International Conference on Rubber Molding

# RUBBER MOLDING ASIA 2014

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## Determination of the Shortest Possible Vulcanization Time in Rubber Injection Molding

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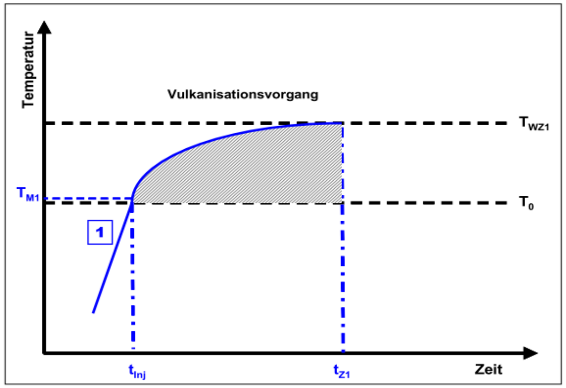
## Content of this presentation

- Introduction
- Operating window
- Porous point of different Rubbers
- Vulcanization time and physical properties
- Experiments guiding to shortest molding time
- Summary

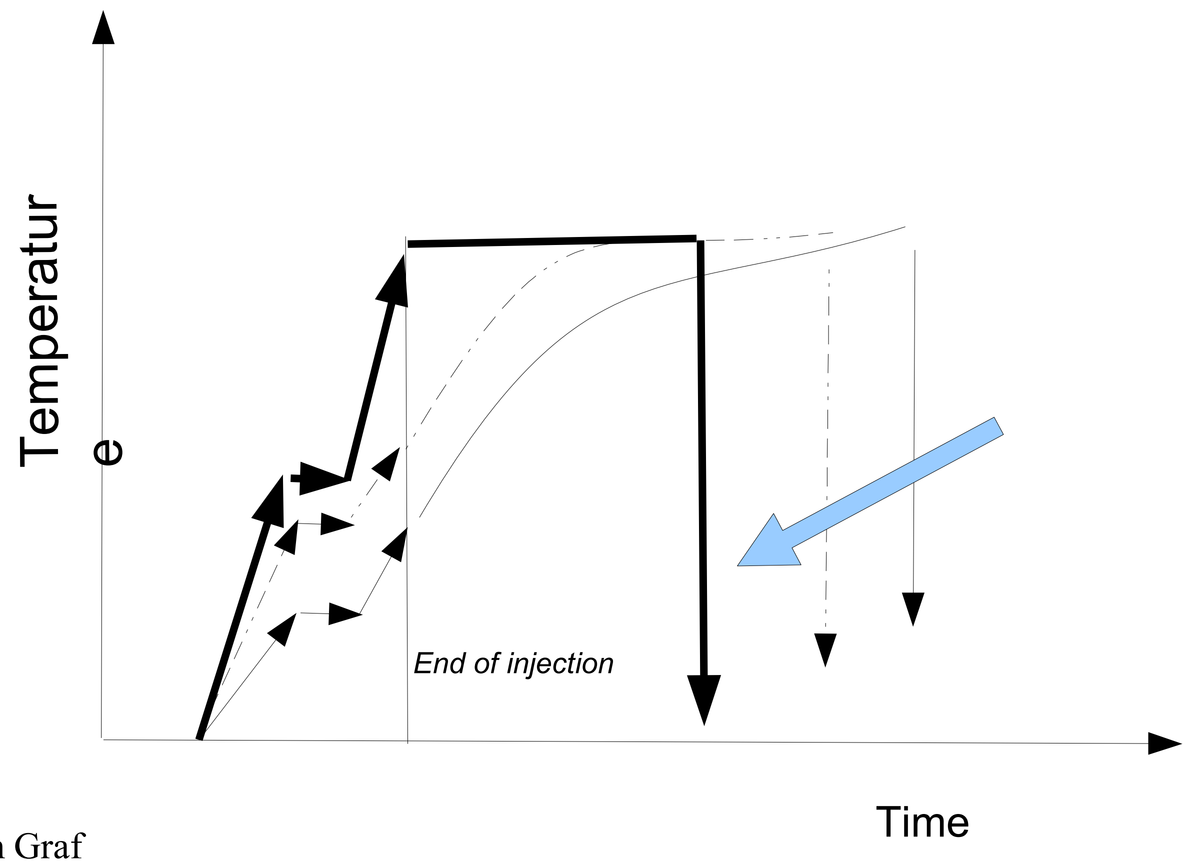
- **The vulcanization time is Dependent from:**
  - **Mold temperature**
  - **Compound cavity entrance temperature**
  - **Thickness of the part due to restricted temperature conductivity**

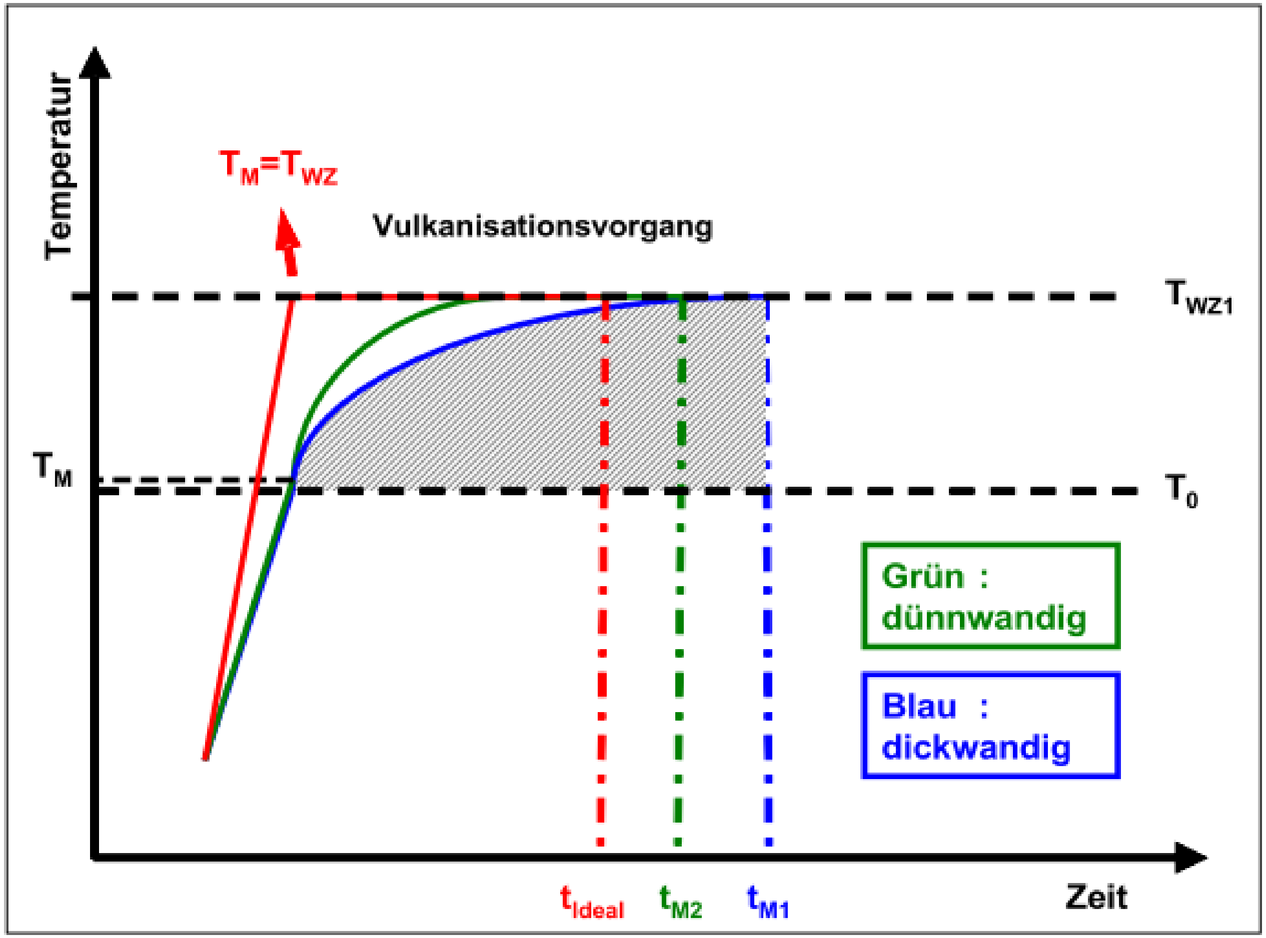
- **Determined by trials**
  - **No simulation tool, because of lack of precision**
  - **Physical properties must be evaluated**



# Heat rise in injection unit

- **Reason for heat rise – shortening of the vulcanization time**




Temperatur
$T_M=T_{WZ}$
Vulkanisationsvorgang
$T_{WZ1}$
$T_M$
$T_0$
Grün : dünnwandig
Blau : dickwandig
$t_{Ideal}$
$t_{M2}$
$t_{M1}$
Zeit

## Regression equations to describe the injection molding process

$$TM_{plast} = f\,(V_{Sc},\ P_{Stau},\ T_{cyl})$$

$$TM_{inj} = f\,(V_{inj},\ T_{Runner},\ TM_{plast})$$

$$t_{vulc} = f\,(T_{Mold},\ V_{cure},\ TM_{inj})$$

***Consequently the time is a response (and not taken as a factor anymore) because it is dependent from TMold and TMinj***

# Regression equations to describe the injection molding process

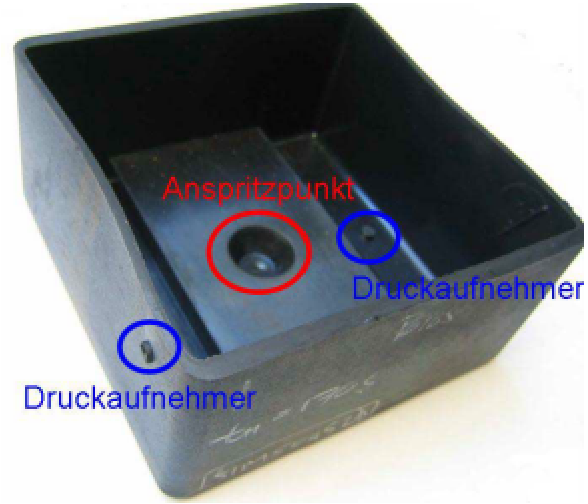
$$t_{vulc} = f\,(T_{Mold},\ V_{cure},\ V_{inj},\ V_{Sc},\ P_{Stau},\ T_{cyl})$$

***In case that the $T_{mold}$ is a constant as well as the compound ($V_{cure}$) vulcanization time depends on four [ 4 !] 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.**



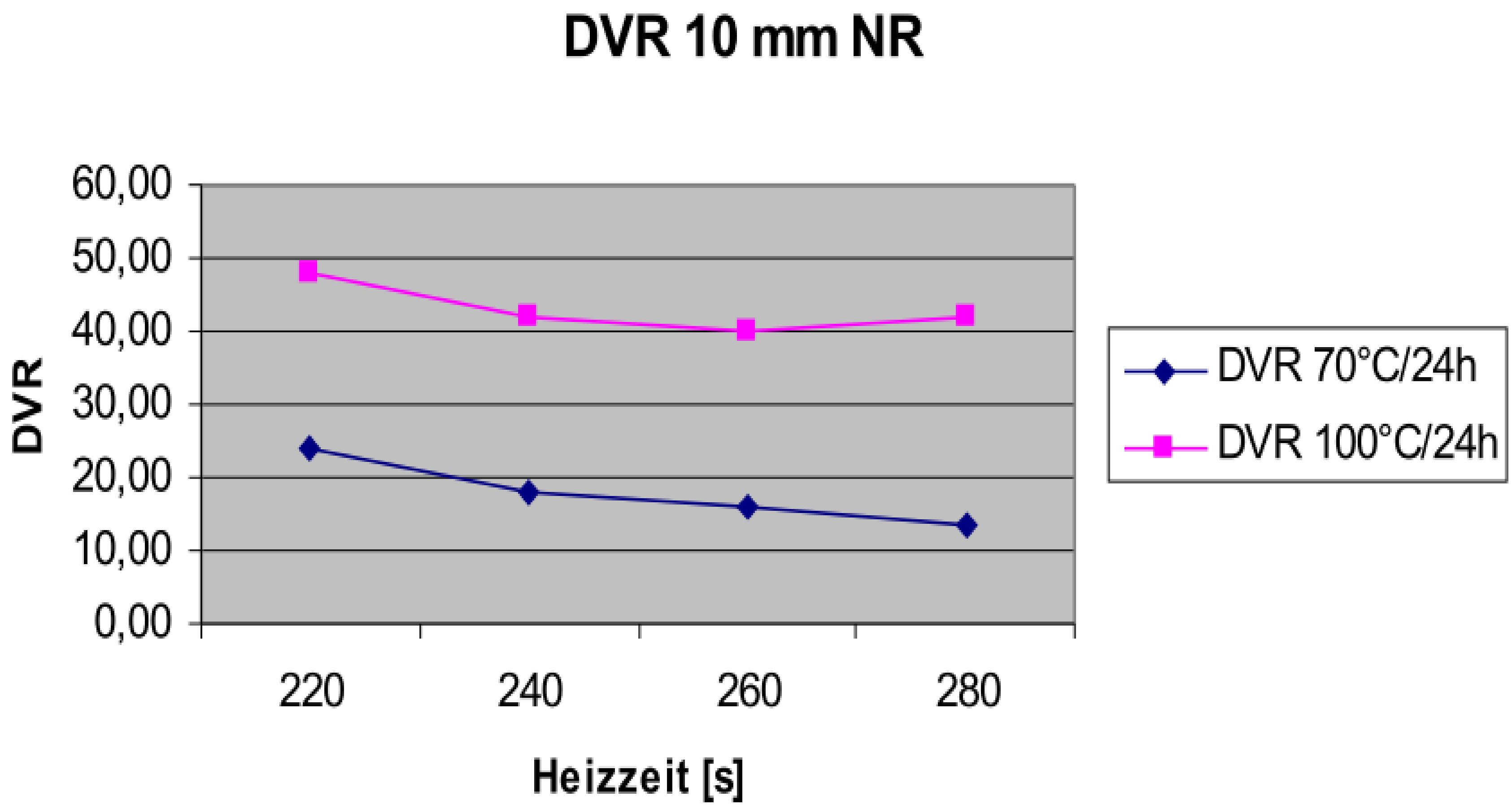

- **The test mold: rectangular cup**

- **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**

Source: Putera Thesis

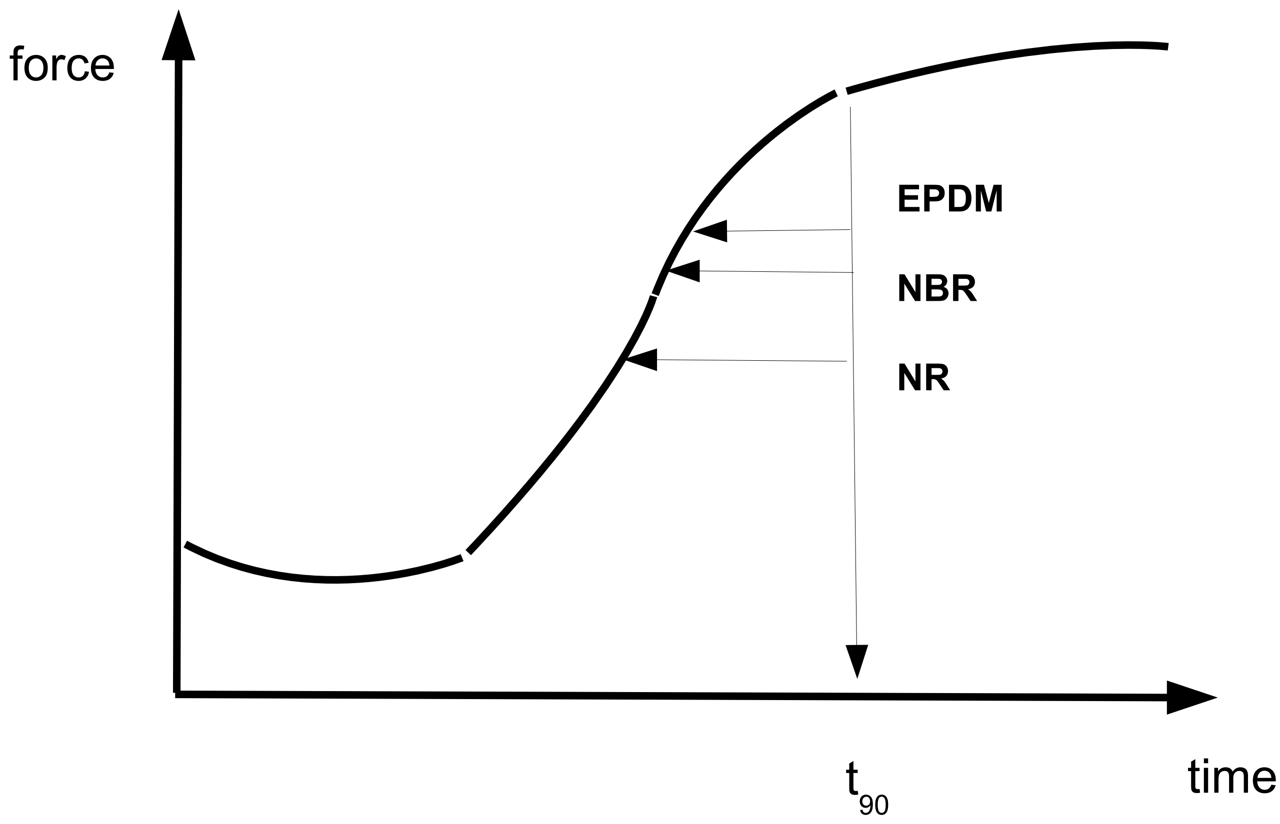
| | | | Temperaturen | | | | | Drucke | | Zeiten | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| No | Material | | Mittelplatte Oberfläche | Kernoberfläche | Zylinder | Masse eingespritzt | Masse-Zylinder | Einspritzen (max) | Nachdruck | Einspritzen+Nachdruck | $t_{Vulc}$ |
| | | | [°C] | [°C] | [°C] | [°C] | [°C] | [bar] | [bar] | [s] | [s] |
| 1 | NBR | t porös | 179 | 179 | 95 | 114 | 19 | 84 | 25 | 10,2 | 140 |
| 2 | NBR | t porös + 20 s | 179 | 179 | 95 | 114 | 19 | 84 | 25 | 10,2 | 160 |
| 3 | NBR | t porös + 40 s | 179 | 179 | 95 | 114 | 19 | 84 | 25 | 10,2 | 180 |
| 4 | NBR | t porös + 60 s | 179 | 179 | 95 | 114 | 19 | 84 | 25 | 10,2 | 200 |
| 5 | NR | $t_{porös}$ | 167 | 165 | 85 | 98 | 13 | 61 | 20 | 10,1 | 220 |
| 6 | NR | $t_{porös}$ + 20 s | 167 | 165 | 85 | 98 | 13 | 61 | 20 | 10,1 | 240 |
| 7 | NR | $t_{porös}$ + 40 s | 167 | 165 | 85 | 98 | 13 | 61 | 20 | 10,1 | 260 |
| 8 | NR | $t_{porös}$ + 60 s | 167 | 165 | 85 | 98 | 13 | 61 | 20 | 10,1 | 280 |
| 9 | EPDM 1 | $t_{porös}$ | 177 | 178 | 95 | 105 | 10 | 51 | 15 | 10,2 | 170 |
| 10 | EPDM 1 | $t_{porös}$ + 20 s | 177 | 178 | 95 | 105 | 10 | 51 | 15 | 10,2 | 190 |
| 11 | EPDM 1 | $t_{porös}$ + 40 s | 177 | 178 | 95 | 105 | 10 | 51 | 15 | 10,2 | 210 |
| 12 | EPDM 1 | $t_{porös}$ + 60 s | 177 | 178 | 95 | 105 | 10 | 51 | 15 | 10,2 | 230 |

Source: Putera Thesis



## Conclusion from compression set measurement *(in conjunction with the other tests)*:

- **NBR1** $t_{pores}$ **+ 45 sec**
- **NR1** $t_{pores}$ **+ 55 sec**
- **EPDM1** $t_{pores}$ **+ 40 sec**

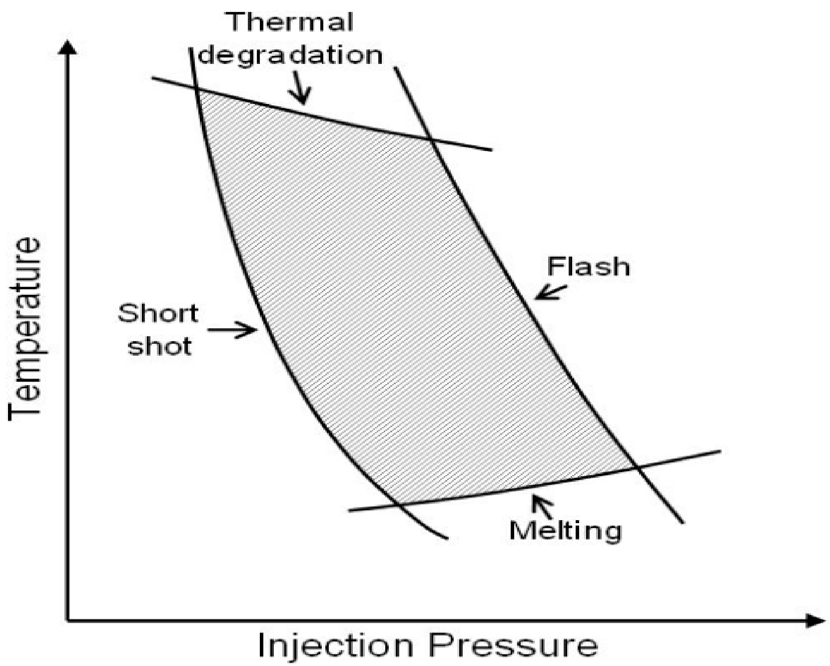

force
EPDM
NBR
NR
t90
time

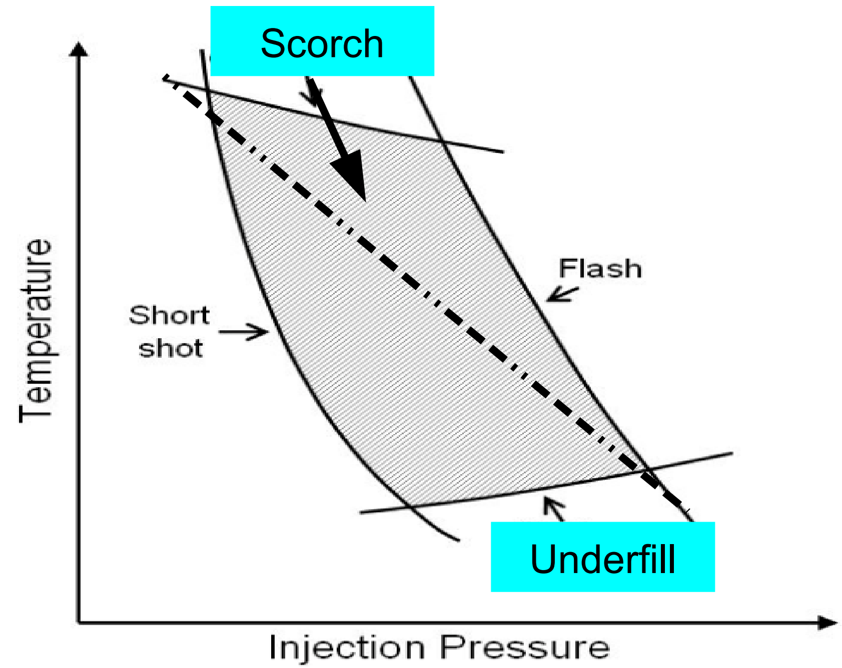
**Operating window in injection molding:**
**The "Operating Window" gives a view about process limits due to:**

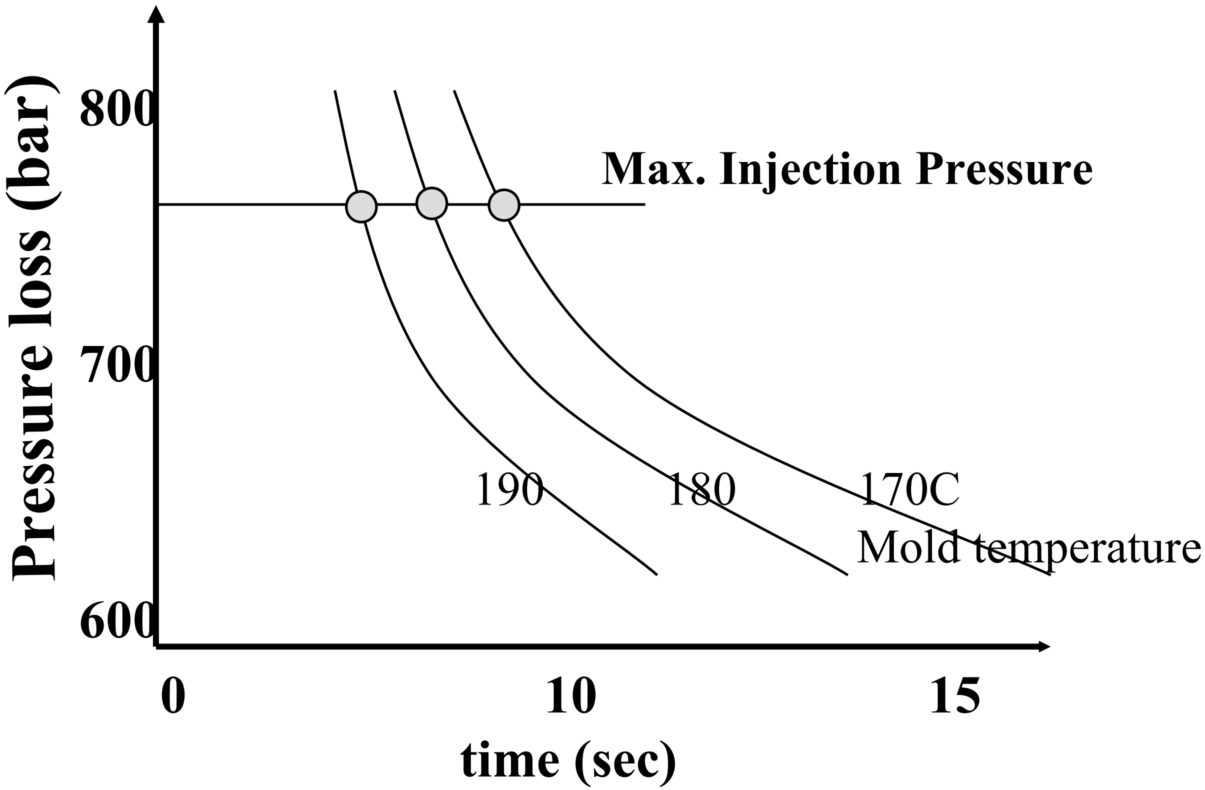
- **Machine design**
  - **Plastification Unit**
  - **Nozzle length and diameter**
- **Mold design**
  - **Runner length, diameter,**
  - **Gate cross section**
  - **Length to total flow path**
- **Processing parameter**
  - **Plastification parameter setting**
  - **Mold temperature**
- **Compound Cure**
- **Part design**
  - **Pressure loss due to flow until cavity is filled**

## TP Moulding / Rubber Molding

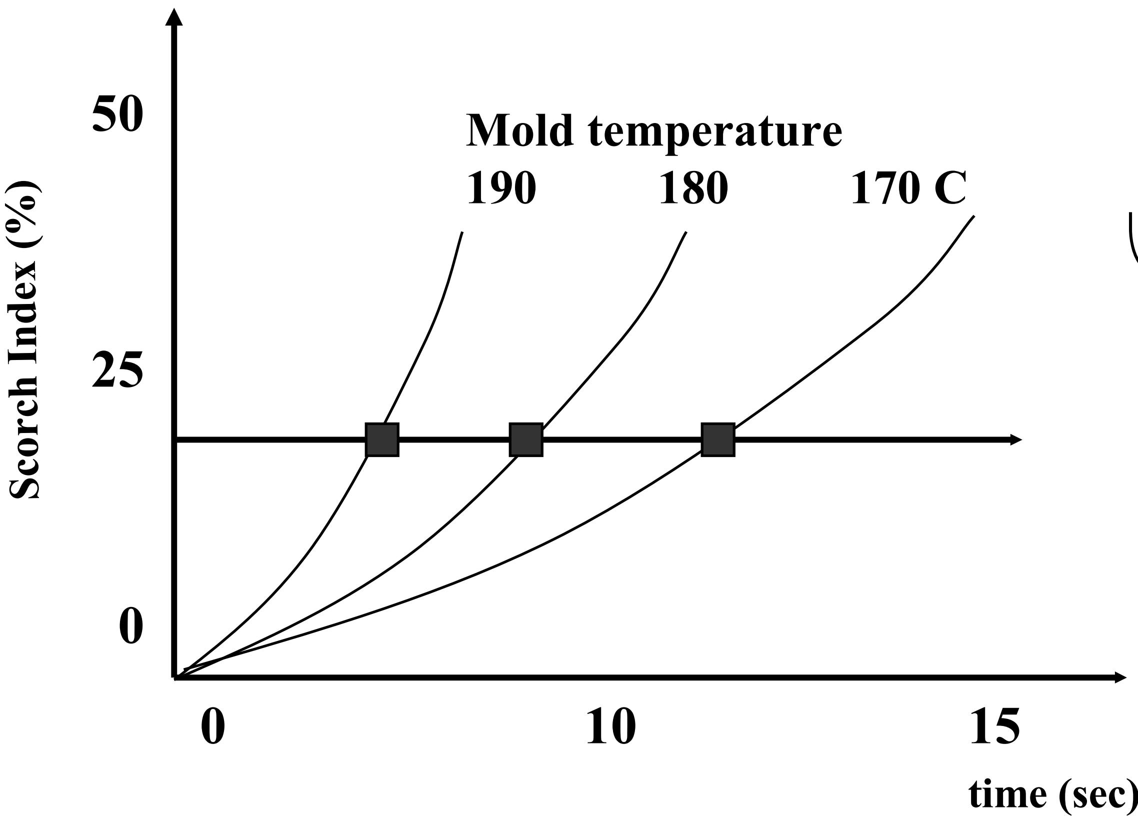
Operating window: comparison with IM-moulding of rubber compound



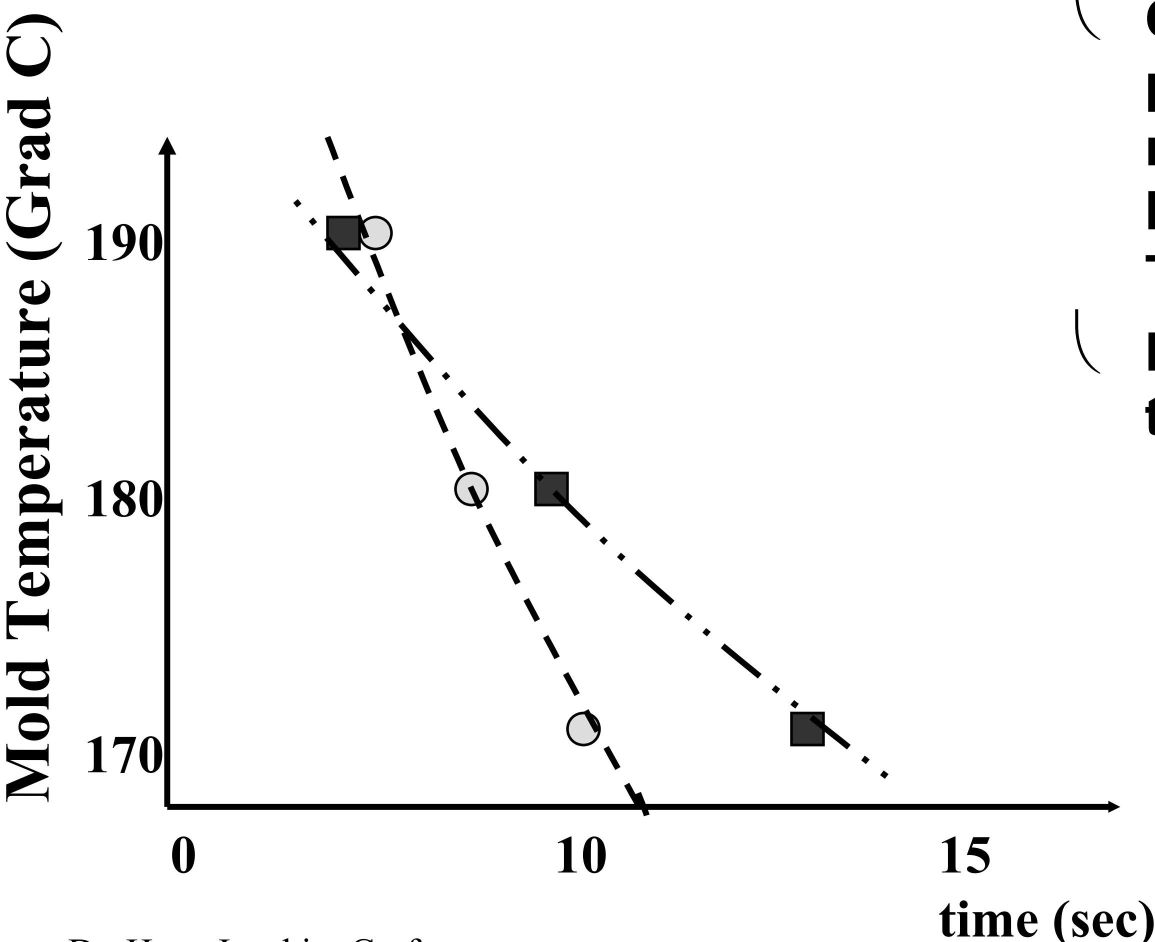



- **Lines are showing the injection time at various pressures at different mold temperatures.**
  - **No scorch during filling**
- **The maximum hydraulic pressure (resp. specific pressure) results the shortest possible injection time.**



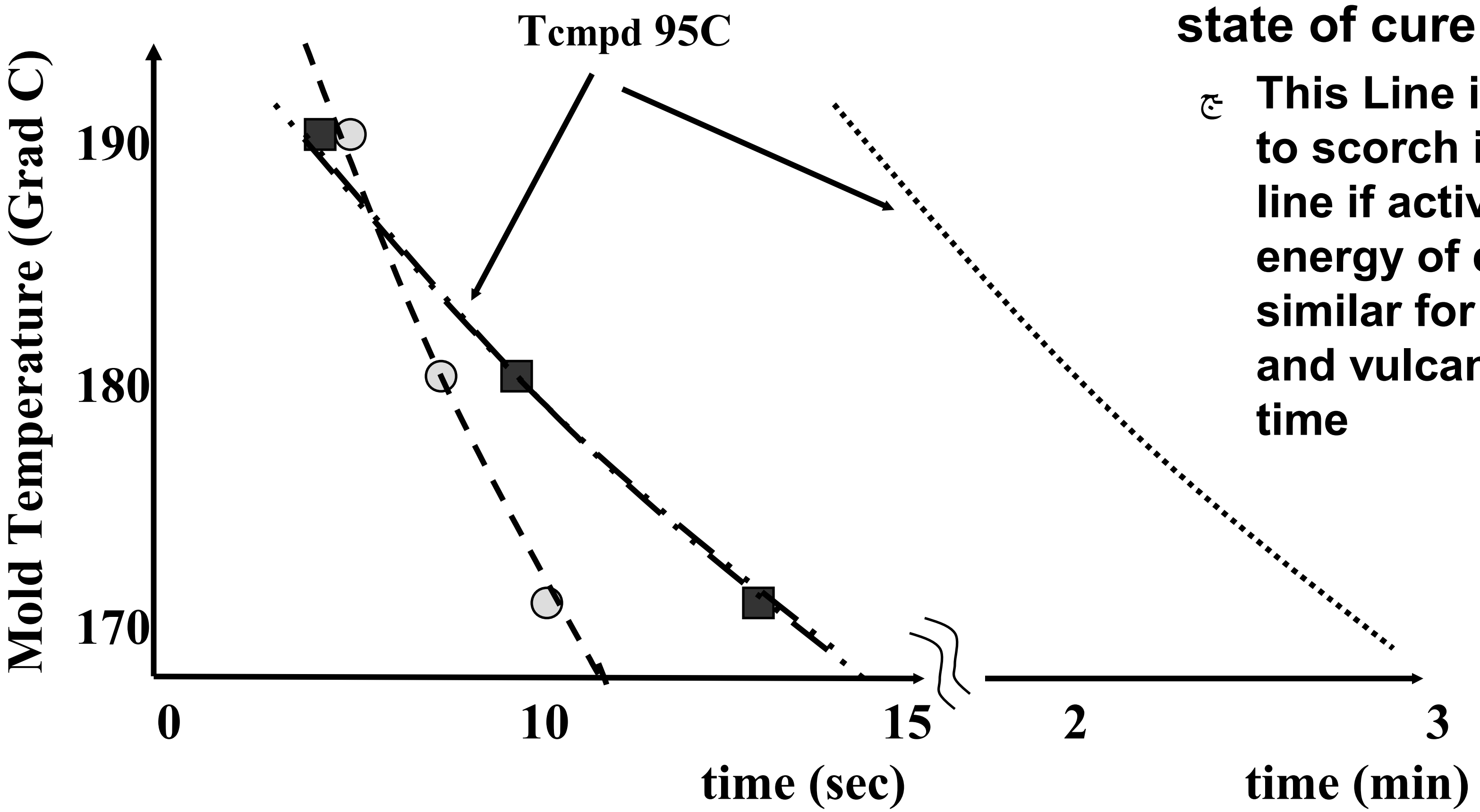

- Lines are showing the scorch index at various mold temperatures.
- The maximum allowed scorch index is defined at 20% prevulcanization.

Source: Graf, DESMA



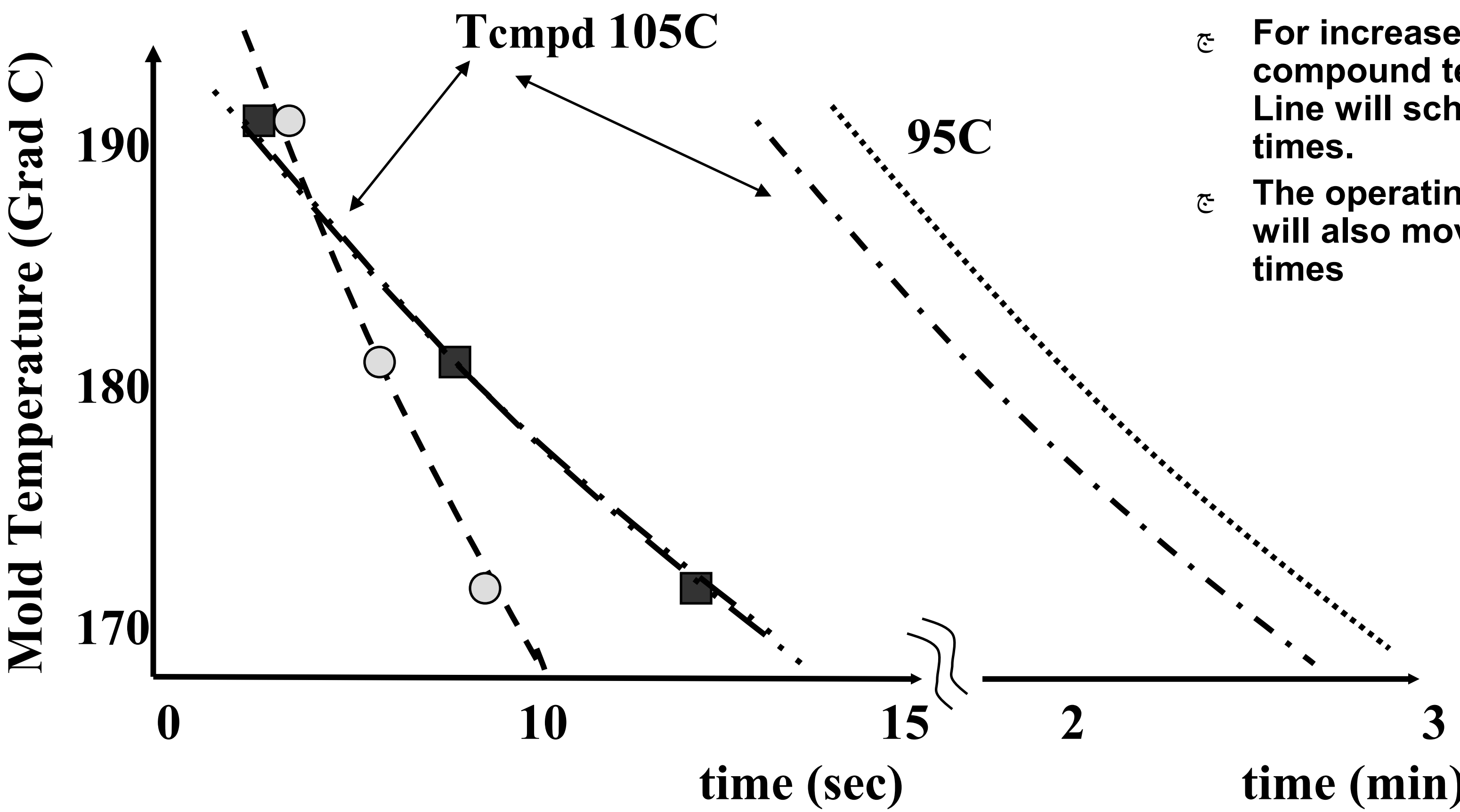

- Crossing points from previous Curves are now transferred to a Mold Temperature / Time Diagram
- Both curves showing the operating window

Source: Graf, DESMA




- **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**

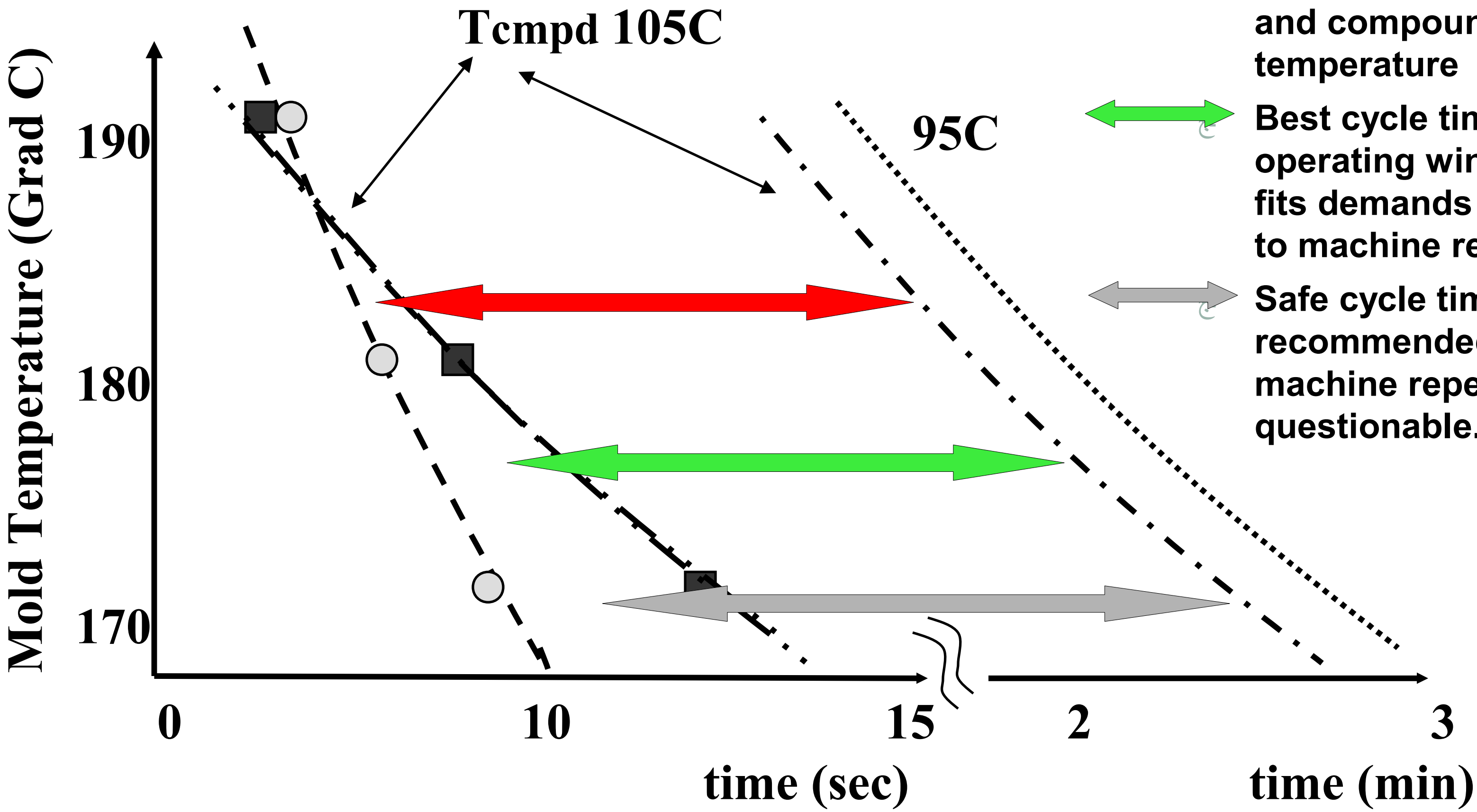
Source: Graf, DESMA




**In case of compound temperature variation the line will shift.**

- **For increase of compound temperature Line will schift to shorter times.**
- **The operating window will also move to shorter times**

Source: Graf, DESMA




## Stages of Processing.

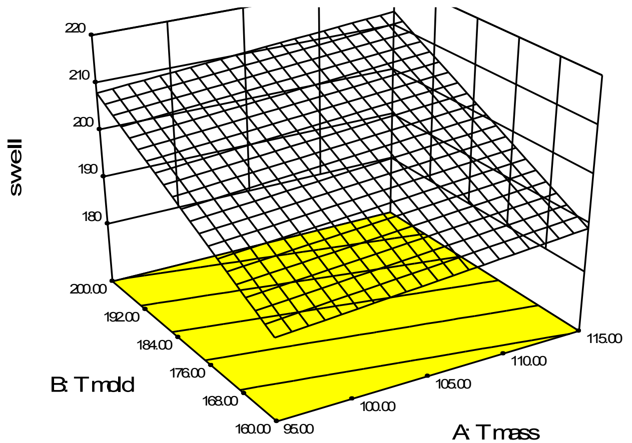
- Shortest possible cycle time at maximum mold and compound temperature
- Best cycle time in an operating window, which fits demands of SPC due to machine repeatability
- Safe cycle time, which is recommended, if machine repeatability is questionable.

# 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**
  - **mold $t_{porous}$ + $t_{addition}$**

Actual Factors
C: V inj = 20.00
D: t vulc = 75.00

| Name | Units | Low | High |
|---|---|---|---|
| T mass | °C | 95 | 115 |
| T mold | °C | 160 | 200 |
| V inj | mm/sec | 15 | 25 |



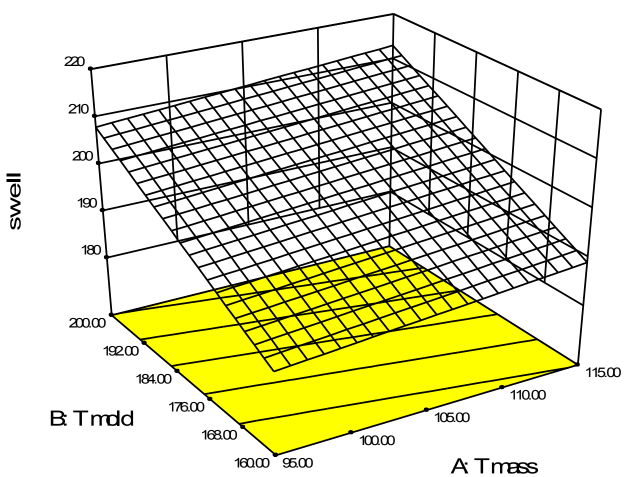
## Vulcanization to meet physical properties

- Porous point of rubbers is a good orientation, if the correlation between
  - Crosslink density and physical properties are known
  - Croslink 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 conluded:
  - 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**

| Name | Units | Low | High |
|---|---|---|---|
| T mass | °C | 95 | 115 |
| T mold | °C | 160 | 200 |
| V inj | mm/sec | 15 | 25 |
| t vulc | sec | 30 | 120 |



## 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**

**Thank you for attention and your comments**