
pirouette
dilatometry for processing

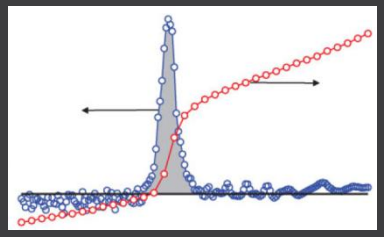
# Company profile

Technische Universiteit Eindhoven University of Technology

TECHNOLOGIES

Commercial partner and liaison for the Department of Mechanical Engineering

Specialized in the development and production of dedicated and innovative R&D equipment



Actively involved in polymer research and development of new dilatometer technologies

# Dilatometry

- Measurement of the specific volume of a material as a function of temperature and pressure

  *Pressure-Volume-Temperature (PVT) behavior*

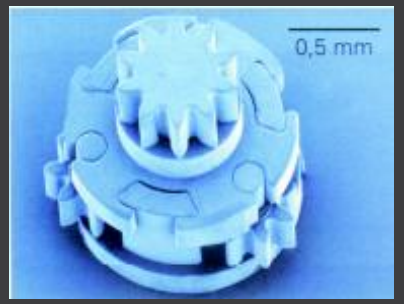
- PVT-behavior of polymers is of great importance for predicting the shrinkage behavior of polymers

- Quantitative prediction of shrinkage can lead to
  - time and cost reduction of process and mold optimization
  - shorter time to market of high precision polymer products

# Dilatometry fields of application

Lightweight construction

High-precision components




Mould design

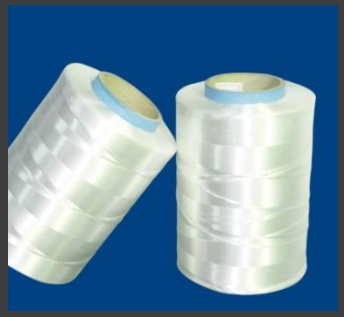
Polymer processing

Polymer research

Grade control

High-strength fibers

Bio-polymers



# PVT at realistic processing conditions

- Semi-crystalline polymers are widely used (e.g. PP, PE, ...)
- PVT-behavior of crystallizing polymers not well understood
- Crystallinity strongly determines specific volume and
  - crystallinity depends on pressure
  - crystallinity depends on thermal history (i.e. cooling rate)
  - crystallinity depends on experienced flow (i.e. shear)

- Injection molding and extrusion processing conditions require measurements of PVT-behavior at
  - elevated pressures of $\mathcal{O}(10^2)$ MPa
  - cooling rates of $\mathcal{O}(10^2)$ °C/s
  - shear rates of $\mathcal{O}(10^2\text{-}10^4)$ 1/s

# Our equipment design philosophy

- Safety
  - Equipment must provide safe working conditions
- Reproducibility
  - Experimental results can be accurately reproduced
- User-friendliness
  - Minimize time and skills required to use the equipment
- Versatility
  - Optimize usability and expansion possibilities

# Pirouette PVT apparatus


## Benefits

- Dilatometry at realistic processing conditions for injection molding
- Safe and easy-to-use system allows for much faster experimentation
- Absolute measurement of specific volume so no reference data needed

## Advantages

- Small sample volumes needed
- Automated control system and HMI
- Only standard power, water and compressed air supply required

## Features

- Cooling rates up to 140 °C/s
- Shear rates up to 180 $s^{-1}$
- Static pressures up to 1000 bar

# Pirouette specifications

## General

| | |
|---|---|
| Dimensions | 600x750x1950 mm (wxdxh) |
| Weight | 300 kg |
| PC outputs | 1x Ethernet RJ45<br>2x USB 2.0<br>CF/SD card reader |
| Lab connections | 230 V AC, 2 kW power supply<br>pressurized air at 5 bar<br>tap water 20 l/min at 4 bar<br>drain water pipe > Ø 32mm |

## Sample

| | |
|---|---|
| Inner sample ring diameter | 21 mm |
| Outer sample ring diameter | 22 mm |
| Sample ring height | 2.5 mm |
| Sample weight[1] (indicative) | 75 mg |

## Measurement conditions

| | |
|---|---|
| Pressure range | 0.1 – 100 MPa |
| Temperature range | 20 – 300 °C |
| Cooling rate | 0.1 – 140 °C $s^{-1}$ |
| Shear rate | 1 – 180 $s^{-1}$ |

## Displacement measurement

| | |
|---|---|
| Type | photoelectric linear encoder |
| Range | 5 mm |
| Resolution | ±0.1 µm |
| Accuracy | ±0.8 µm |

## Temperature measurement

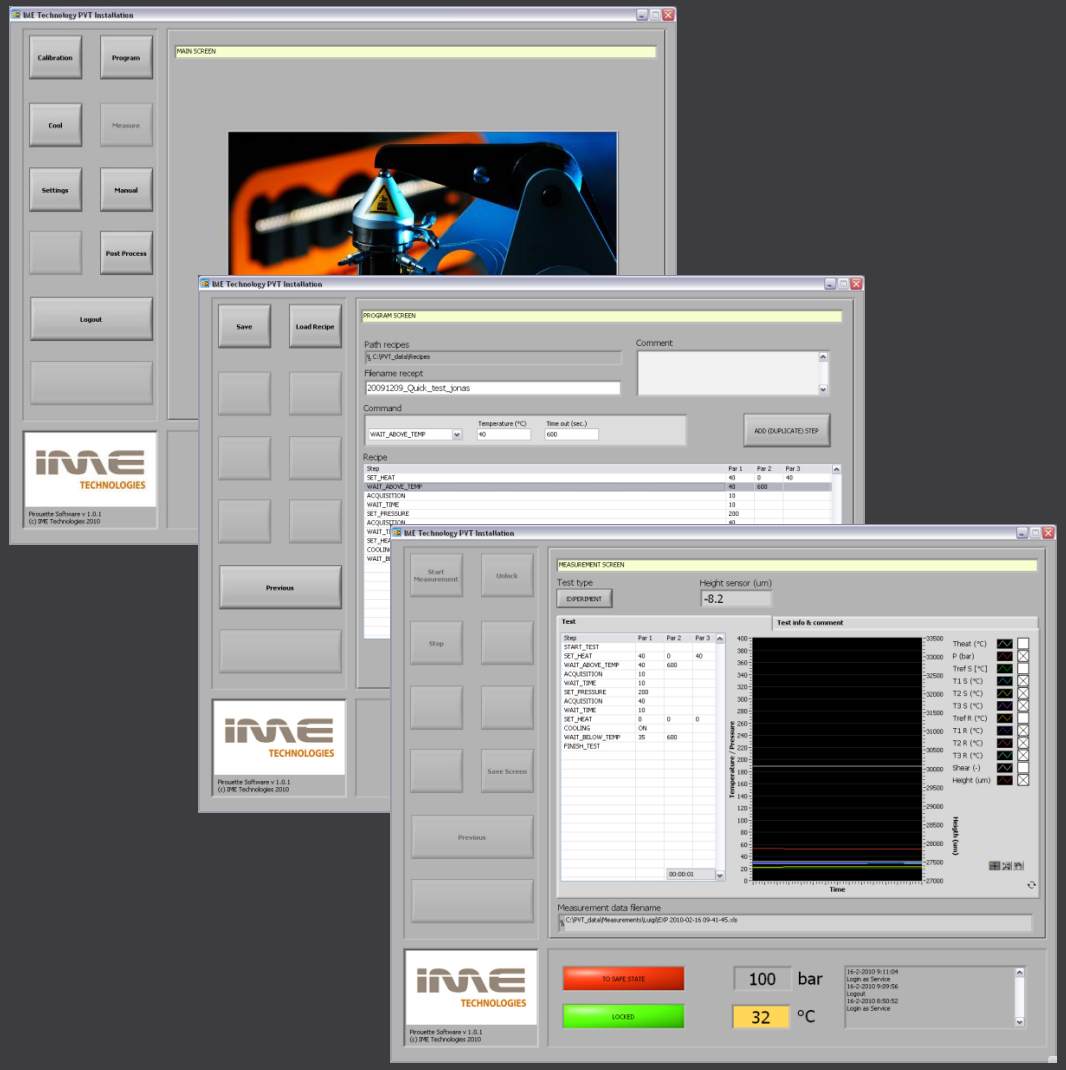
| | |
|---|---|
| Type | J-type thermocouples |
| Positions[2] | height 0, 1.25, and 2.5 mm<br>Ø 19.5 and Ø 23.5 mm |
| Range | 0 – 750 °C |
| Accuracy | ±2.5 °C |

## Process control

| | |
|---|---|
| Pressure control | electronic 3-way valve<br>0 – 6 bar inlet pressure<br>accuracy < 1.0 % of range |
| Temperature control | digital temperature controller<br>accuracy < 1% of range<br>750 W heating power |
| Shear control | digitally controlled servo<br>servo error < 1% of range |

[1] Indicative value for polypropylene; total amount of material needed depends on the material and production method.
[2] Heights specified relative to the bottom edge of the material sample.

# Pirouette control software

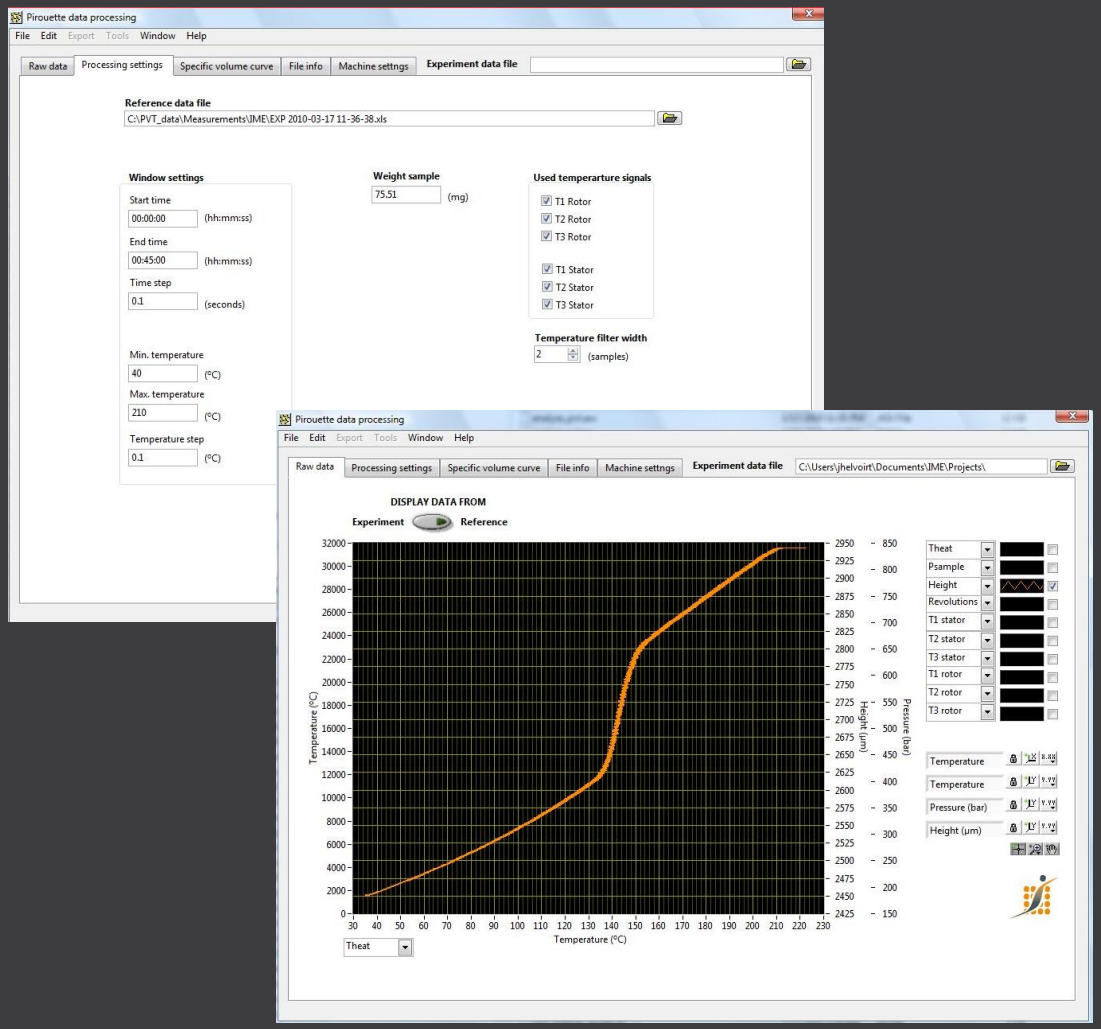



## HMI

- Intuitive user interface
- Measurement automation
- Robust touch-panel and keyboard
- Online monitoring of experiments
- Machine status and safety checks

# Pirouette data processing

Combining baseline and test data into PVT diagram



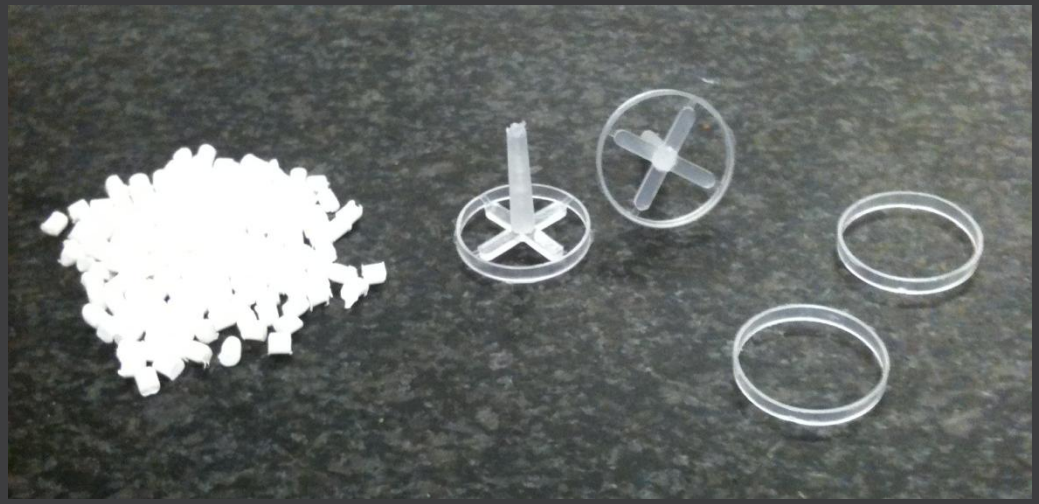

## Pirouette viewer

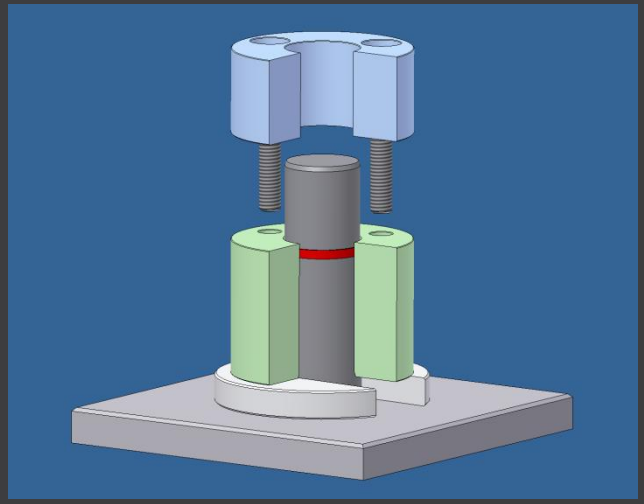
- Stand-alone processing tool
- Raw data visualization
- Data conditioning
- Automated PVT curve generation
- Temperature correction algorithm for quenching experiments

# Pirouette sample production

## Sample rings

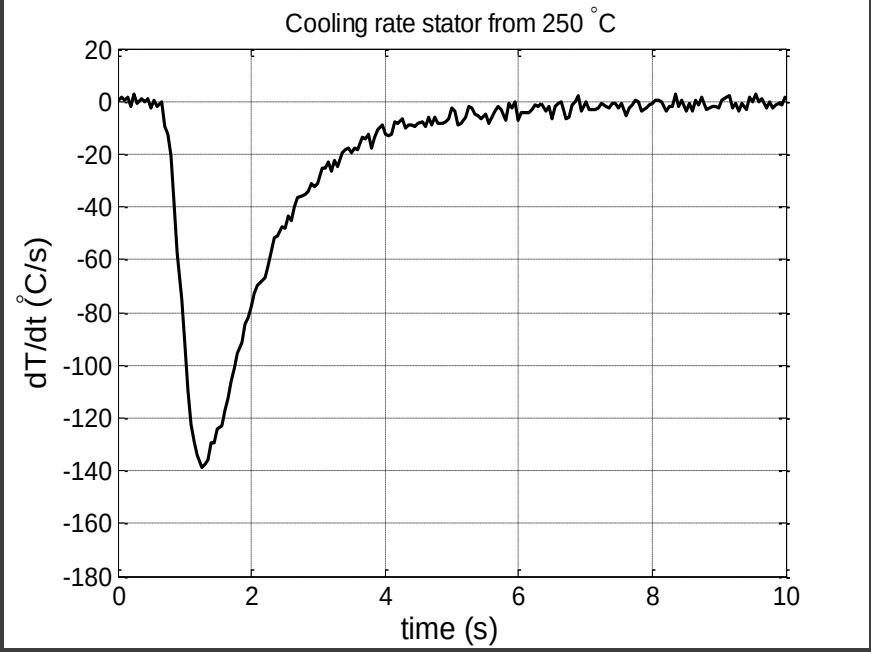
- Only a very small amount of sample material needed
- Produced with small injection molding machine like Babyplast
- Customized mold design available
- Powder press mold available for single ring production

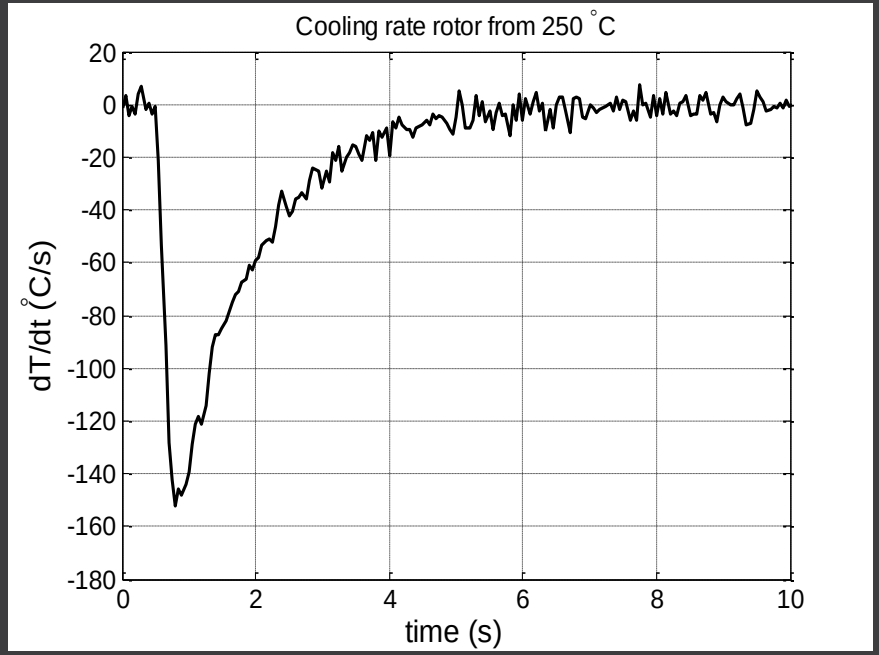




# Pirouette performance – cooling

Measuring the maximum cooling rate during quenching with water

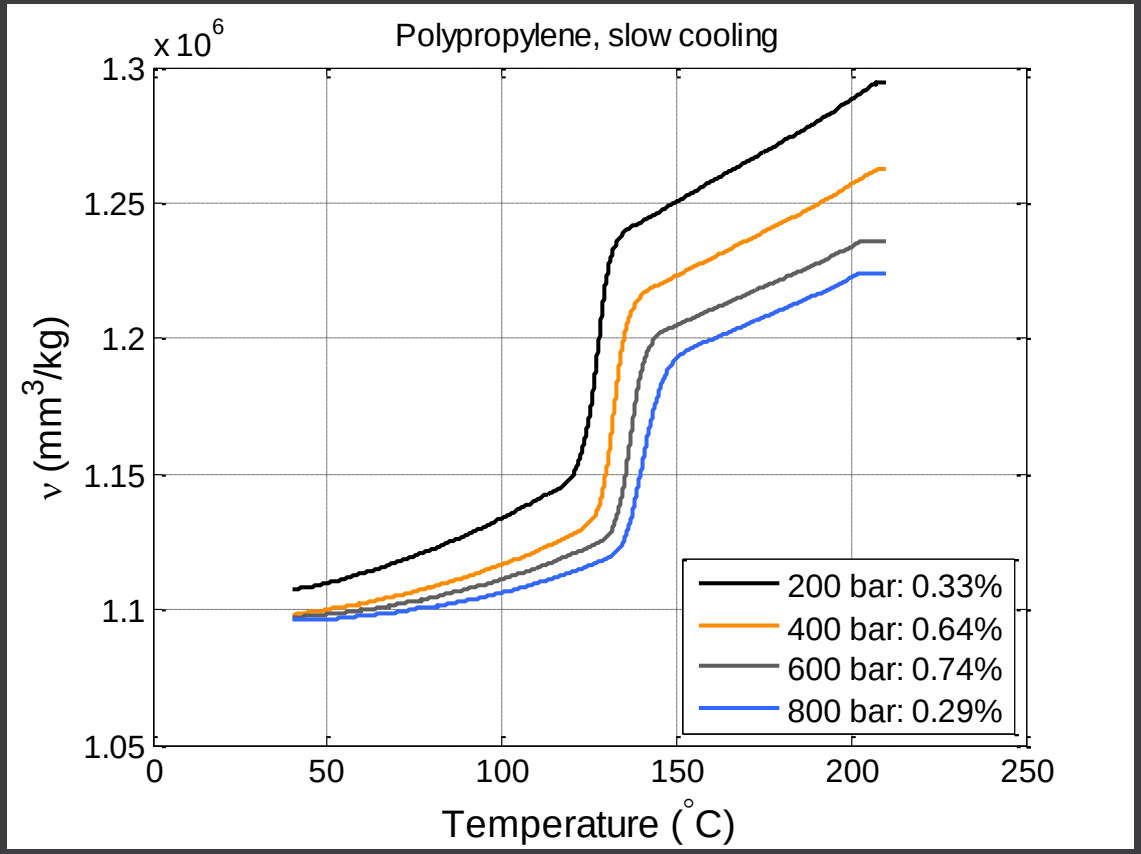



Cooling rate during quenching around 140 °C/s

# Pirouette performance – specific volume

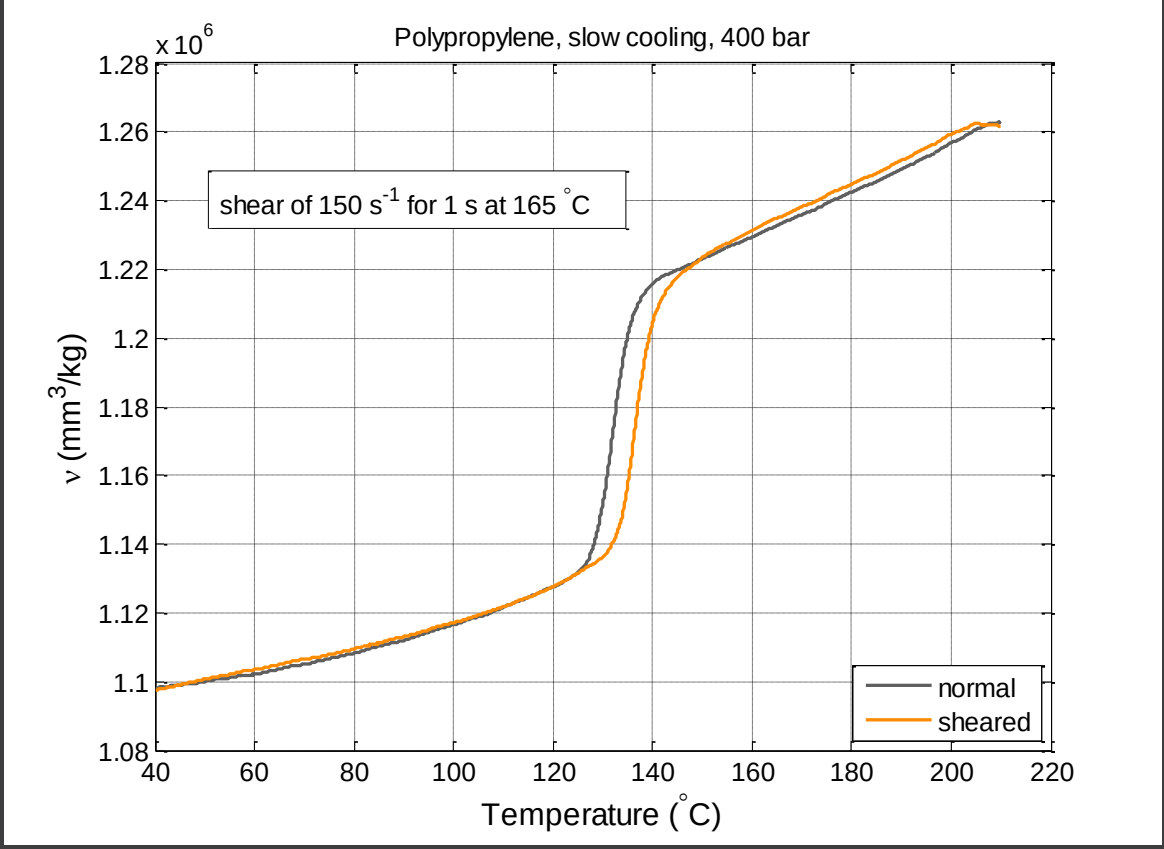
Specific volume measurements at different pressures




Specific volume at 200 °C within 1% of benchmark

# Pirouette performance – shear flow

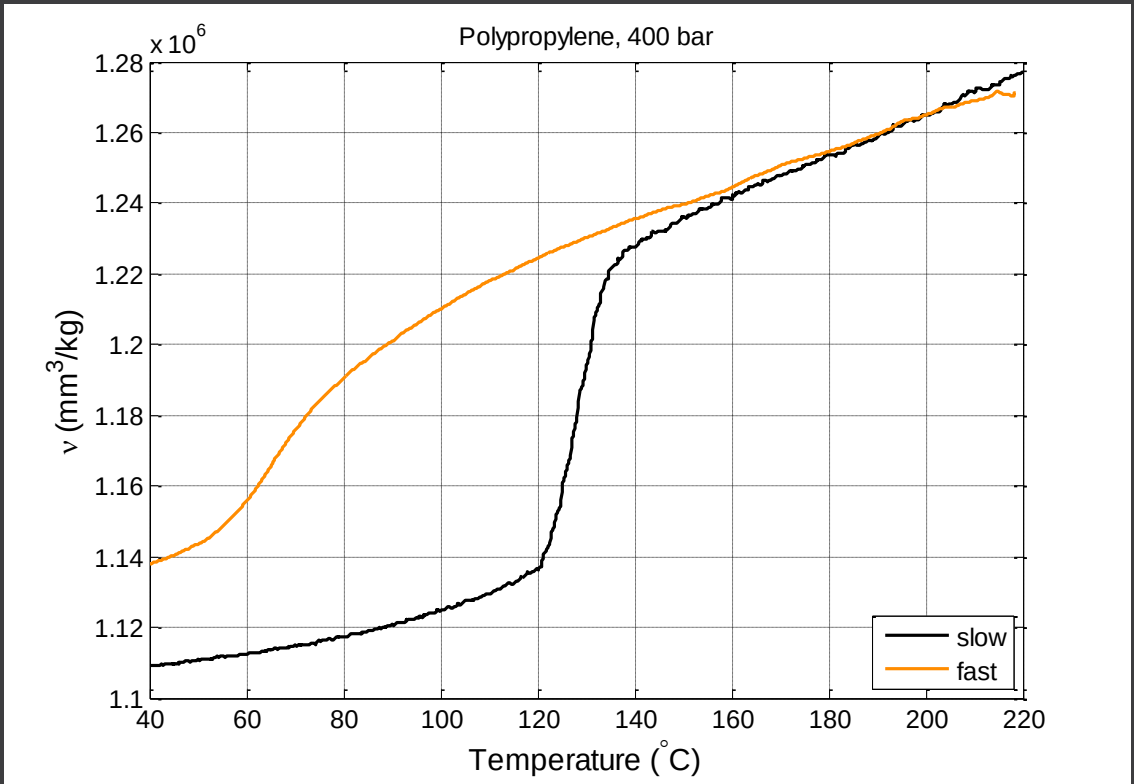
Shear applied to investigate the effect of flow on the specific volume




Shear flow accelerates the onset of crystallization

# Pirouette performance – cooling rates

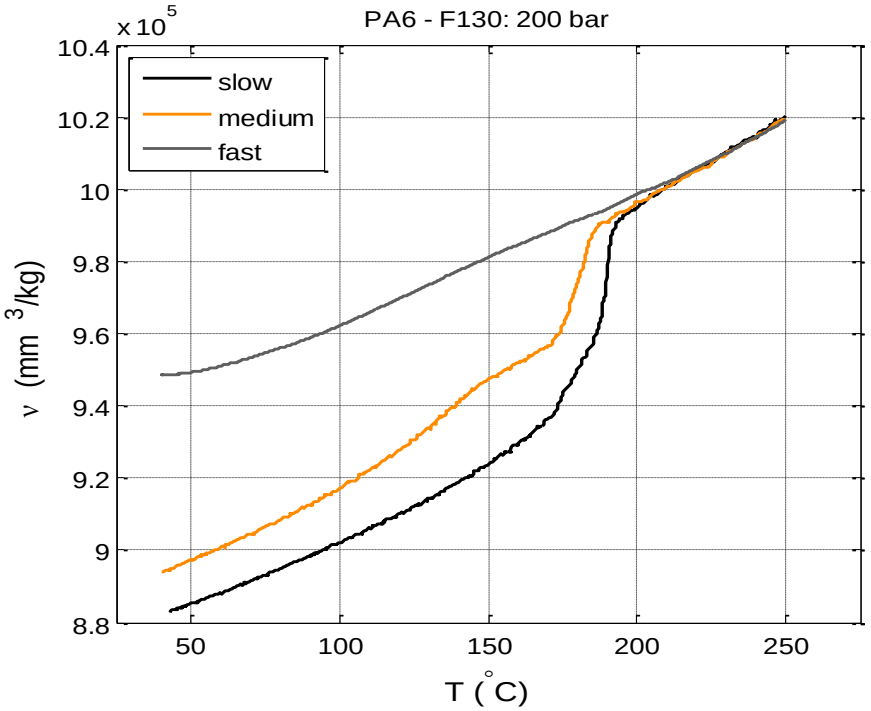
Studying the effect of cooling rates on the specific volume

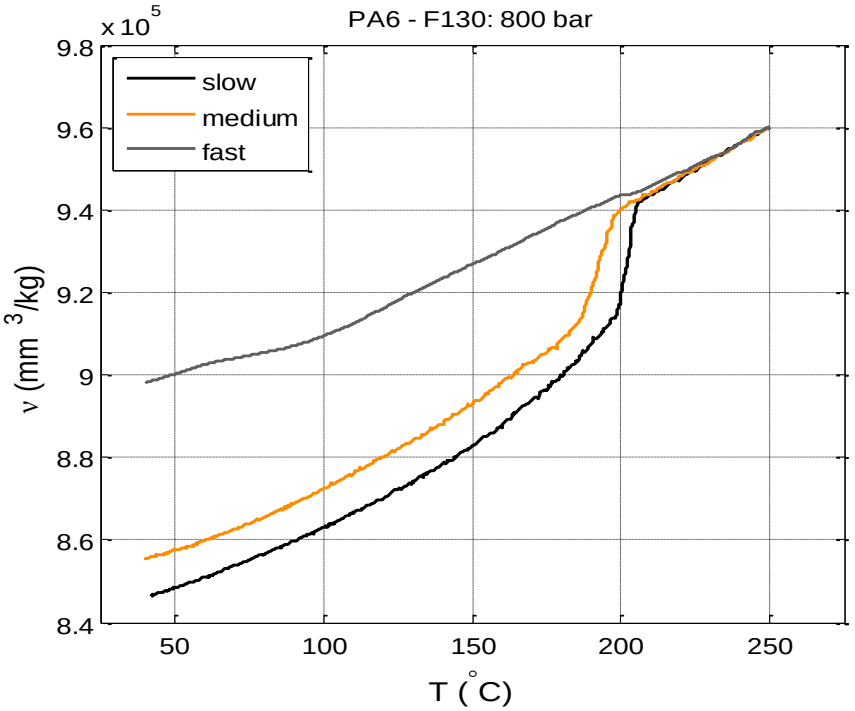



Onset and amount of crystallization affected by cooling rate

# Pirouette performance – nylon (PA) data

Unique measurements of PVT behavior of nylon




Very good results despite difficulties with sticking behavior of PA

# References

- Eindhoven University of Technology, the Netherlands
- DSM, the Netherlands
- Transfercenter für Kunststofftechnik GmbH, Austria
- Borealis, Austria
- SABIC, the Netherlands
- ...

# Pirouette sales information

## Standard deliverables

- Pirouette PVT apparatus
- Pirouette system cabinet with
  - Electronic control module
  - Process control module
  - Housing with connection panel
  - Touch-panel and keyboard
- Measurement & control software
- System documentation
- Set of PTFE confinement rings
- On-site installation and instructions

## Options

- Pirouette advanced data processing software
- Sample mold for Babyplast
- Powder press mold for single ring production
- Calibration and maintenance contract
- Various extended service level agreements

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## Contact

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# Scientific publications

Publications on PVT related research by the Polymer Technology group, TU/e


R. Forstner, G.W.M. Peters, H.E.H. Meijer, *Int. Polymer Processing*, pp. 114-121, 2009
A novel dilatometer for PVT measurements at high cooling – and shear rates

J.W. Housmans, L. Balzano, M. Adinolfi, G.W.M. Peters, H.E.H. Meijer, *Macromol. Mater. Eng.*, vol. 294, 2009
Dilatometry: A tool to measure the influence of cooling rate and pressure on the phase behavior of nucleated polypropylene

R. Forstner, G.W.M. Peters, C. Rendina, J.W. Housmans, H.E.H. Meijer, *J. Therm. Analysis & Calorimetry*, vol. 98, pp. 683-691, 2009
Volumetric rheology of polymers: The influence of shear flow, cooling rate and pressure on the specific volume of iPP and P/E random copolymers

M.H.E. van der Beek, G.W.M. Peters, H.E.H. Meijer, *Macromolecules*, vol. 39(26), pp. 9278-9284, 2006
Classifying the combined influence of shear rate, temperature, and pressure on crystalline morphology and specific volume of isotactic (Poly)Propylene

M.H.E. van der Beek, PhD Thesis, Polymer Technology group, TU/e, 2005
Specific volume of polymers: Influence of the thermomechanical history

M.H.E. van der Beek, G.W.M. Peters, H.E.H. Meijer, *Int. Polymer Processing*, pp. 111-120, 2005
A dilatometer to measure the influence of cooling rate and melt shearing on specific volume

M.H.E. van der Beek, G.W.M. Peters, H.E.H. Meijer, *Macromol. Mater. Eng.*, vol. 290(5), pp. 443-455, 2005
The influence of cooling rate on the specific volume of isotactic polypropylene at elevated pressures

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