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Rubber Molding Asia 2014

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Dr. Hans-Joachim Graf
Determination of the Shortest Possible Vulcanization Time in Rubber Injection Molding

TechnoBiz – Asia
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Bangkok, Thailand

Dr. Hans-Joachim Graf
Content of this presentation

- Introduction
- Operating window
- Porous point of different Rubbers
- Vulcanization time and physical properties
- Experiments guiding to shortest molding time
- Summary
The vulcanization time is Dependent from:

- Mold temperature
- Compound cavity entrance temperature
- Thickness of the part due to restricted temperature conductivity

Determined by trials

- No simulation tool, because of lack of precision
- Physical properties must be evaluated
Heat rise in injection unit

Reason for heat rise – shortening of the vulcanization time

End of injection
Finding of the Cycle Time in Injection Molding

Introduction
Porous Point
Cycle Time and Porous Point
Operating Window
Shortest Vulcanization Time
Summary
Regression equations to describe the injection molding process

\[
\begin{align*}
\text{TM}_{\text{plast}} &= f (V_{\text{Sc}}, P_{\text{Stau}}, T_{\text{cyl}}) \\
\text{TM}_{\text{inj}} &= f (V_{\text{inj}}, T_{\text{Runner}}, \text{TM}_{\text{plast}}) \\
\text{t}_{\text{vulc}} &= f (T_{\text{Mold}}, V_{\text{cure}}, \text{TM}_{\text{inj}})
\end{align*}
\]

Consequently the time is a response (and not taken as a factor anymore) because it is dependent from TMold and Tminj
Finding of the Cycle Time in Injection Molding

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Regression equations to describe the injection molding process

\[ t_{vulc} = f (T_{Mold}, V_{cure}, V_{inj}, P_{Stau}, T_{cyl}) \]

In case that the mold temperature \( T_{mold} \) is a constant as well as the compound \( V_{cure} \) vulcanization time depends on four factors.

The difficulty is, to determine the vulcanization time which corresponds to the mold temperature.
Vulcanization of rubber in injection molding happens under pressure of 250 bar – 450 bar

All volatiles are compressed and probably expand if mold opens and pressure is reduced to atmospheric pressure.

The definition of the porous point: Modulus of rubber, which just prevent pores.

Question:
What is the correlation between porous point and physical properties?
In this work to evaluate the cycle time 3 WOCO compounds used:

- NBR 1 – 51D50 (polar)
- NR 1 – 51A45 (slightly polar)
- EPDM 1 – 51M50 (none polar)

All 3 materials are sulphur cured.

The test mold: rectangular cup
1. Trial
- Vulcanization time until none pores observed

2. Trial
- None pores time +20 sec
- None pores time +40 sec
- None pores time +60 sec

Measurement of
- Crosslink density with equilibrium swelling (Flory Rehner)
- Compression set
### Finding of the Cycle Time in Injection Molding

**Introduction**
- Porous Point
- Cycle Time and Porous Point
- Operating Window
- Shortest Vulcanization Time
- Summary

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#### Table: Temperatures, Drucke, Zeiten, and vorlc

<table>
<thead>
<tr>
<th>No</th>
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<th>Temperatur</th>
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Source: Putera Thesis

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Finding of the Cycle Time in Injection Molding

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Source: Putera Thesis
Conclusion from compression set measurement *(in conjunction with the other tests)*:

- NBR1 \( t_{\text{pores}} + 45 \text{ sec} \)
- NR1 \( t_{\text{pores}} + 55 \text{ sec} \)
- EPDM1 \( t_{\text{pores}} + 40 \text{ sec} \)
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force

\[ t_{90} \]

EPDM
NBR
NR
time
Operating window in injection molding:
The “Operating Window” gives a view about process limits due to:

- Machine design
  - Plastification Unit
  - Nozzle length and diameter

- Mold design
  - Runner length, diameter,
  - Gate cross section
  - Length to total flow path

- Processing parameter
  - Plastification parameter setting
  - Mold temperature

- Compound Cure

- Part design
  - Pressure loss due to flow until cavity is filled
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TP Moulding
Operating window: comparison with IM-moulding of rubber compound

TP Moulding
Rubber Molding

Temperature
Injection Pressure

Thermal degradation
Flash
Melting
Short shot
Scorch
Underfill
Lines are showing the injection time at various pressures at different mold temperatures.

- No scorch during filling

The maximum hydraulic pressure (resp. specific pressure) results the shortest possible injection time.
Lines are showing the scorch index at various mold temperatures. The maximum allowed scorch index is defined at 20% prevulcanization.
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Source: Graf, DESMA

Crossing points from previous Curves are now transferred to a Mold Temperature / Time Diagram
Both curves showing the operating window
Finding of the Cycle Time in Injection Molding

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Dotted Line shows in addition the 90% state of cure.

This Line is parallel to scorch index line if activation energy of cure is similar for scorch and vulcanization time.

Source: Graf, DESMA
In case of compound temperature variation the line will shift.

- For increase of compound temperature Line will shift to shorter times.
- The operating window will also move to shorter times.
Finding of the Cycle Time in Injection Molding

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Source: Graf, DESMA

Stages of Processing.
- Shortest possible cycle time at maximum mold and compound temperature
- Best cycle time in an operating window, which fits demands of SPC due to machine repeatability
- Safe cycle time, which is recommended, if machine repeatability is questionable.

Graph showing the relationship between mold temperature (Grad C) and time (sec) and (min).

- Tmpd 105C
- 95C
- 190, 180, 170
Finding of the Cycle Time in Injection Molding

Statistic Experimental Design (DoE) procedure

- Factor 1 = $T_M$  
  Mass Temperature
- Factor 2 = $T_{mold}$  
  Mold Temperature
- Factor 3 = $v_{inf}$  
  Injection Speed

(little influence EPDM, but big with NBR)

3 Factor Design,
- but detect the porous point,
- add the appropriate time or increase stepwise
- $t_{porous} + t_{addition}$

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<th>Units</th>
<th>Low</th>
<th>High</th>
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<td>115</td>
</tr>
<tr>
<td>T mold</td>
<td>°C</td>
<td>160</td>
<td>200</td>
</tr>
<tr>
<td>V inj</td>
<td>mm/sec</td>
<td>15</td>
<td>25</td>
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</tbody>
</table>
Vulcanization to meet physical properties

Porous point of rubbers is a good orientation, if the correlation between

- Crosslink density and physical properties are known
- Crosslink density can be measured with equilibrium swelling test precisely
- Physical properties with the standard methods in rubber industry

As a rule of thumb the following can be concluded:

- NR + 55 sec (for 10 mm thickness)
- EPDM / NBR + 40 sec for 10 mm thickness

This is in line with experience
Statistic Experimental Design (DoE) procedure

- Factor 1 = $T_M$  
  Mass Temperature
- Factor 2 = $T_{mold}$  
  Mold Temperature
- Factor 3 = $v_{inf}$  
  Injection Speed  
  (little influence EPDM, but big with NBR)
- Factor 4 = $t_{vulc}$  
  Vulcanization time, but dependent from factor 2

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<tr>
<td>$t_{vulc}$</td>
<td>sec</td>
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</table>
Post cure operation

- For parts with even higher thickness post cure operation is saving machine time
  - Vulcanization time must succeed the porous point!
  - It is recommended for compounds based on NR with an early porous point and high wall thickness
  - Post cure is a pressure less vulcanization, but there is no negative effect on physical properties because the starting modulus is high enough to prevent pores.
Conclusion

- Optimum cycle time depends on temperatures in injection molding:
  - Compound cavity entrance temperature
  - Mold temperature
- None porous point depends on compound modulus at a time, when the vulcanization has progressed
  - Porous point is different for each compound / polymer
- With the knowledge about the correlation between porous point an physical properties a optimum cycle time can be determined.
- Introducing the vulcanization time as a factor, the shortest vulcanization time can be determined with 9 experiments
Finding of the Cycle Time in Injection Molding

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Operating Window
Vulcanization to performance
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Thank you for attention and your comments