



**ThermoFisher**  
S C I E N T I F I C

## Rotational Experiment

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# The basics of rheology and rotational rheometry

## Content

- Definition of basic rheological parameters
  - Viscosity and elasticity
  - Deformation, shear stress and shear rate
- Parameters and factors affecting the viscosity
  - Temperature
  - Shear rate (Newtonian and non-Newtonian behavior)
  - Time and shear time (thixotropic behavior)

# Definition of basic rheological parameters

## *Rheology and viscosity*

### Rheology

Rheology is the study of deformation and flow of matter

### Viscosity

Viscosity is a measure for the resistance of a material against deformation and flow

# Definition of basic rheological parameters

## *Viscosity and elasticity*

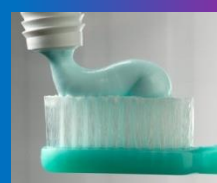
### Viscous behavior

irreversible flow:  
all applied energy dissipates  
completely

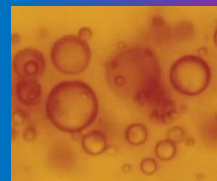
Dashpot:



Investigated mainly with tests  
performed in rotation



### Complex Systems



### Elastic behavior

reversible deformation:  
all applied energy is stored  
in the system

Spring:



Investigated mainly with tests  
performed in oscillation

# Definition of basic rheological parameters

## Calculation of the dynamic viscosity

Viscosity cannot be measured directly!

$$\text{Viscosity } \eta = \frac{\text{Shear stress } \tau}{\text{Shear rate } \dot{\gamma}}$$

### Units

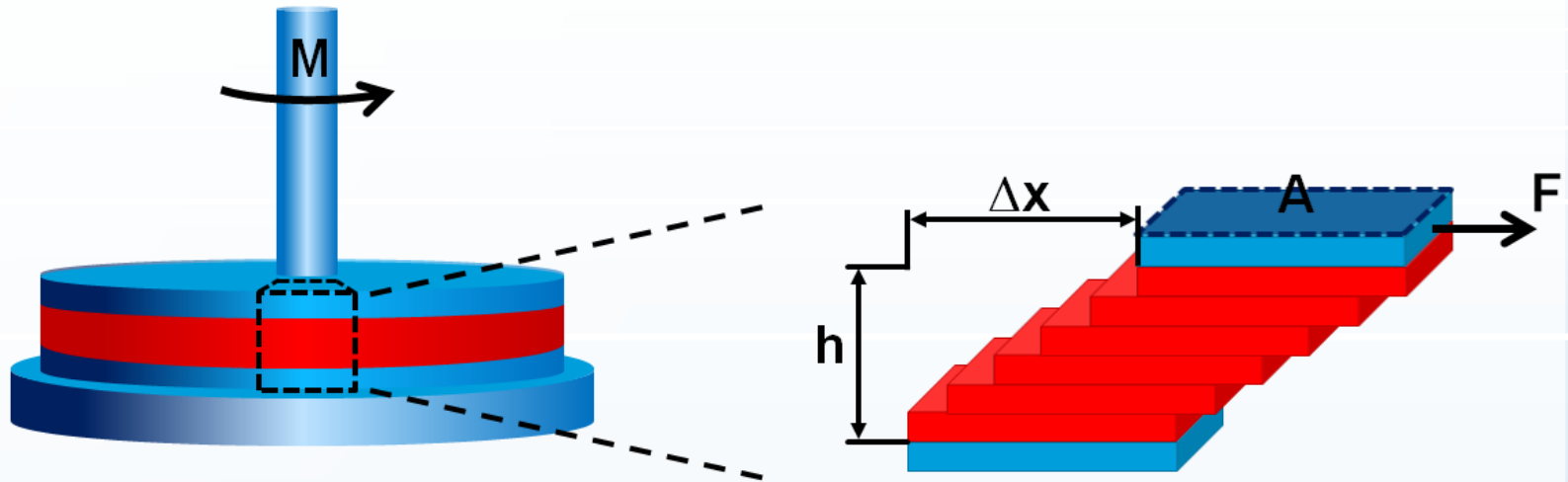
Shear stress  $\tau$ : Pascal (Pa)

Shear rate  $\dot{\gamma}$ : 1/s

Viscosity  $\eta$ : 1 Pa·s = 1000 mPa·s = 1000 centi Poise (cP)

# Definition of basic rheological parameters

## Calculation of the dynamic viscosity



Shear stress:

$$\tau = \frac{\text{Force } F}{\text{Area } A}$$

Unit: Pascal (Pa)

Deformation:

$$\gamma = \frac{\text{Displacement } \Delta x}{\text{Gap } h}$$

Unit: - or %

Shear rate:

$$\dot{\gamma} = \frac{\text{Change in deformation } d\gamma}{\text{Time } t}$$

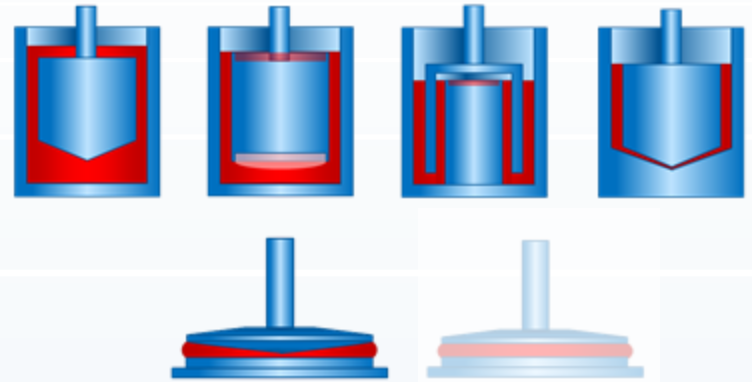
Unit: 1/s

# Definition of basic rheological parameters

## *Experimental determination of the viscosity*

### Absolute Measurement

The geometry factors  $A$  and  $M$  can be calculated for the measuring geometry



### Relative Measurements

The geometry factors  $A$  and  $M$  can not (entirely) be calculated for the measuring geometry



# Parameters and factors affecting viscosity

*Viscosity is not a constant term*



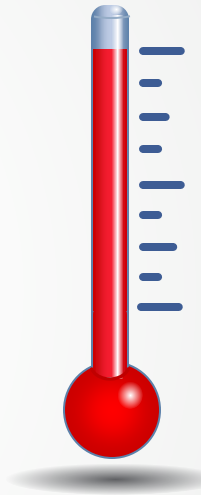
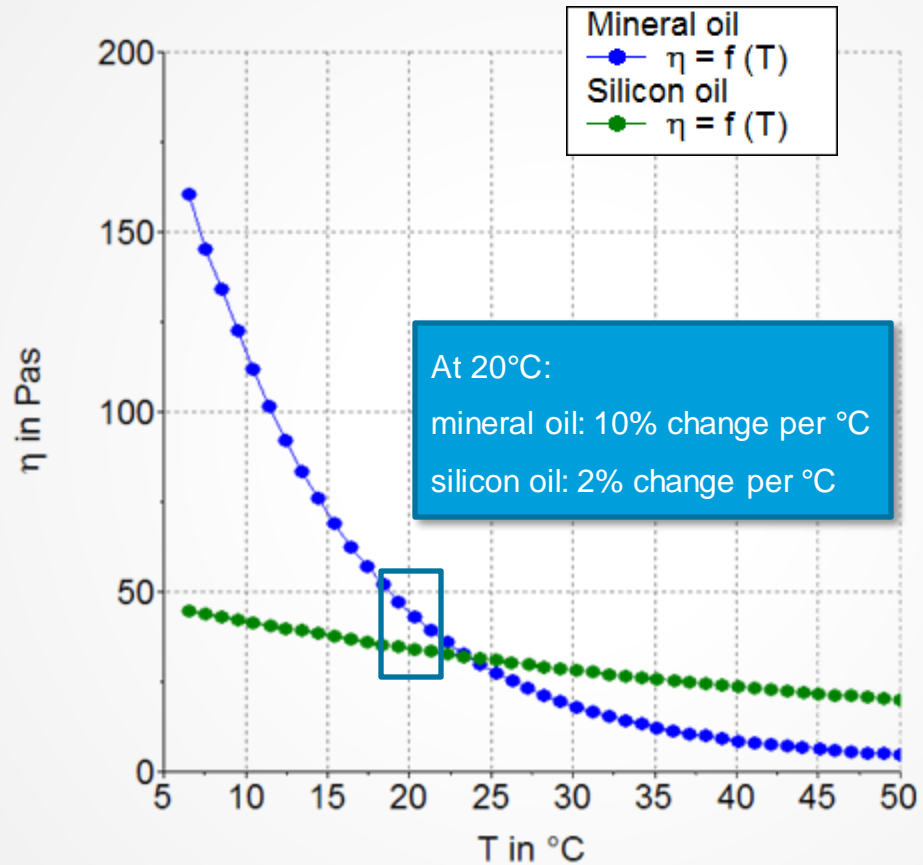


# Parameters and factors affecting viscosity

## Temperature depending flow behavior

Temperature depending viscosity of two standard fluids

- The higher the absolute value of the viscosity, the higher the temperature dependency
- Different fluids show different temperature dependency



# Parameters and factors affecting viscosity

*Viscosity is not a constant term*



# Parameters and factors affecting viscosity

## *Shear rate depending flow behavior*

- Fluids whose viscosity is independent of the applied shear rate are called **Newtonian fluids**
- Most complex fluids as well as semi solid materials usually show a **non-Newtonian behavior**
- There are several different types of non-Newtonian behavior

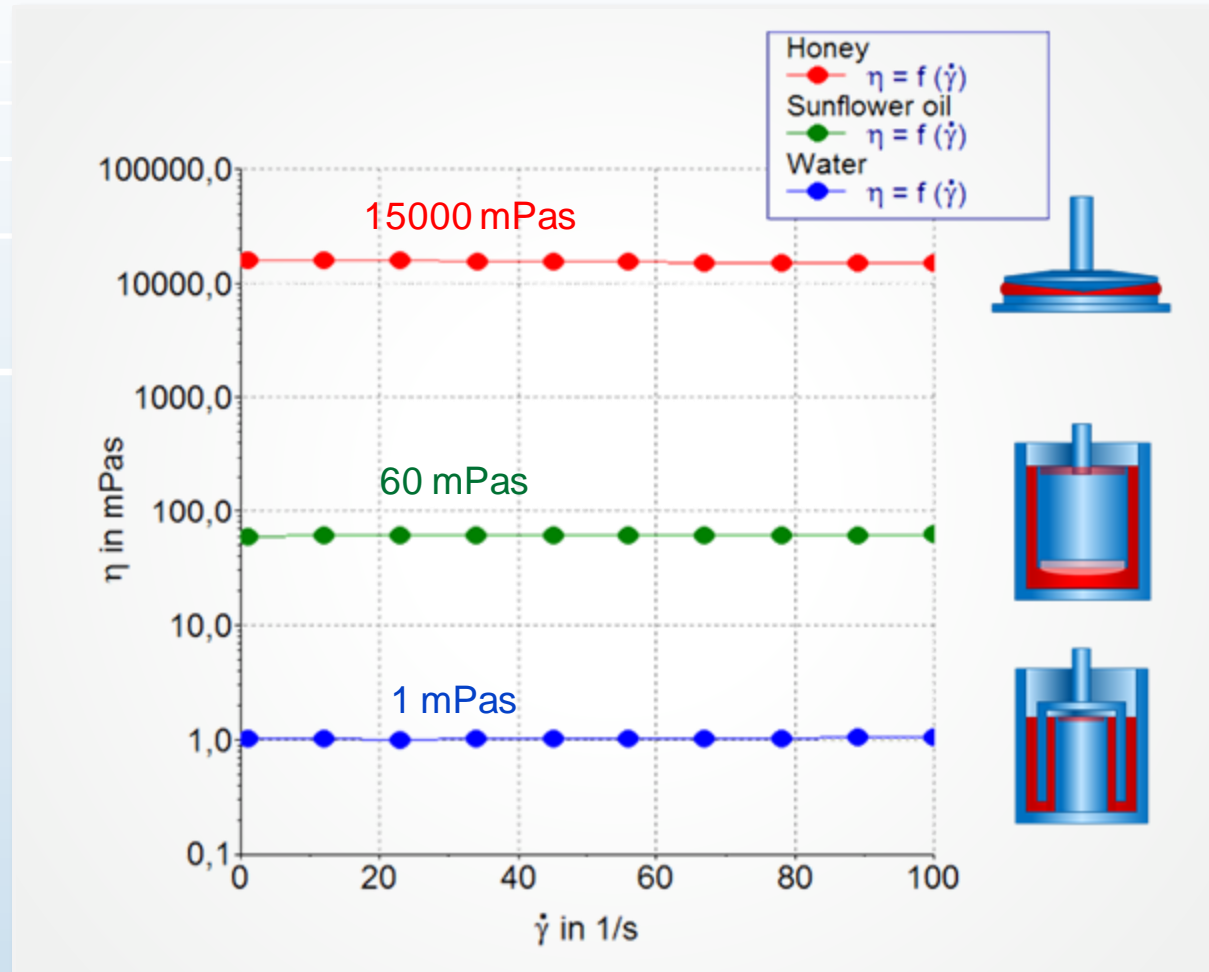
# Parameters and factors affecting viscosity

## Shear rate depending flow behavior

Newtonian flow behavior

$$\eta \neq f(\dot{\gamma})$$

- The viscosity is independent of the applied shear rate
- Only 'simple' fluids show a completely Newtonian behavior
- More complex fluids have a shear rate depending viscosity



# Parameters and factors affecting viscosity

## *Shear rate depending flow behavior*

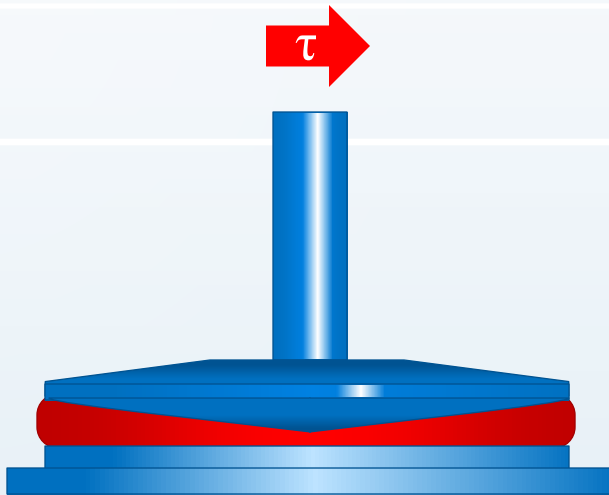
Application	Shear rate in $s^{-1}$	
Sedimentation	$10^{-6} - 10^{-4}$	Storage, shelf life and post application <b>Slow Processes</b>
Phase separation	$10^{-6} - 10^{-4}$	
Leveling, running	$10^{-1} - 10^1$	
Extrusion	$10^0 - 10^2$	Processing and application <b>Fast Processes</b>
Dip coating	$10^1 - 10^2$	
Chewing	$10^1 - 10^2$	
Pumping, stirring	$10^1 - 10^3$	
Brushing	$10^1 - 10^4$	
Spraying	$10^3 - 10^4$	

# Parameters and factors affecting viscosity

## Shear rate depending flow behavior

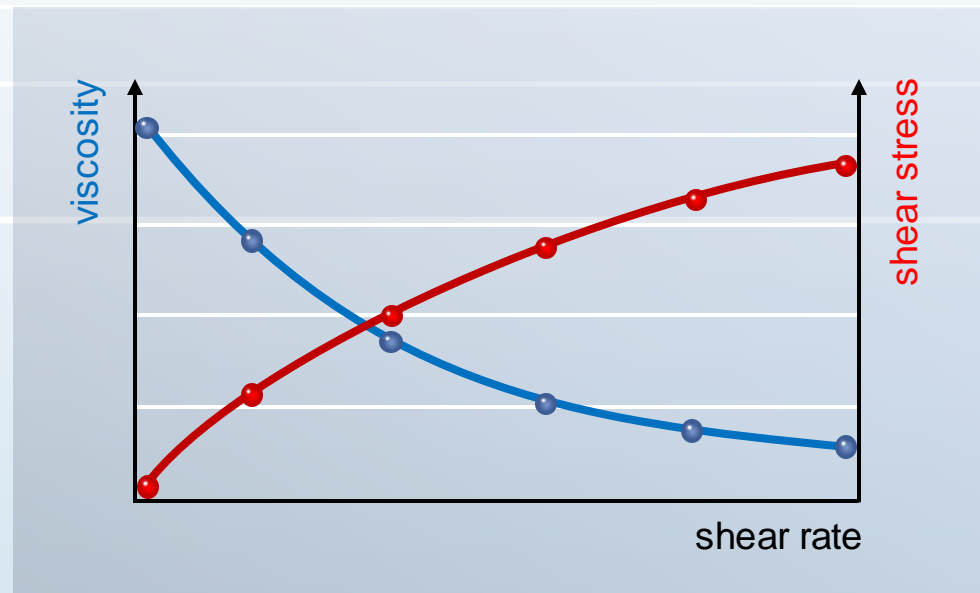
Non-Newtonian flow behavior

$$\eta = f(\dot{\gamma})$$



- Most complex fluids like solutions, emulsions or suspensions

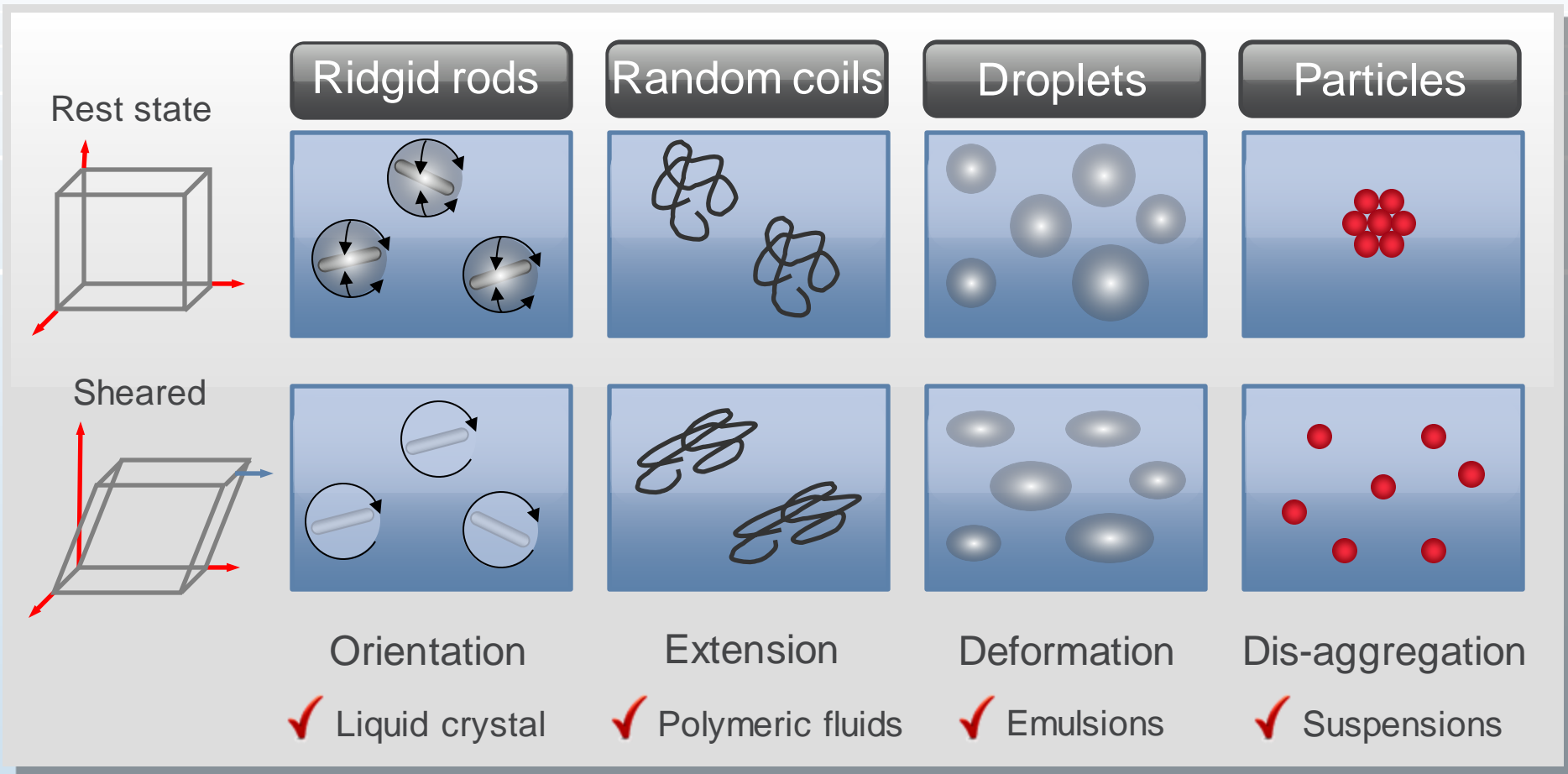
Shear thinning



The viscosity decreases with increasing shear rate

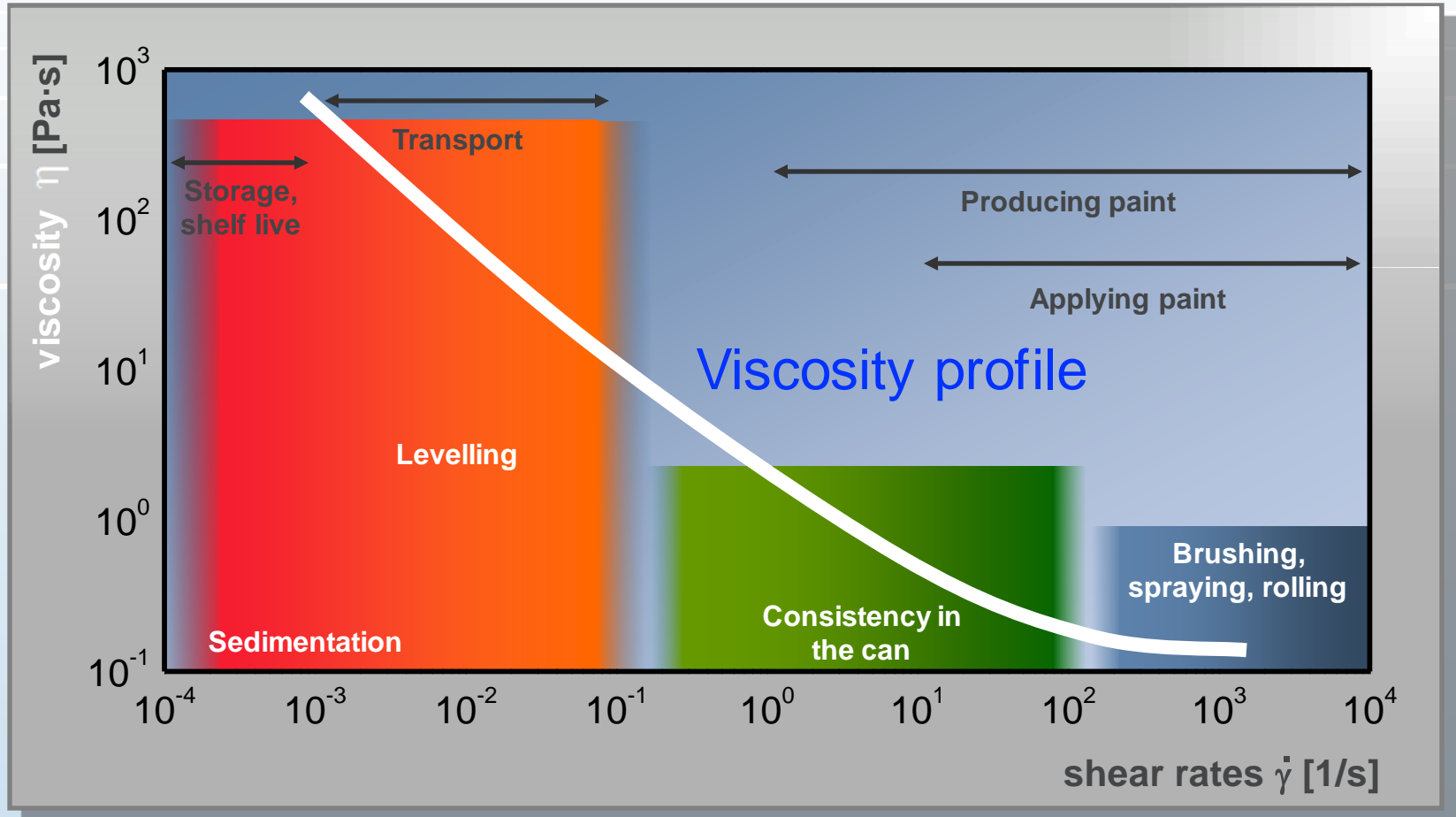
# Newtonian and Non-Newtonian flow behavior

## Shear thinning flow behavior



# Newtonian and Non-Newtonian Flow (viscosity) curve

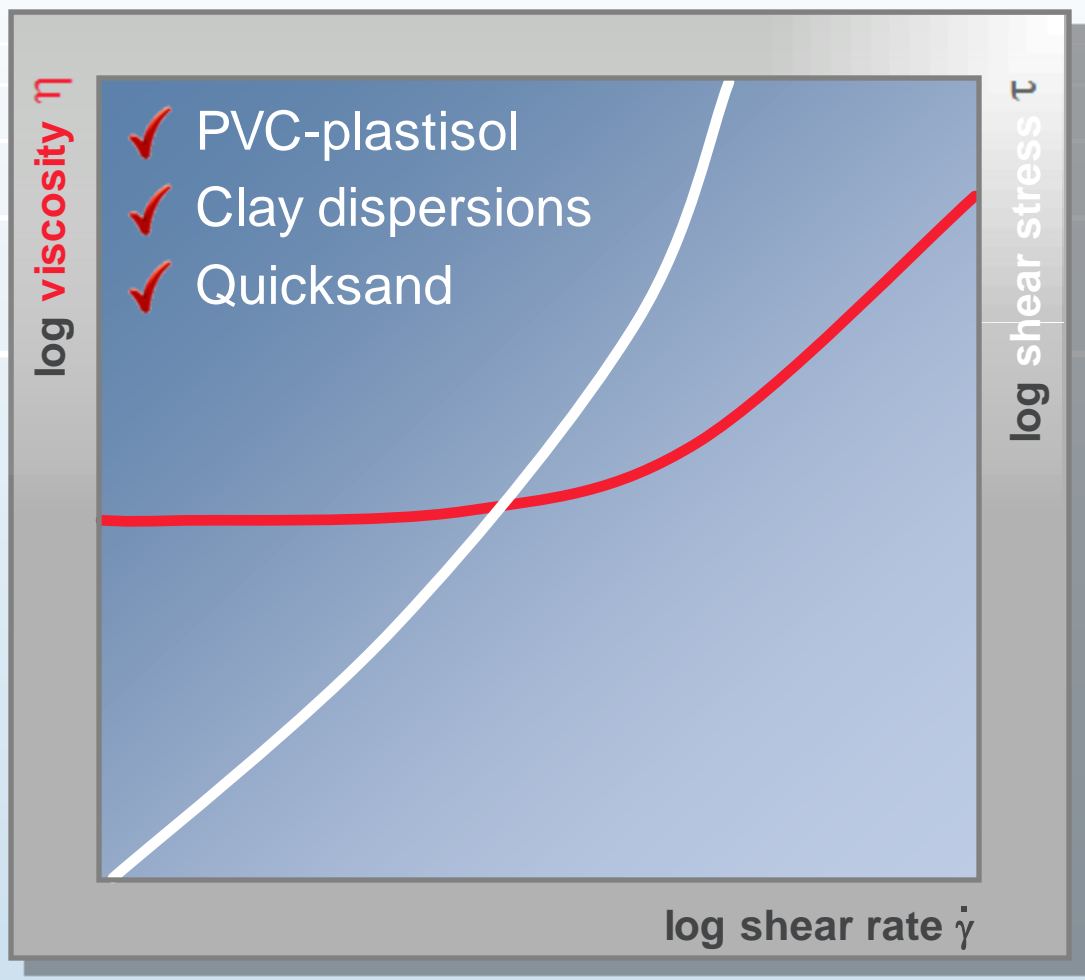
## Shear rates for different paint applications





# Newtonian and Non-Newtonian flow behavior

## Dilatant flow behavior



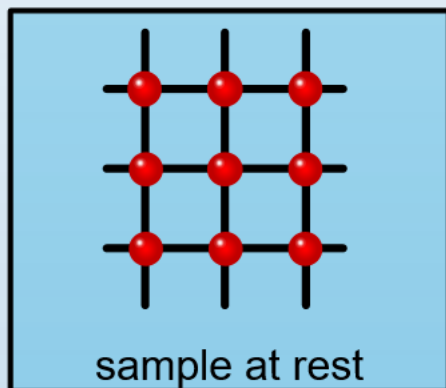
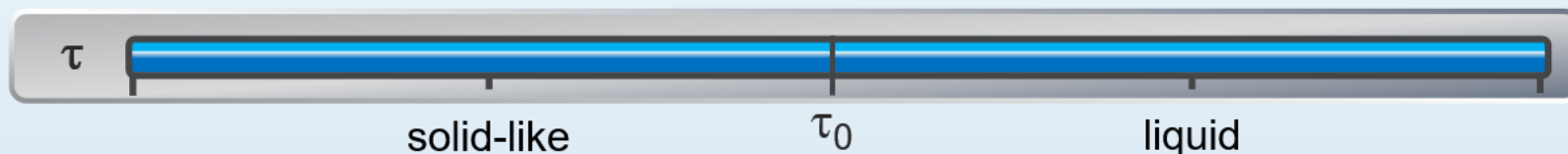
# Parameters and factors affecting viscosity

## Shear rate depending flow behavior

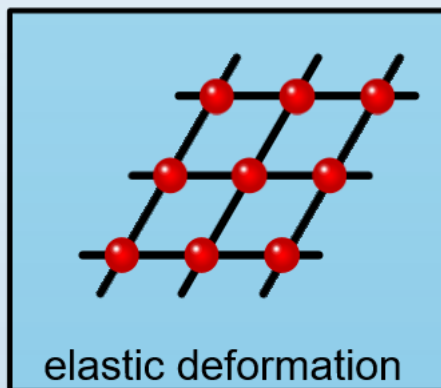
### Yield stress

The yield stress  $\tau_0$  is the minimum shear stress  $\tau$  required, to

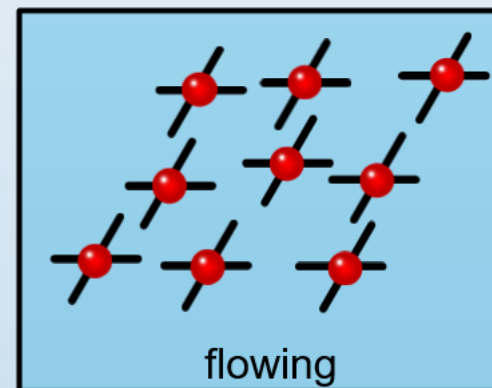
- overcome elastic behavior and
- obtain stationary flow behavior



$$\tau < \tau_0$$

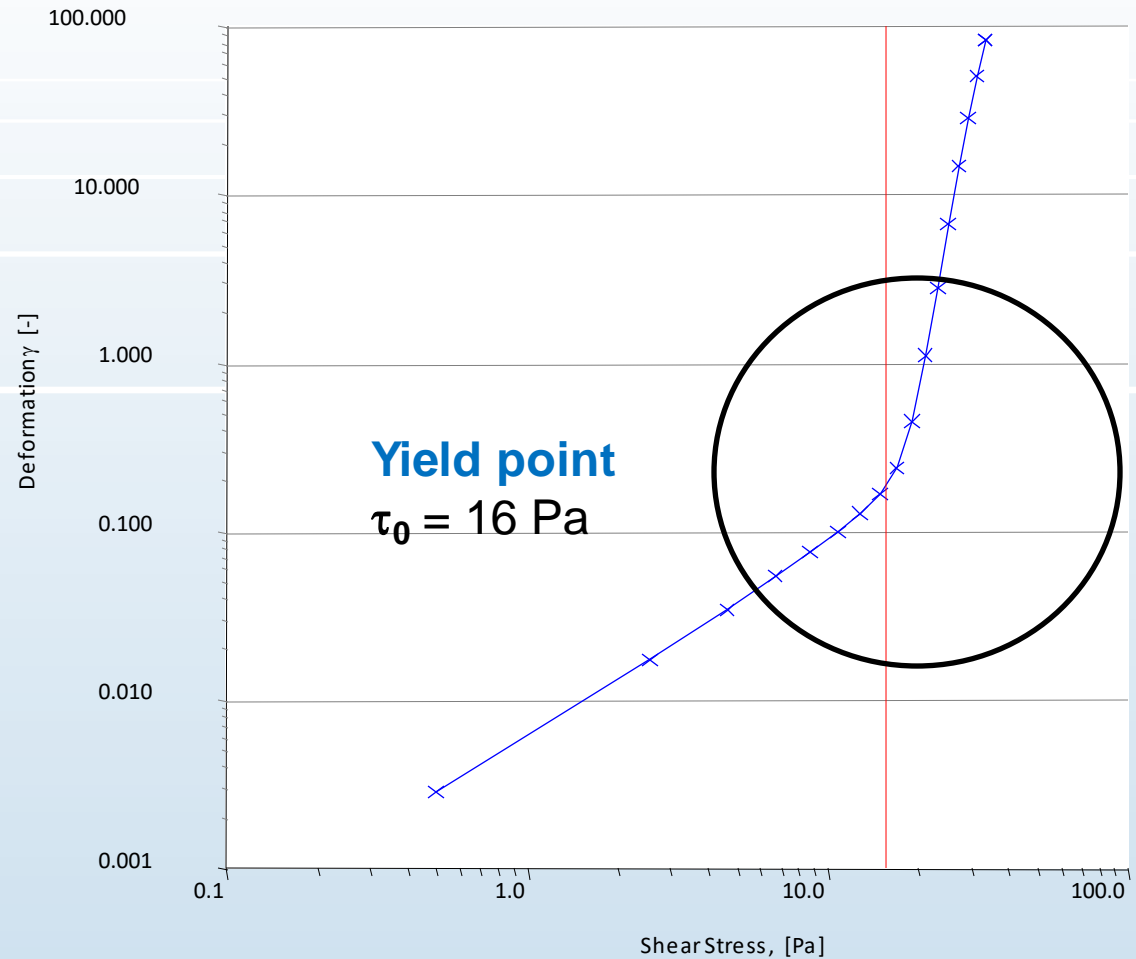


$$\tau > \tau_0$$



# Yield stress $\tau_0$ : Determination in CS-mode

- **Input:**  
shear stress  $\tau$   
(increase logarithmic)
- **Measurement:**  
deformation  $\gamma$
- **Result:**  
log deformation  $\gamma =$   
 $f(\log \text{ shear stress } \tau)$
- **Evaluation :**  
Transition between the  
linear regimes  
(= yield stress  $\tau_0$ )



# Parameters and factors affecting viscosity

*Viscosity is not a constant term*

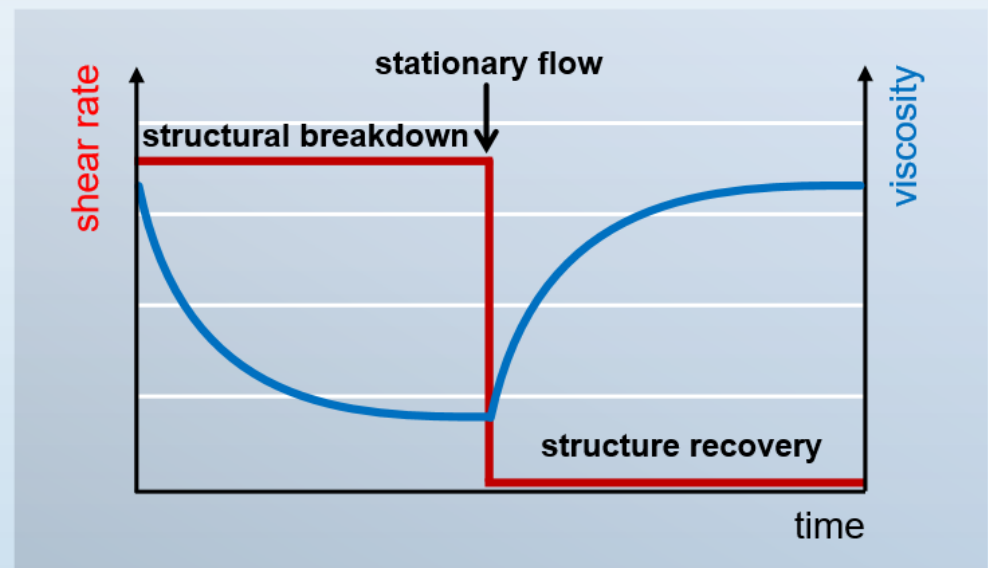
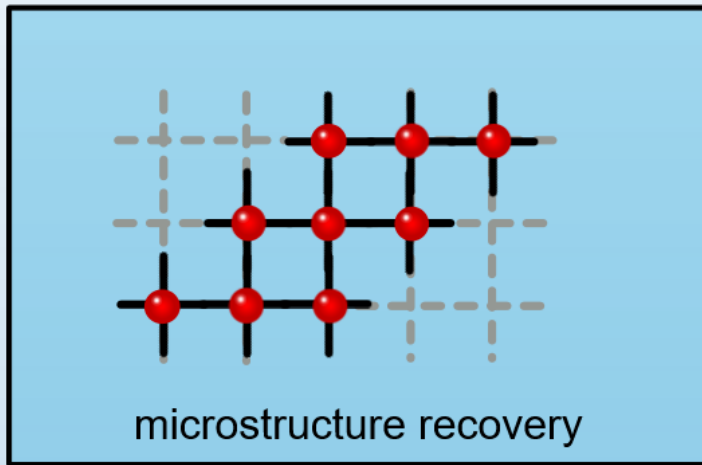


# Parameters and factors affecting viscosity

## Shear time depending flow behavior

### Thixotropy

- Thixotropy is a time delayed structural breakdown of a materials microstructure under shear
- When shear forces are removed microstructure will recover entirely



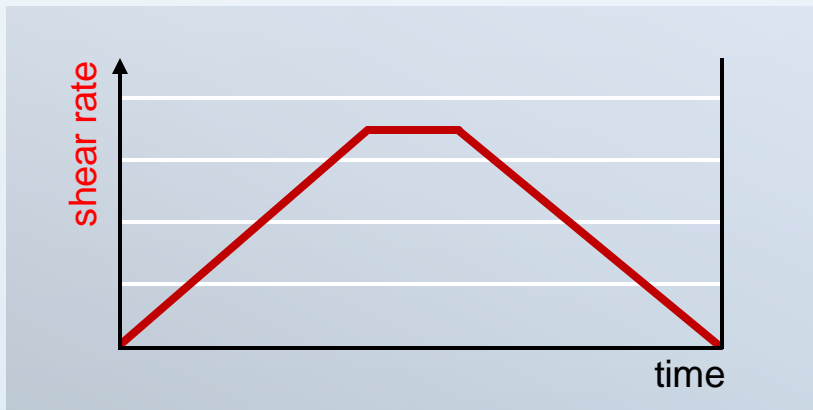
# Parameters and factors affecting viscosity

## Shear time depending flow behavior

### Measuring thixotropic behavior

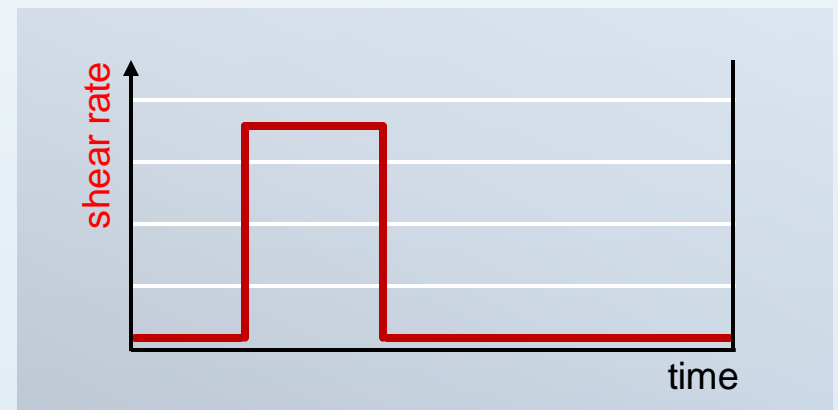
#### Thixotropy loop:

- Shear rate ramp up, peak hold and ramp down
- Shear stress signal is monitored
- Hysteresis area as a measure for thixotropy



#### 3-Interval Test:

- Recording of initial state at low shear stress, shear rate or oscillation
- Disaggregation at constant shear rate
- Re-aggregation at low shear stress, shear rate or oscillation



# Parameters and factors affecting viscosity

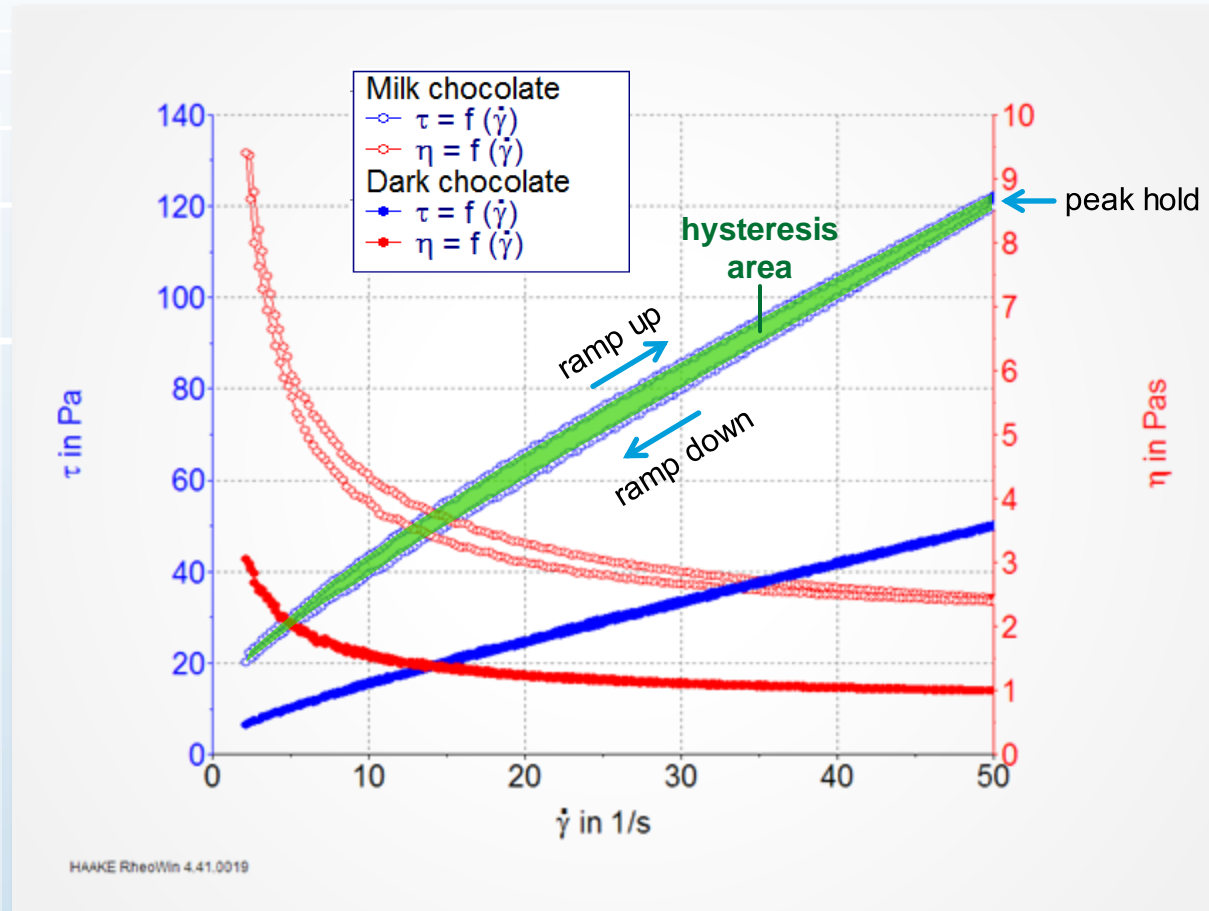
## Shear time depending flow behavior

### Thixotropic loop

- Comparison of milk and dark chocolate melt
- Thixotropy loop performed at 40°C
- Milk chocolate shows a much stronger thixotropic behavior compared to dark chocolate

milk chocolate: 203 Pa/s

dark chocolate: 22 Pa/s

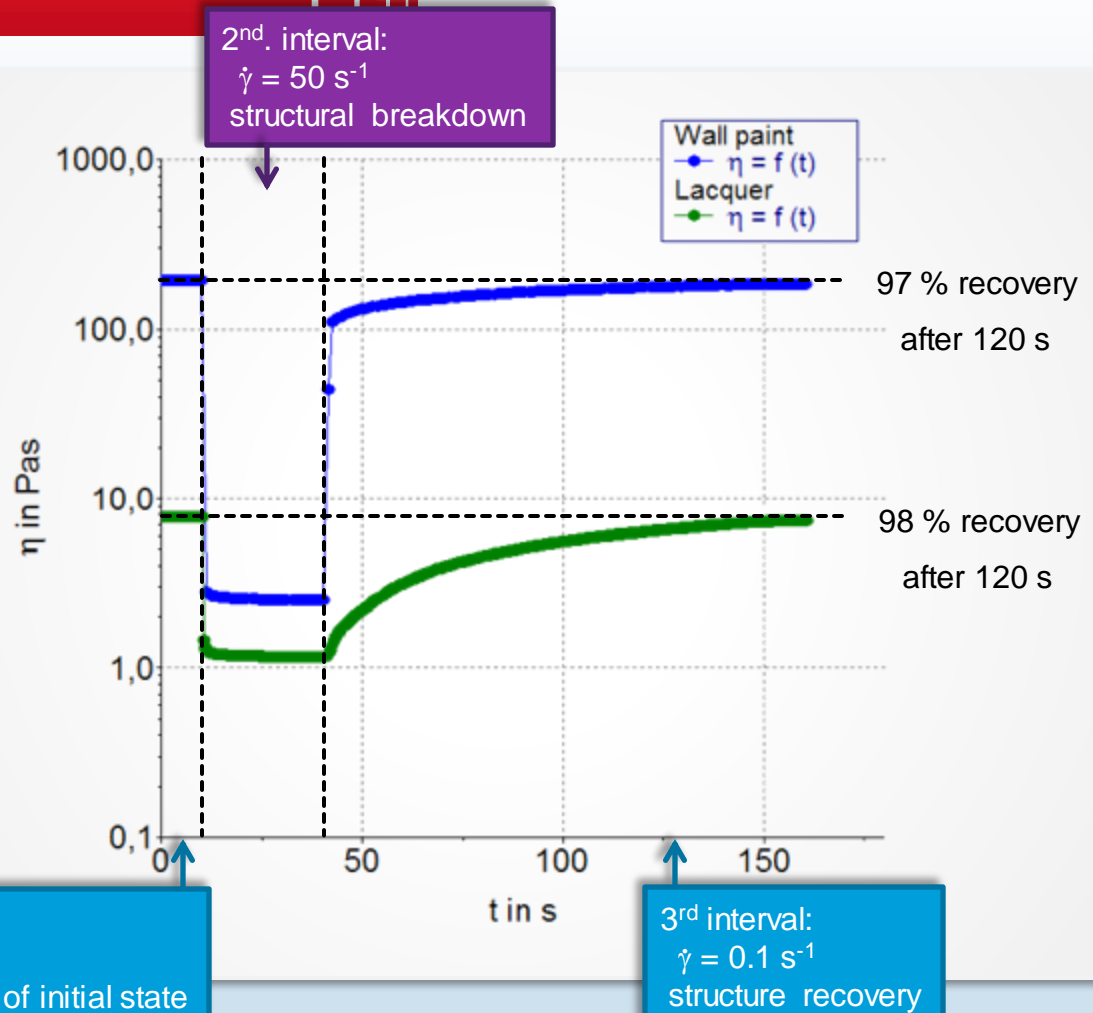


# Parameters and factors affecting viscosity

## Shear time depending flow behavior

### 3-Intervall test

- Comparison of a wall paint and a lacquer
- Both show complete recovery after 120 s at low shear rate
- The wall paint shows a more instantaneous recovery than the lacquer





# Flow behavior

## Conclusions

Newtonian flow behavior:  $\eta \neq f(\dot{\gamma})$

Non-Newtonian Flow behavior:  $\eta = f(\dot{\gamma})$

- ✓ Bingham (yield stress)
- ✓ Shear thinning (pseudoplastic)
- ✓ Plastic (yield stress)
- ✓ Dilatant (shear thickening)

Time dependant flow behavior:  $\eta = f(t, \dot{\gamma})$

- ✓ Thixotropy
- ✓ Rheopexy

# The basics of rheology and rotational rheometry

## Summary

- Viscosity is a measure for the resistance of a material against deformation and flow
- Viscosity is calculated from the shear stress and the shear rate
- The viscosity of a fluid can be affected by different parameters like temperature and shear rate
- Fluids with a shear rate independent viscosity are called Newtonian fluids
- Most complex fluids show a shear thinning behavior
- Time delayed breakdown and recovery of a material is called thixotropy



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