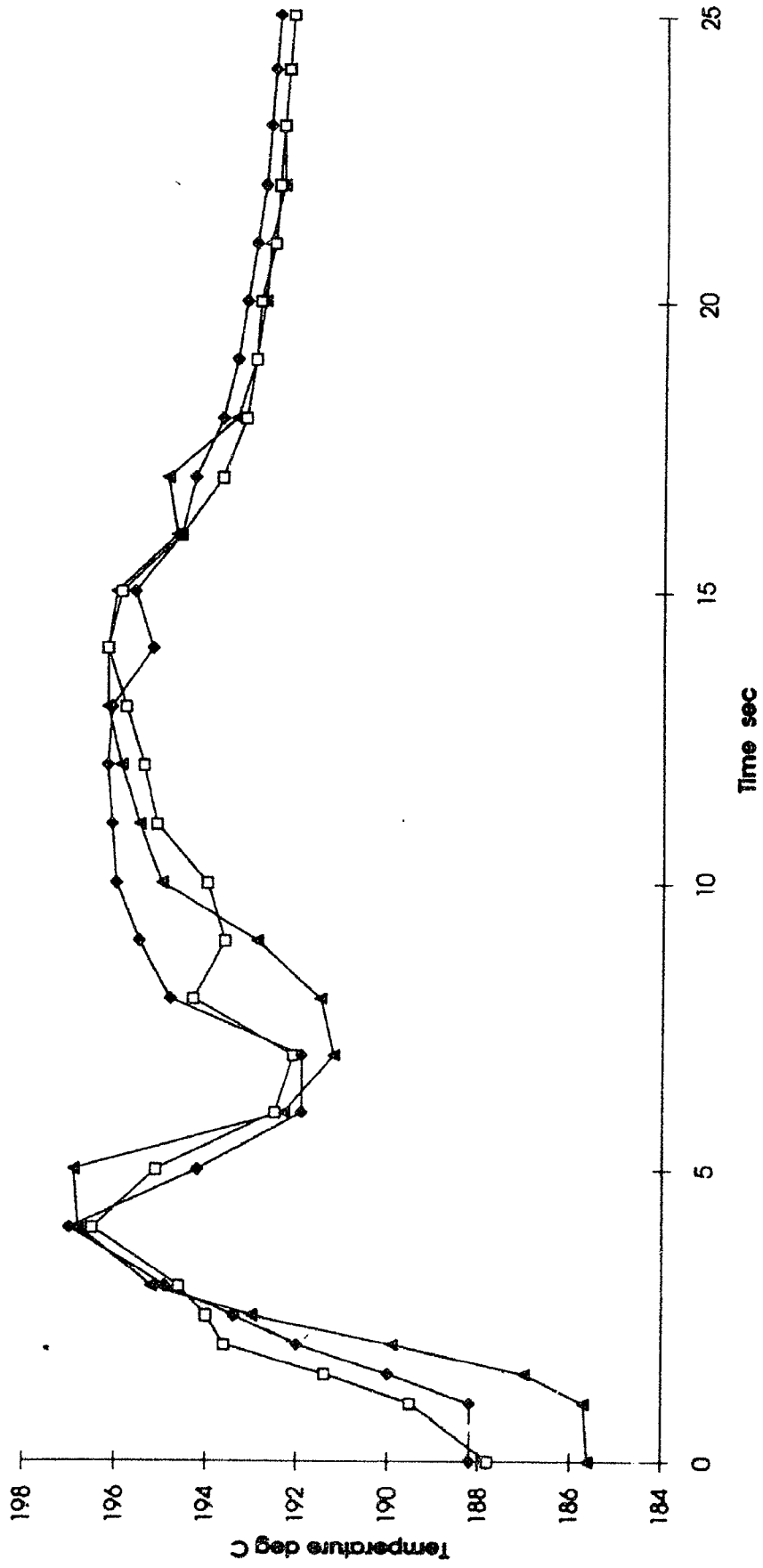


Figure - 2B

**Temperature Measurement at T5 with Infrared (0mm) and with Cheese cutter, Spacer Plate
6mm, Piston Speed 50cm/min, MDPE(HAD000)**

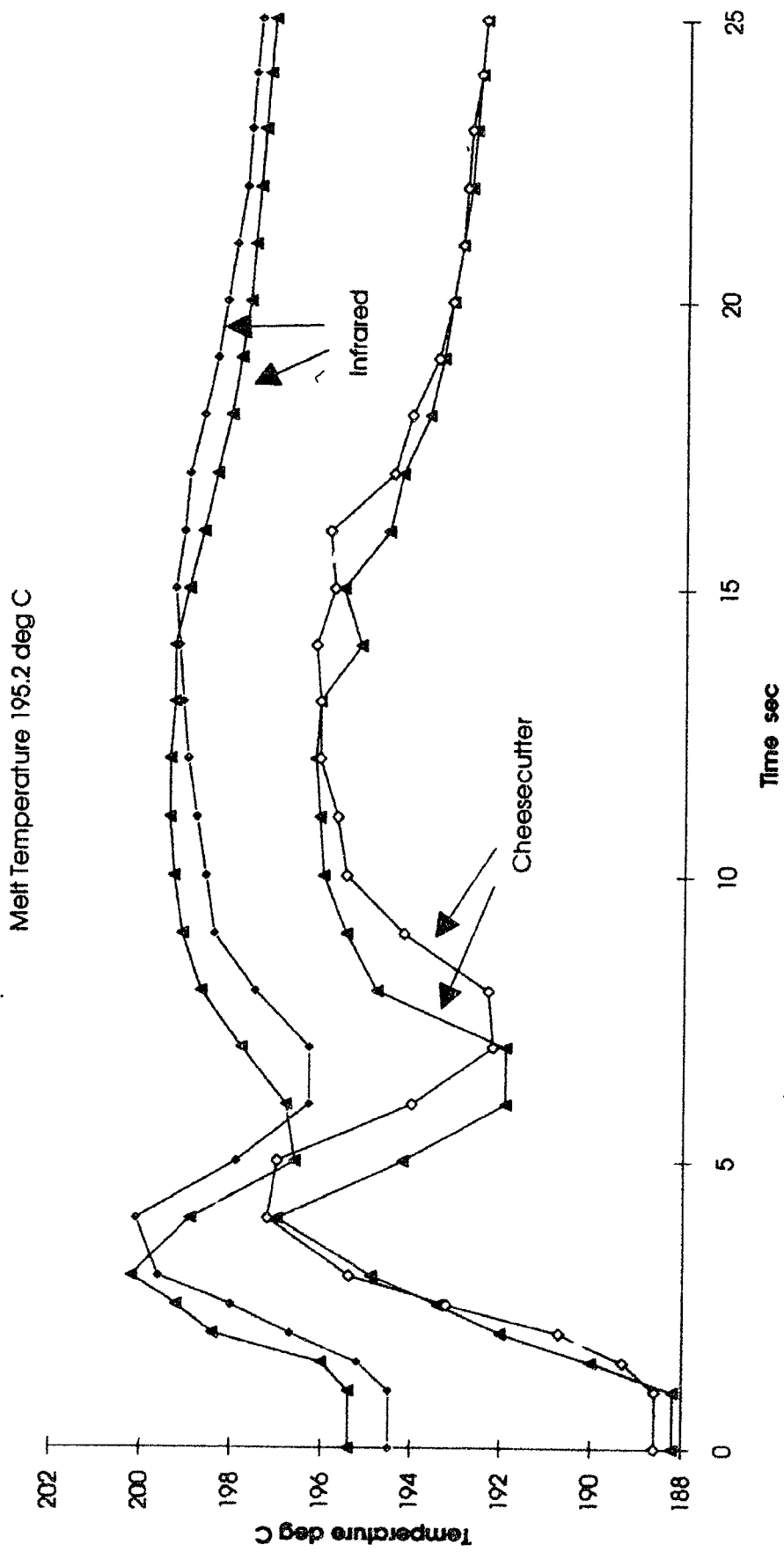


Figure - 3

