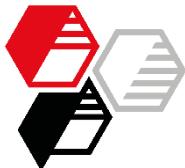


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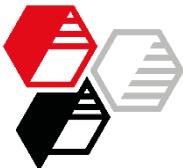
# Calcined Neuburg Siliceous Earth in white colored building profiles

Author: Nicole Holzmayr



# Contents

- Introduction
- Experimental
- Part 1: Filler comparison
  - Formulation
  - Base properties and color
  - Summary Part 1
- Part 2: Formulation optimization – Exploiting the potential of **Aktifit**
  - Formulation variations
  - Base properties and color
  - Extrusion
  - Compound costs
  - Summary Part 2
- Summary



# Status Quo

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SUMMARY

Calcined clays are being used in white colored building profiles because of their following properties:

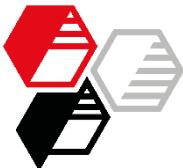
- ✓ high brightness and color-neutrality
- ✓ good extrusion properties
- ✓ good mechanical properties

**Neuburg Siliceous Earth** is an excellent alternative regarding

- ✓ extrusion properties
- ✓ mechanical properties

Until now, it was only suitable for grey profiles due to its yellowish tint, whereas its use in white profiles was problematic regarding

- ✗ color-neutrality



# Objective

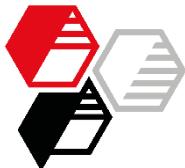
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- Effect of **Calcined Neuburg Siliceous Earth** in grey-white building profiles regarding
  - base properties  
orientation to the standard for extruded sealing profiles of RAL GZ 716/1, elastomer class B II
  - color  
referring to RAL 9002
- Comparison to calcined clay
- Impact of the used polymer
- Influence of the filler loading and peroxide dosage



# Fillers, Characteristics

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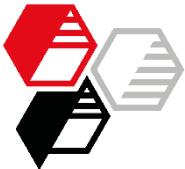
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		Particle Size		Oil absorption [g/100g]	Specific surface area BET [m <sup>2</sup> /g]	Calcination
		d <sub>50</sub> [μm]	d <sub>97</sub> [μm]			Surface treatment
	Polestar 200 R	3.6	19	60	6.5	with none
	Sillitin Z 89	1.6	6.1	51	11	none none
	Silfit Z 91	1.9	8.6	52	6.5	with none
	Aktisil VM 56/89	1.8	7.4	45	7.3	none vinyl
	Aktifit VM	1.9	8.7	49	6.2	with vinyl



# Preparation and Curing of the Compound

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- **Mixing**

Open mill Ø 150 x 300 mm

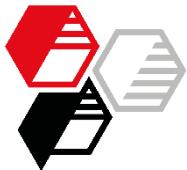
Batch volume: approx. 750 g

Temperature: 50 °C

Mixing time: approx. 15 min.

- **Curing**

Press, 180 °C, 5 min.

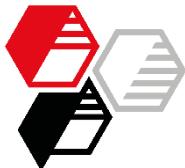


# Overview

Part 1: Filler comparison

Part 2: Formulation optimization –  
Exploiting the potential of **Aktifit VM**

Summary



# Formulation

INTRODUCTION

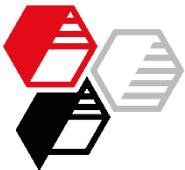
EXPERIMENTAL

Filler comparison

RESULTS

SUMMARY

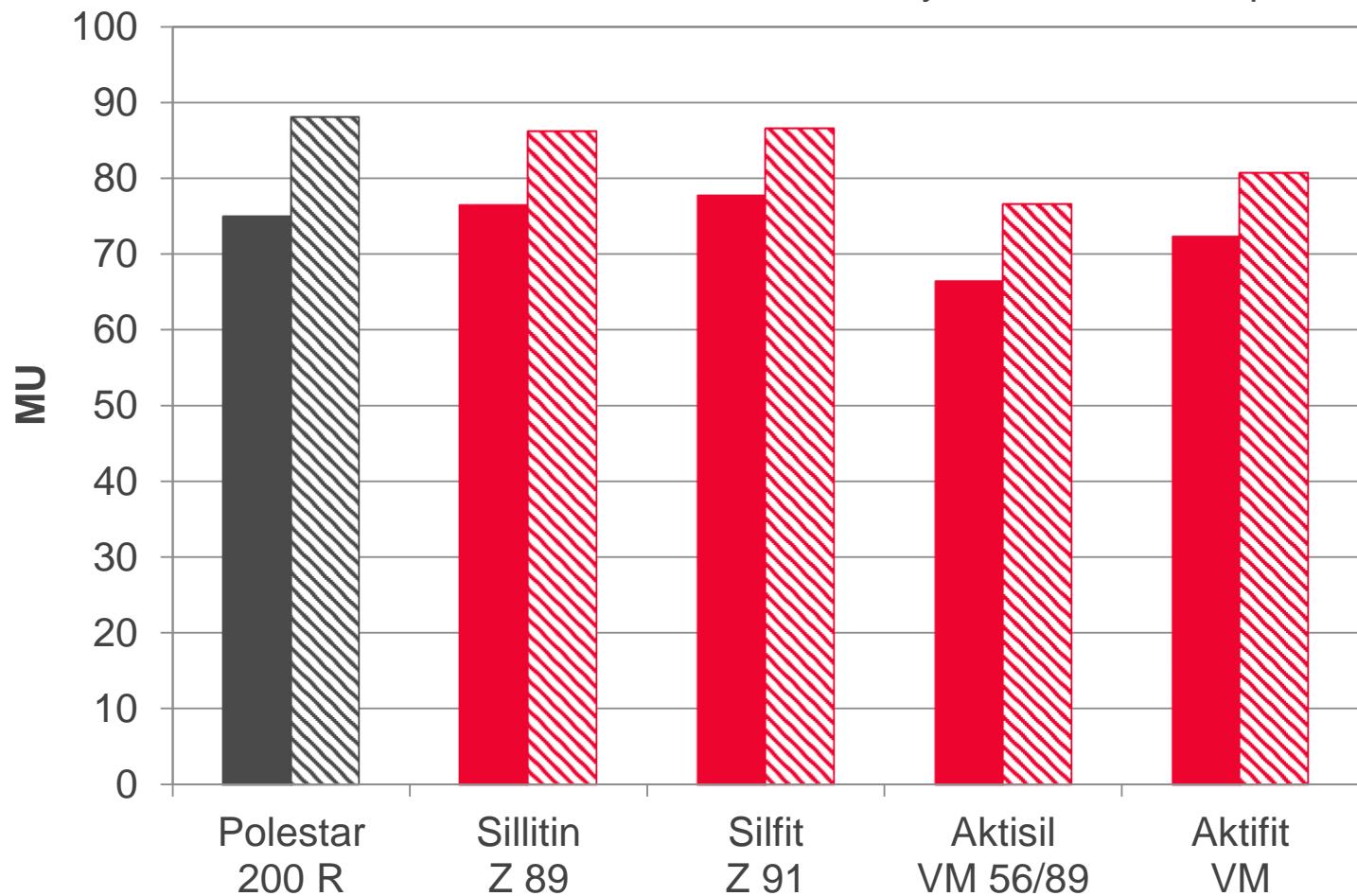
	in phr
Keltan 778 Z – crystalline or Keltan 8340 A – amorphous	100.00
Zinkoxyd aktiv	5.00
Stearic acid	0.50
Vulkanox HS/LG	0.75
Lipoxol 3000	2.00
Kronos 2222	15.00
Kezadol GR	10.00
Aflux 42	2.00
Trigonox 29/40 B pd	3.00
Perkadox 14/40 B pd	3.00
Rhenofit TRIM/S	1.00
Primol 352	45.00
Mineral filler	200.00
<b>Total</b>	<b>387.25</b>



# Mooney-Viscosity

DIN 53 523 Part 3, ML +5, 120 °C

■ crystalline ▨ amorphous



INTRODUCTION

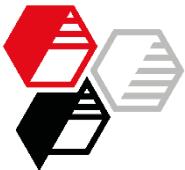
EXPERIMENTAL

## RESULTS

Filler comparison

• Rheology

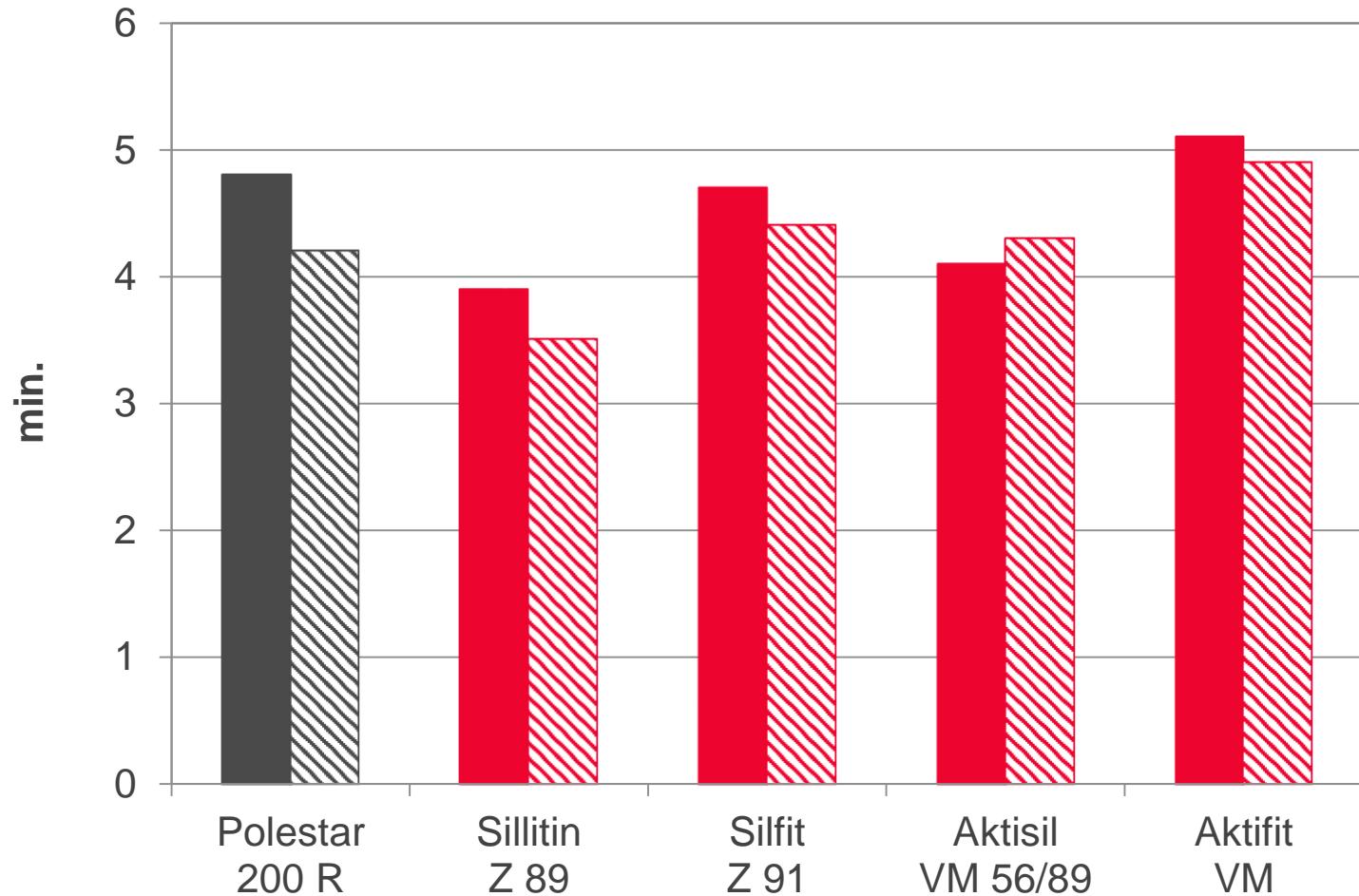
SUMMARY



# Mooney Scorch Time

DIN 53 523 Part 4, ML +5, 120 °C

■ crystalline ▨ amorphous



INTRODUCTION

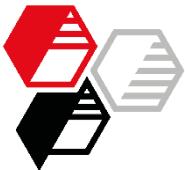
EXPERIMENTAL

## RESULTS

Filler comparison

• Rheology

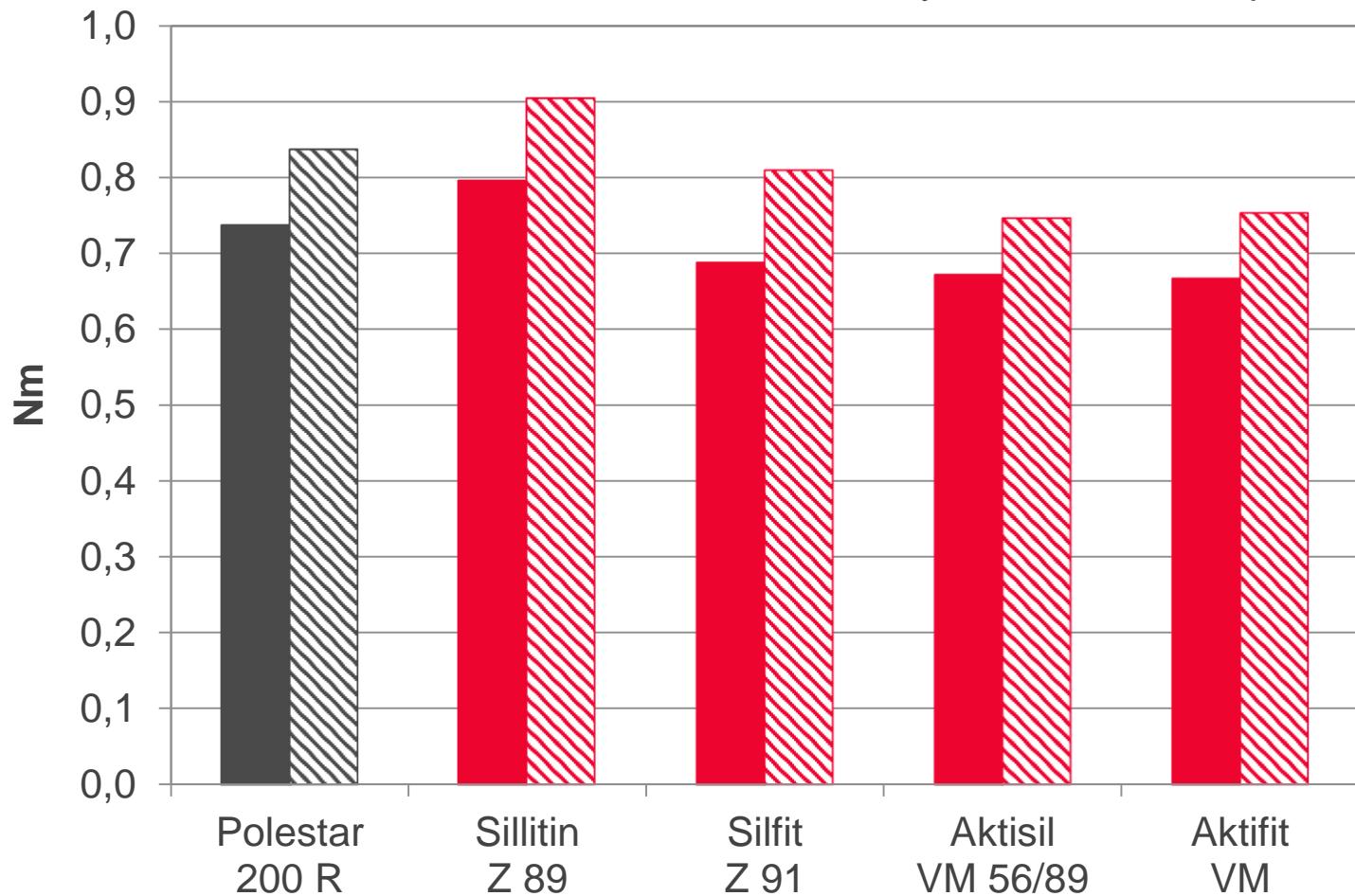
SUMMARY



# Torque Maximum

DIN 53 529-A3, 180 °C, 0,2° deflection – Göttfert Elastograph

■ ■ crystalline ■ ■ amorphous



INTRODUCTION

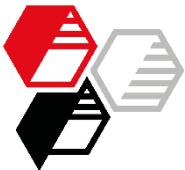
EXPERIMENTAL

## RESULTS

Filler comparison

• Rheology

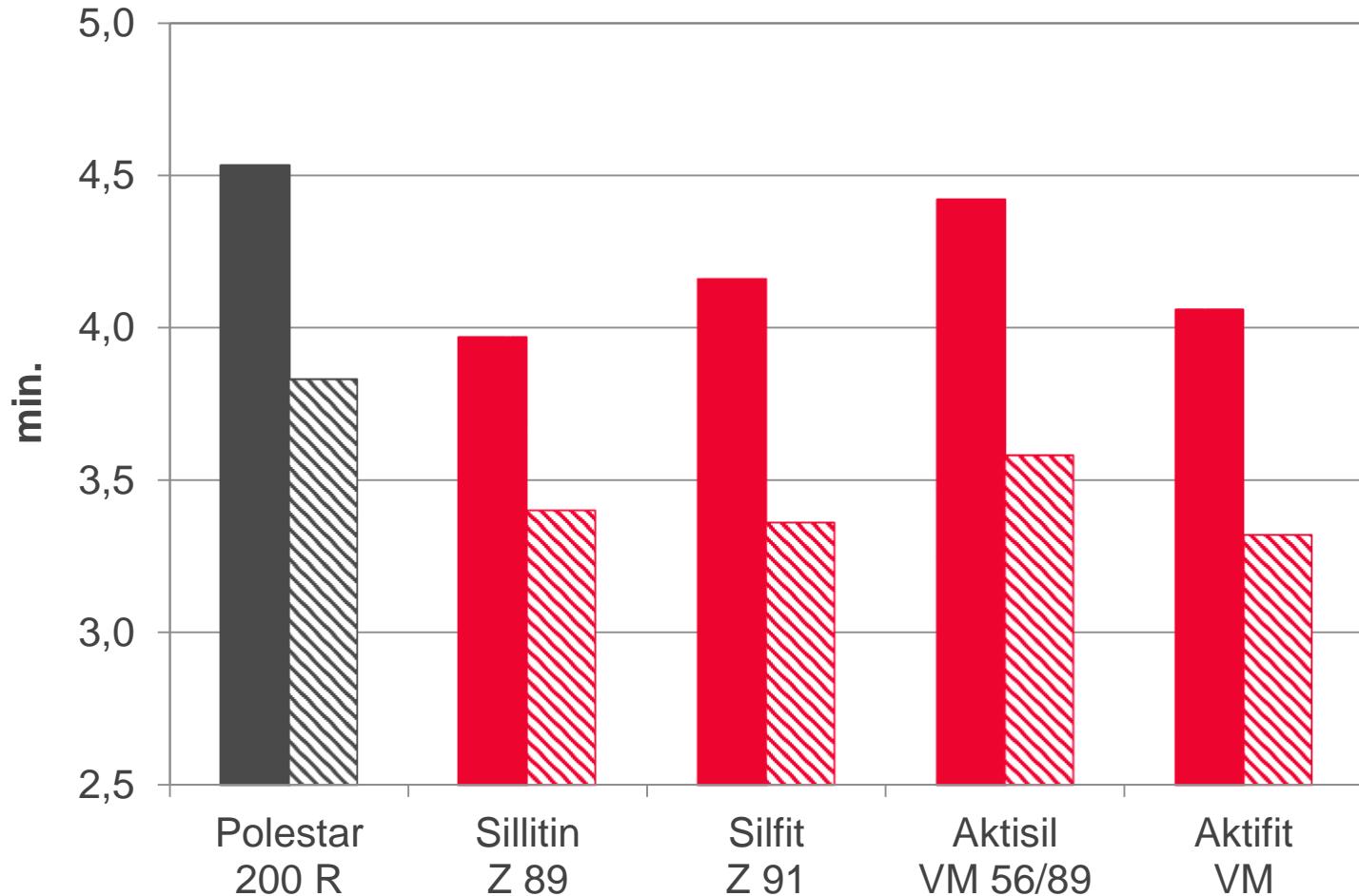
SUMMARY



# Conversion Time $t_{90}$

DIN 53 529-A3, 180 °C, 0,2° deflection – Göttfert Elastograph

■ ■ crystalline   ■ ■ amorphous



INTRODUCTION

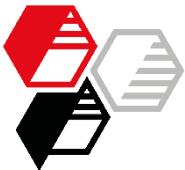
EXPERIMENTAL

## RESULTS

Filler comparison

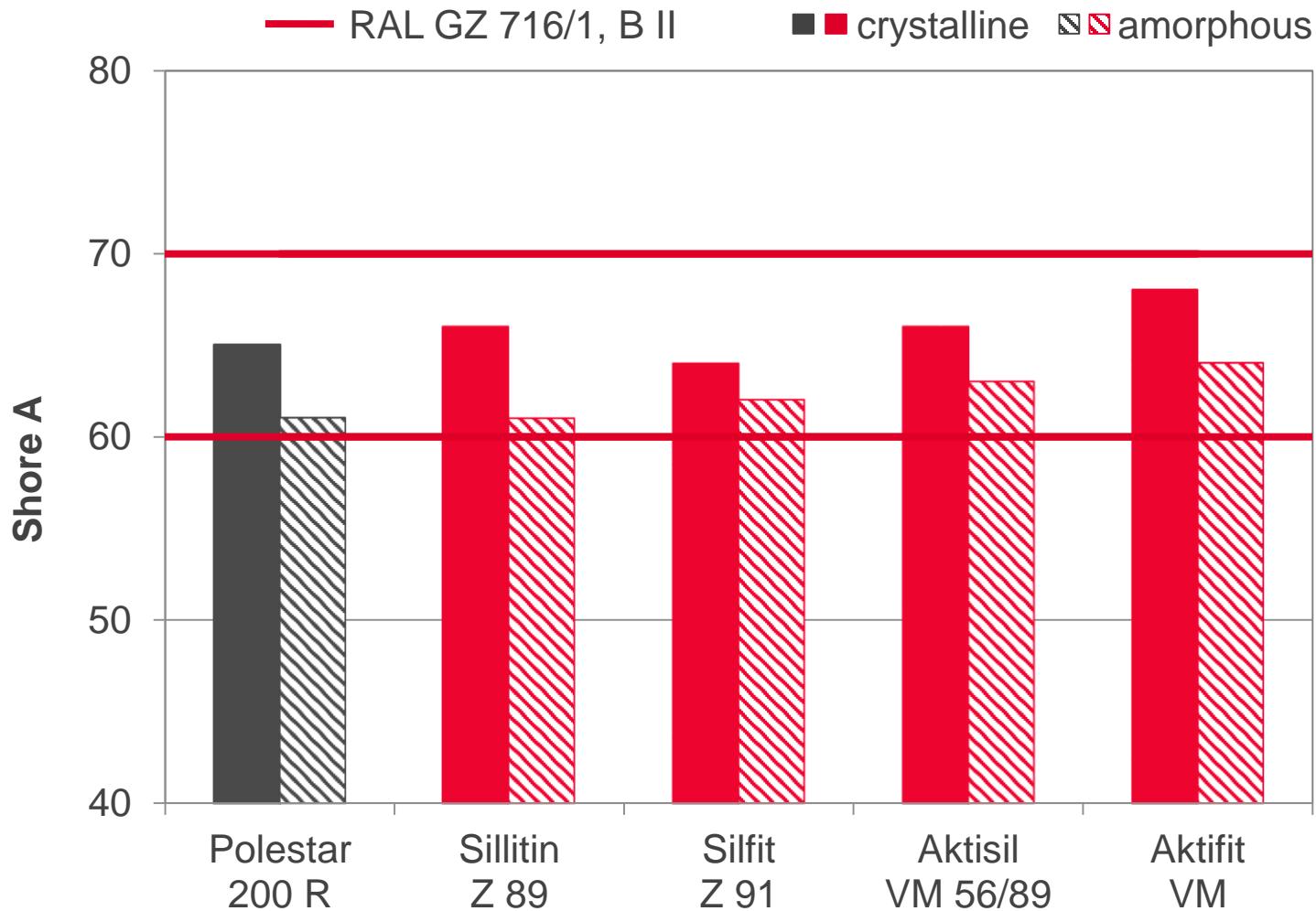
• Rheology

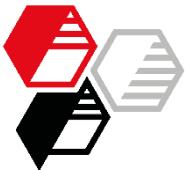
SUMMARY



# Hardness

DIN 53 505-A, piled-up S2-dumbbells





# Tensile Strength

DIN 53 504, S2

— RAL GZ 716/1, B II

■ crystalline ▨ amorphous

INTRODUCTION

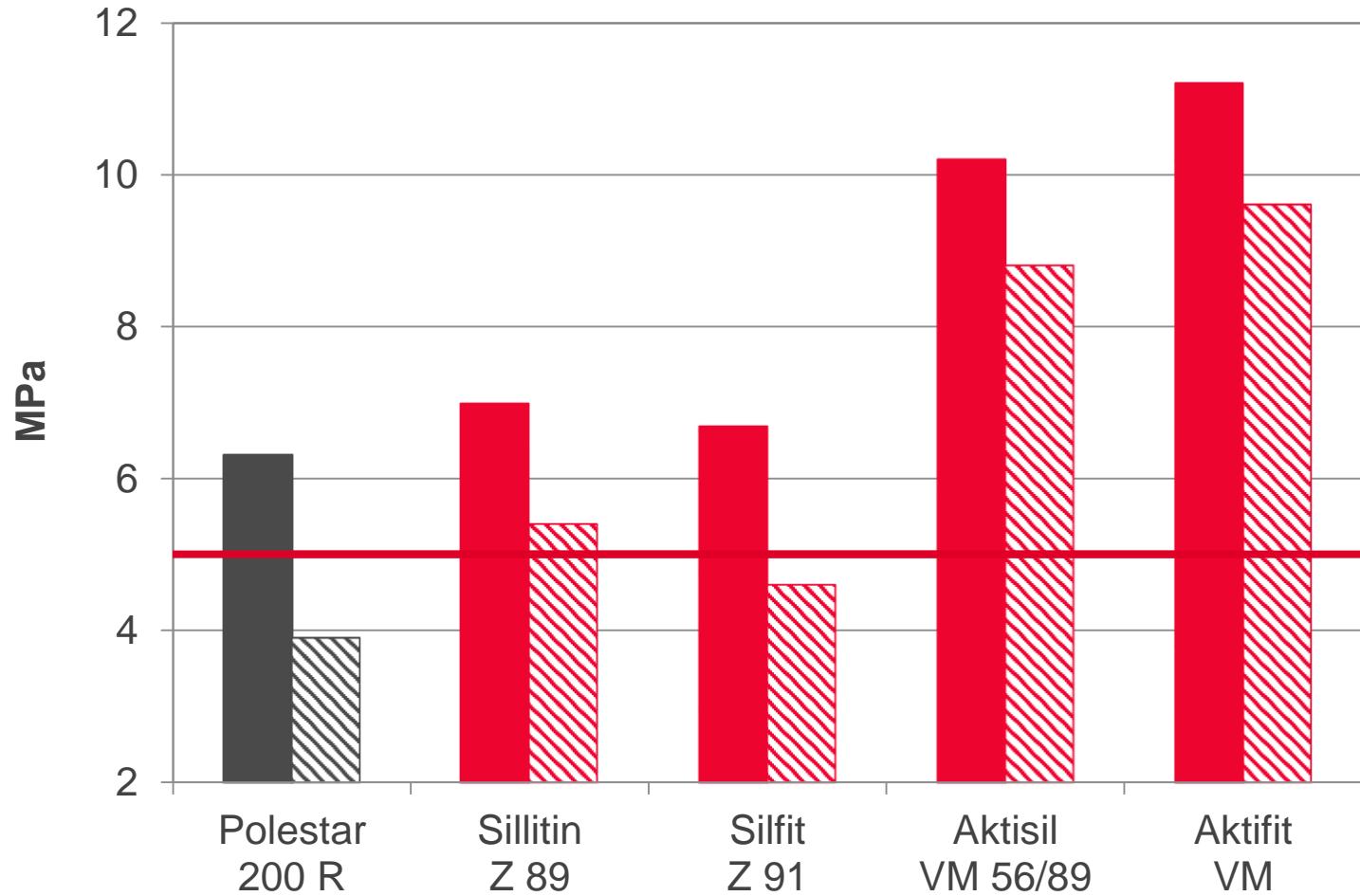
EXPERIMENTAL

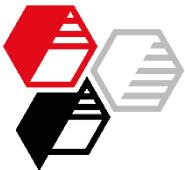
## RESULTS

Filler comparison

- Base properties

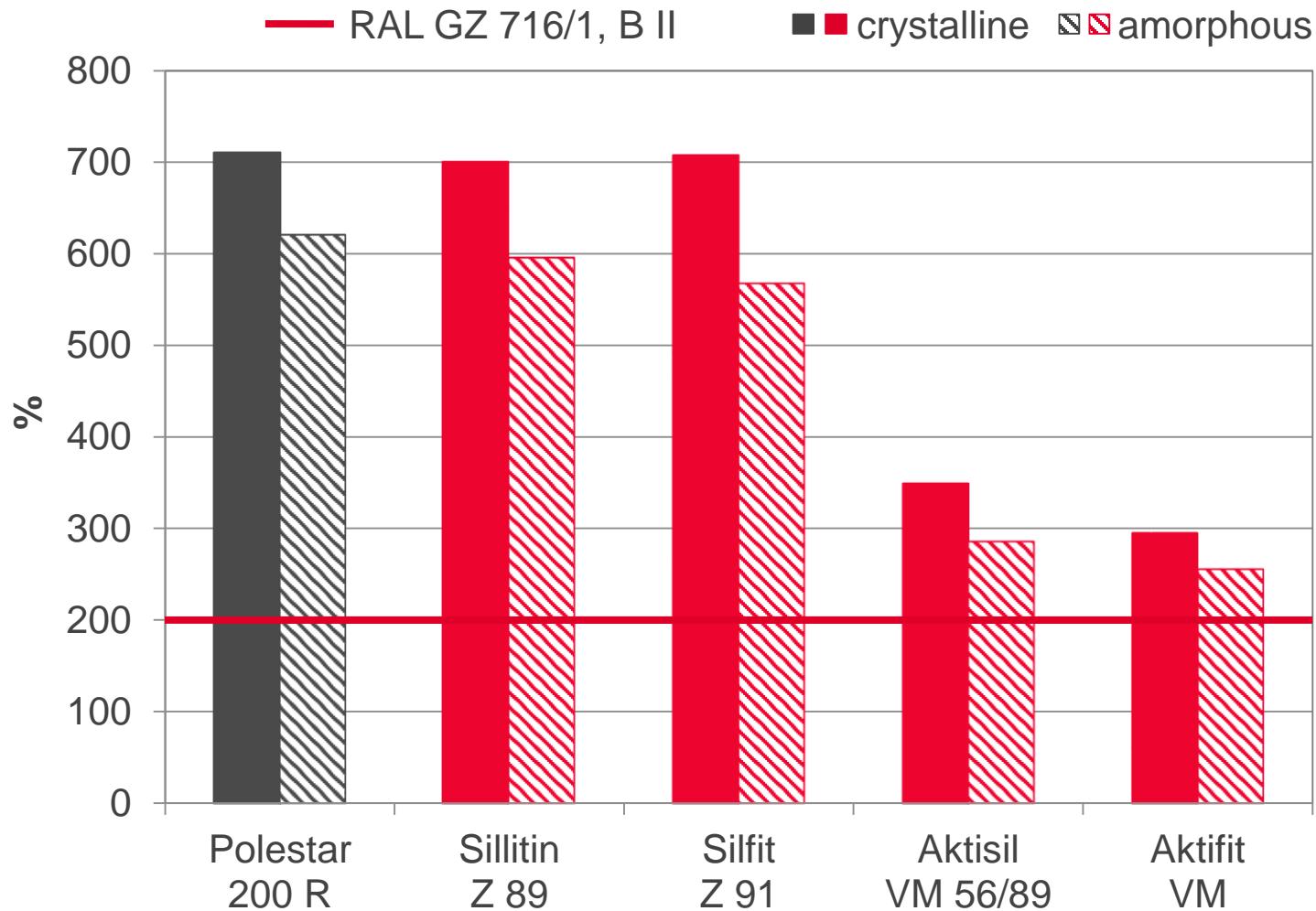
SUMMARY





# Elongation at Break

DIN 53 504, S2



INTRODUCTION

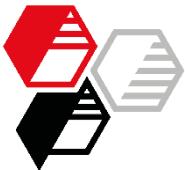
EXPERIMENTAL

## RESULTS

Filler comparison

- Base properties

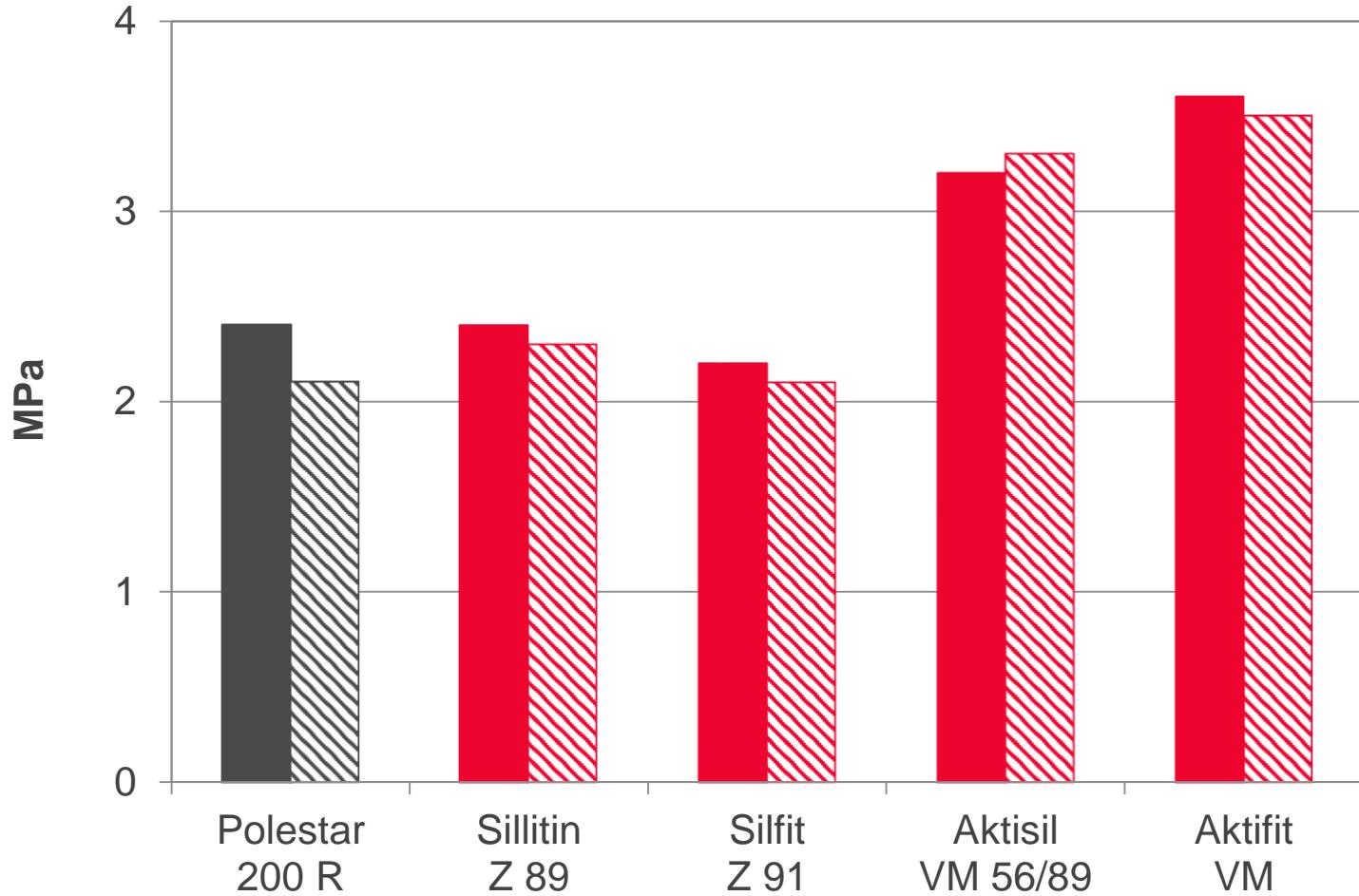
SUMMARY



# Modulus 100 %

DIN 53 504, S2

■ crystalline ▨ amorphous



INTRODUCTION

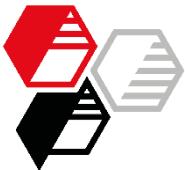
EXPERIMENTAL

## RESULTS

Filler comparison

- Base properties

SUMMARY



# Compression Set

## 24 h / 125 °C

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INTRODUCTION

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### RESULTS

Filler comparison

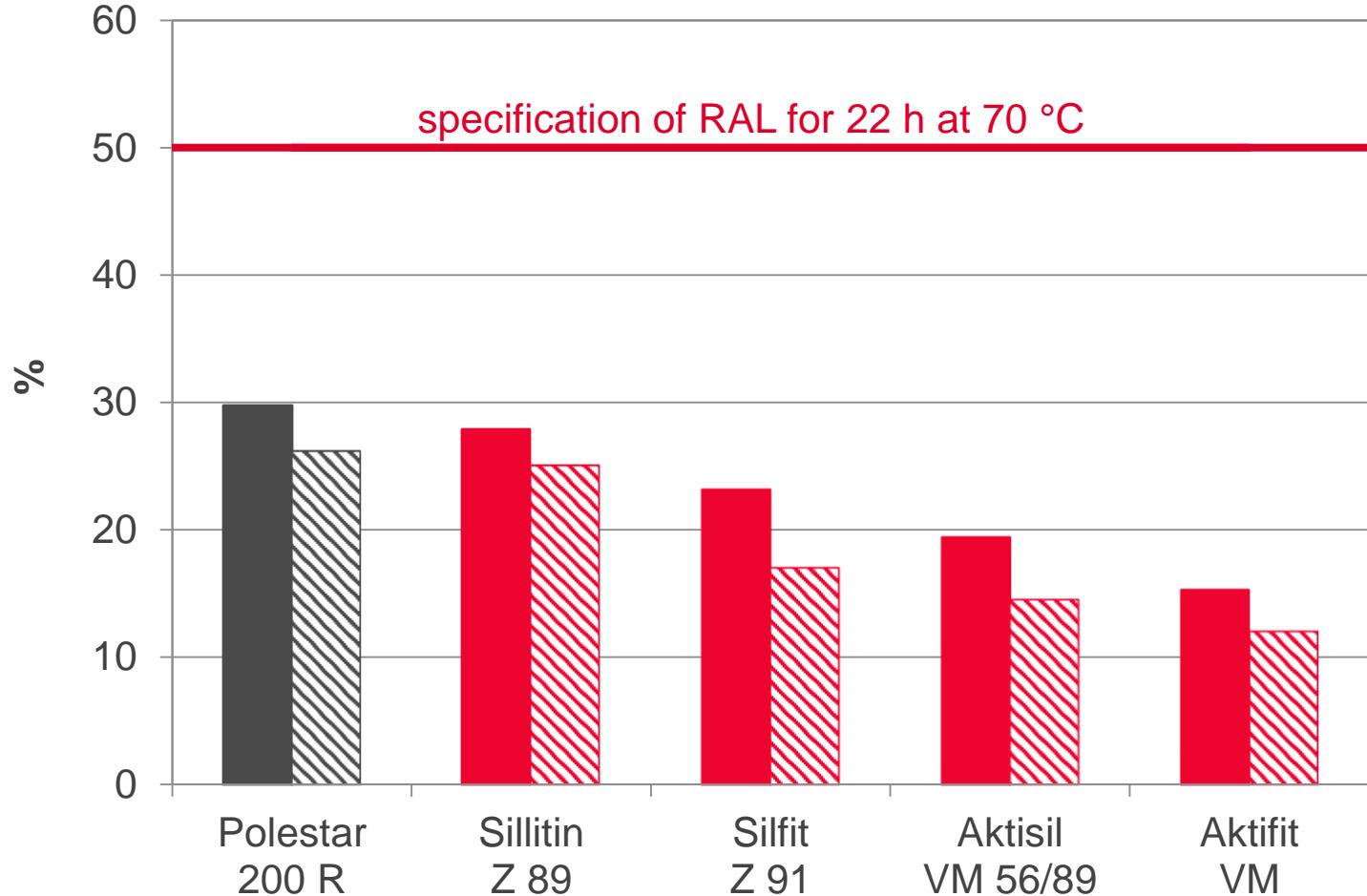
- Base properties

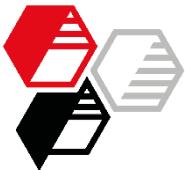
SUMMARY

DIN ISO 815-1 B, cooling method A, 25 % defl.

— RAL GZ 716/1, B II

■ crystalline ▨ amorphous





# Compression Set

## 22 h / 23 °C

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INTRODUCTION

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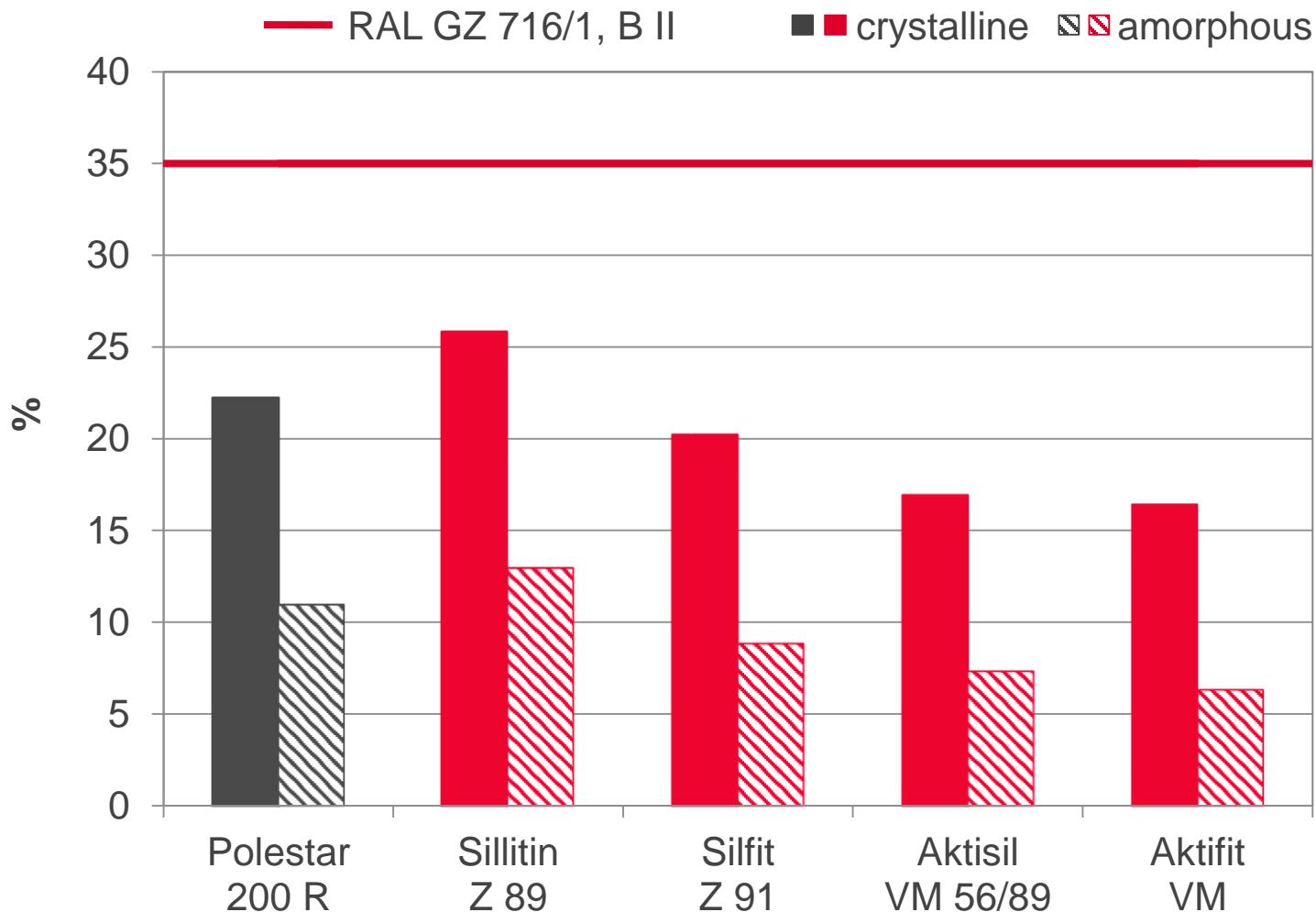
### RESULTS

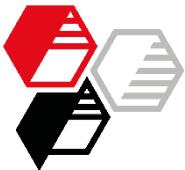
Filler comparison

• Base properties

SUMMARY

DIN ISO 815-1 B, cooling method A, 25 % defl.





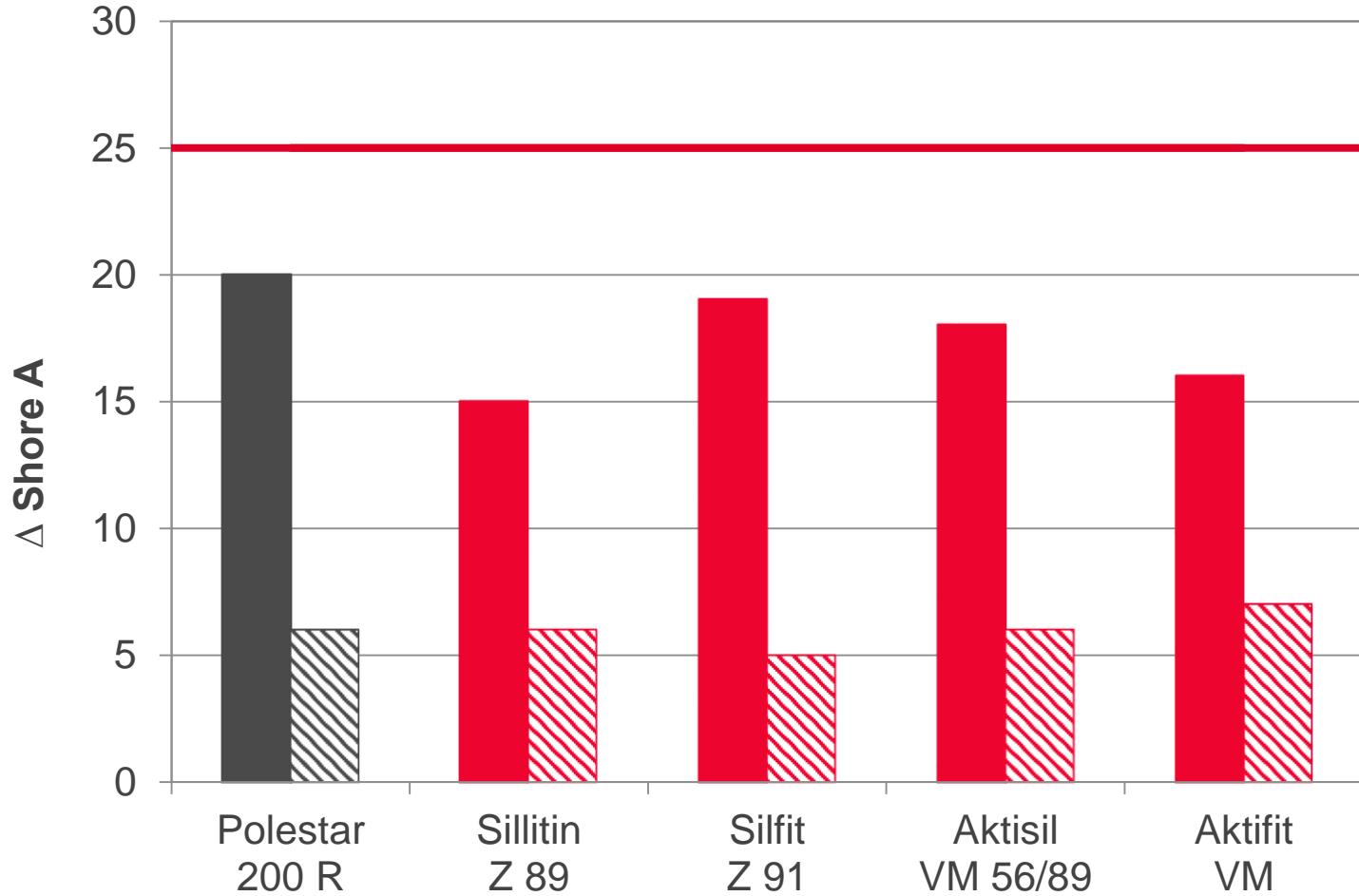
# Low Temperature Exposure Change of Hardness

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22 h / -10 °C

— RAL GZ 716/1, B II

■ crystalline ▨ amorphous



INTRODUCTION

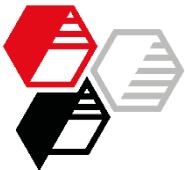
EXPERIMENTAL

## RESULTS

Filler comparison

- Base properties

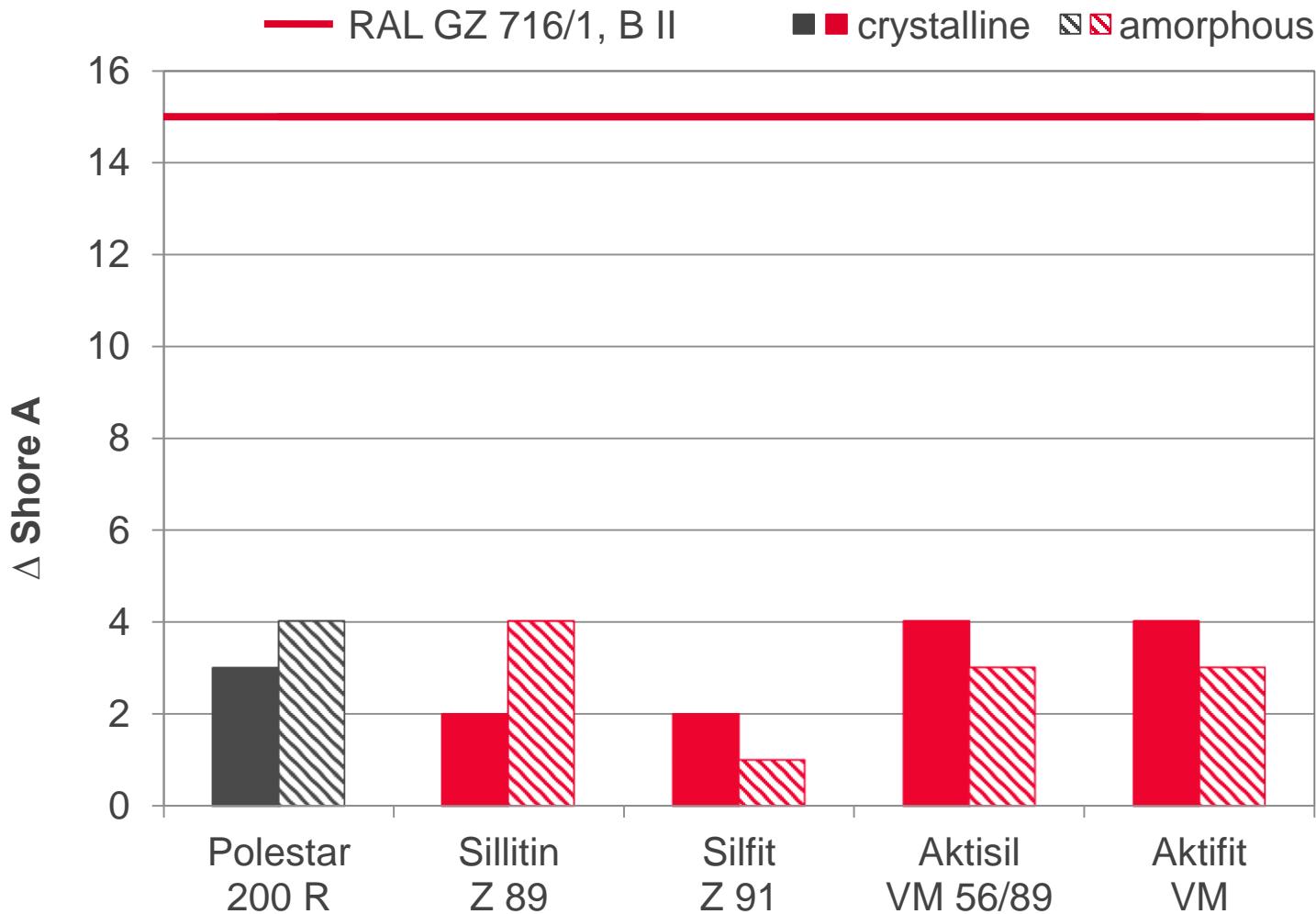
SUMMARY

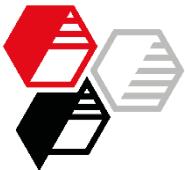


# Hot Air Aging Change of Hardness

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DIN 53 508, 168 h / 100 °C





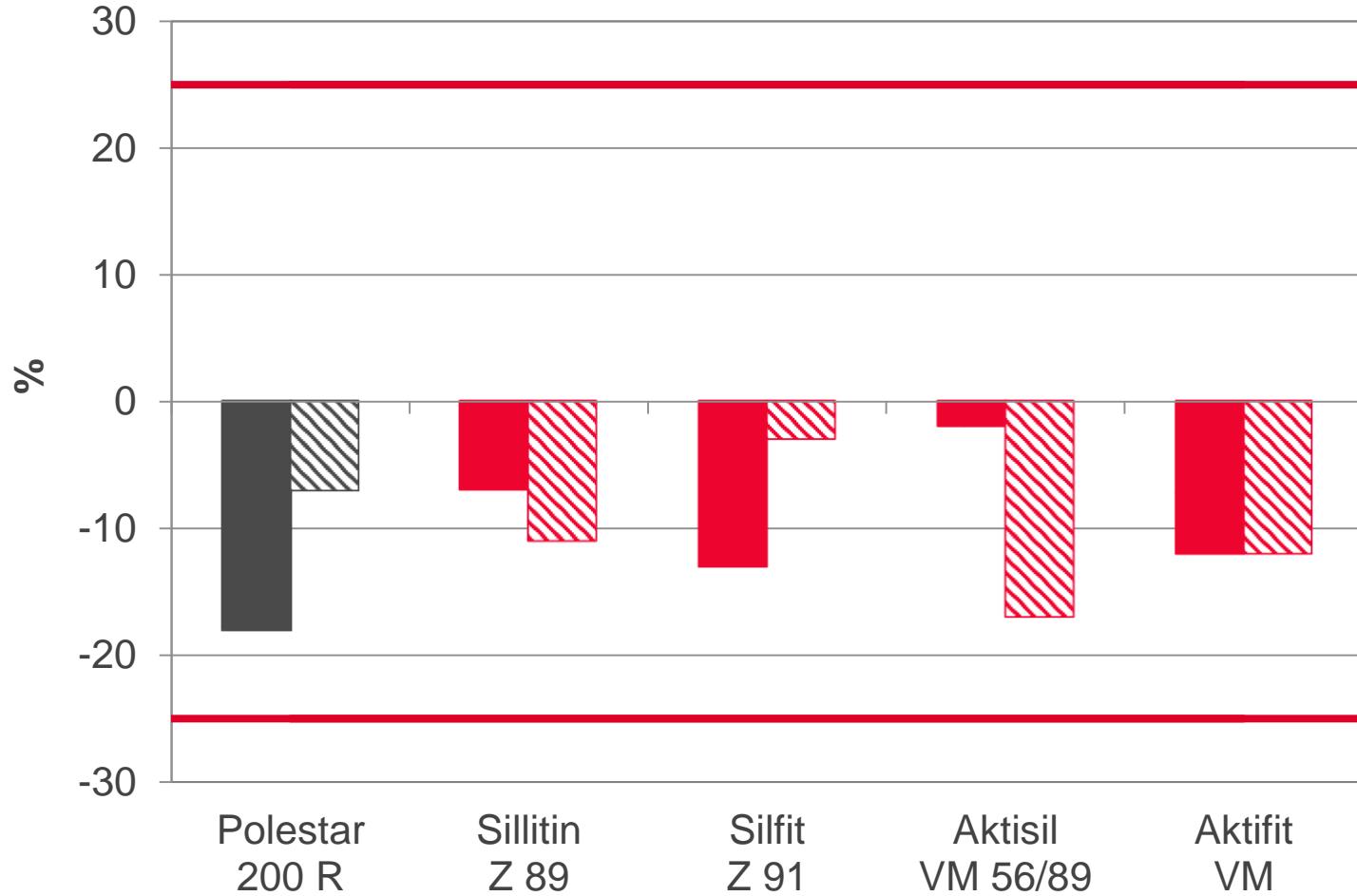
# Hot Air Aging Change of Tensile Strength

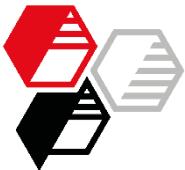
**HOFFMANN**  
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DIN 53 508, 168 h / 100 °C

— RAL GZ 716/1, B II

■ crystalline ▨ amorphous





# Hot Air Aging Remaining Elongation at B.

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INTRODUCTION

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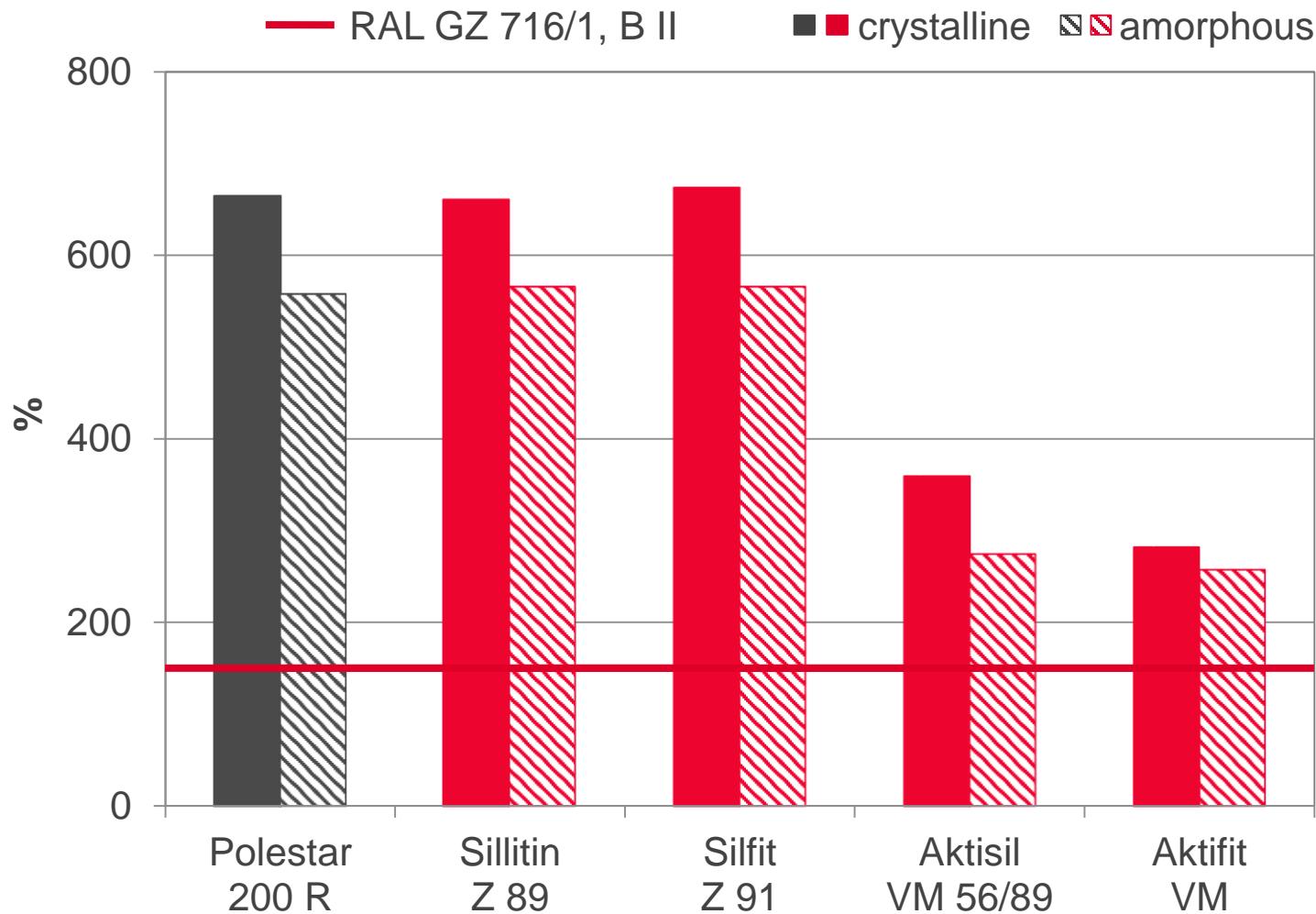
## RESULTS

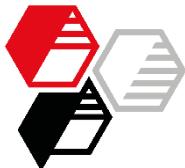
Filler comparison

- Base properties

SUMMARY

DIN 53 508, 168 h / 100 °C





# Measurement of Color

ISO 7724

- **Parameter**

Spectral photometer (Luci 100, Dr. Lange)

Light D 65

Geometry d/8°, without gloss trap

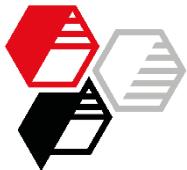
Observation angle 10°

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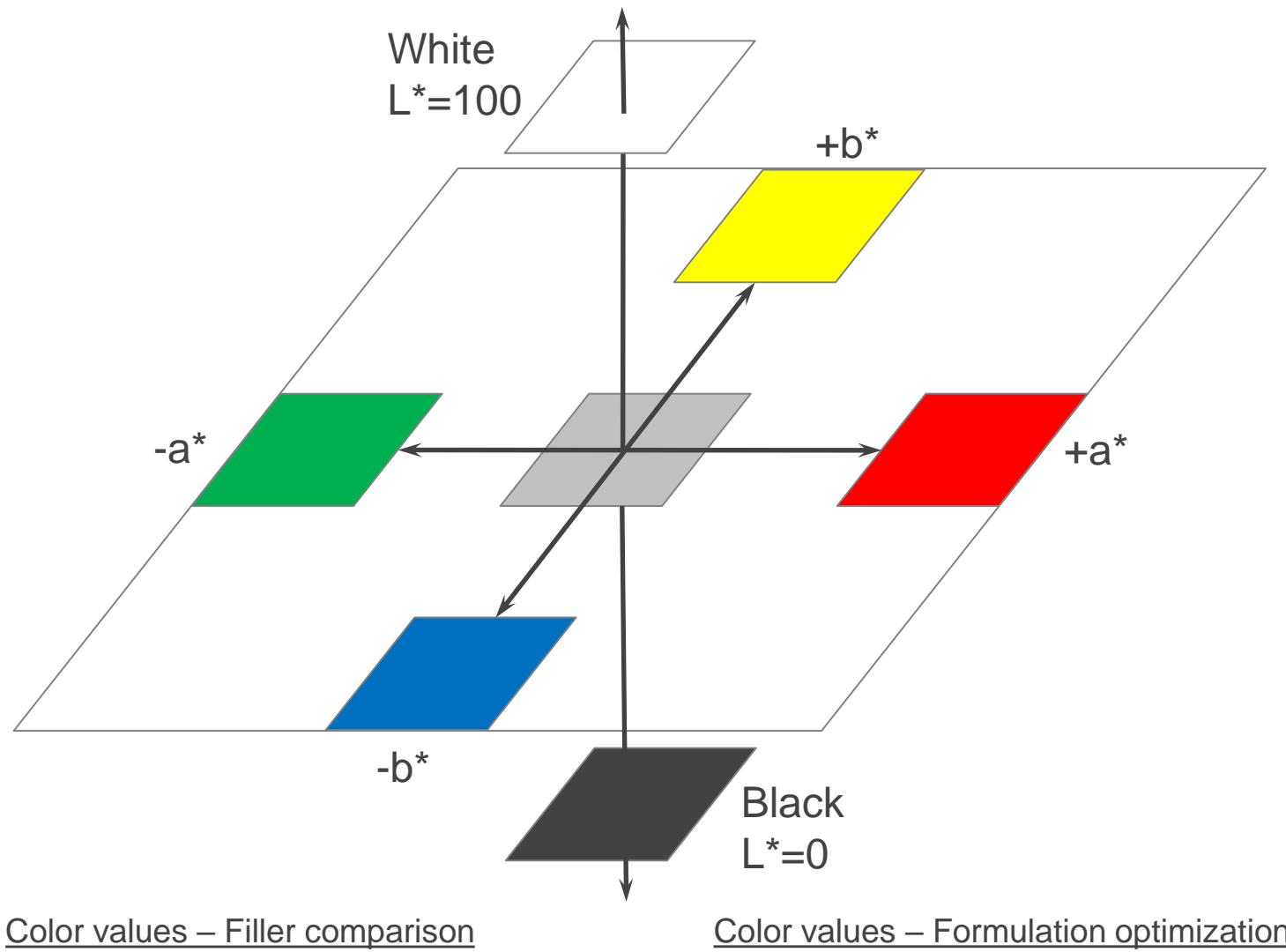
# CIE-LAB-System

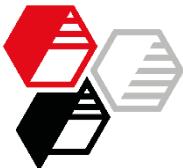
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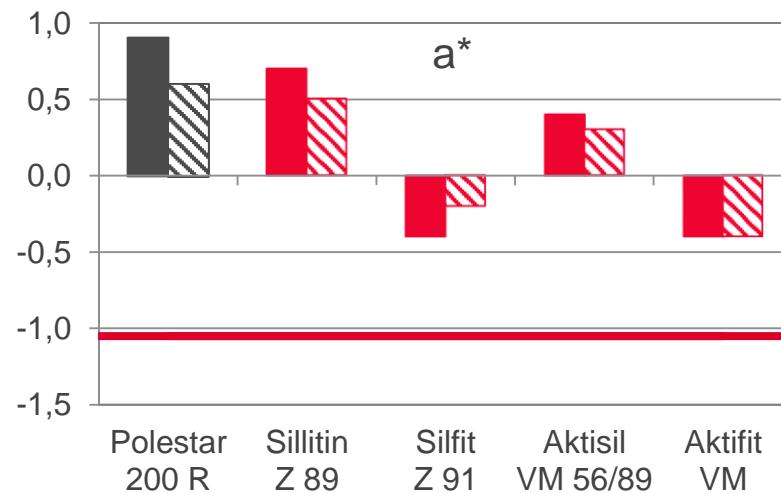
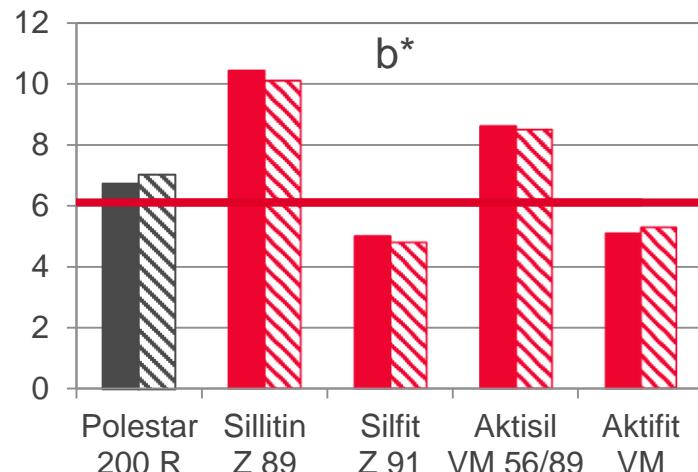
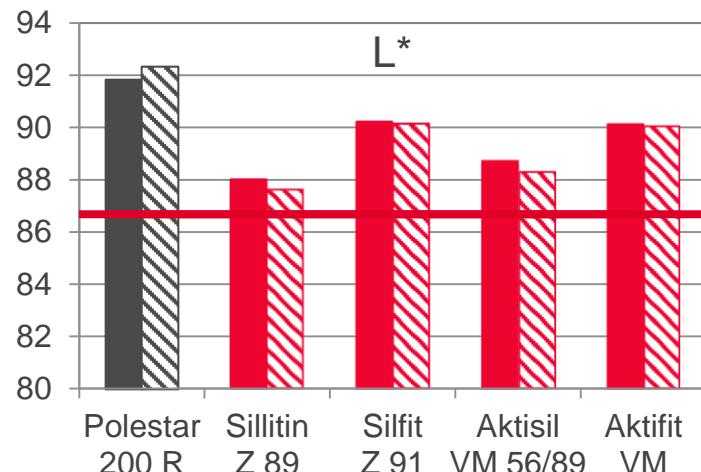
# Color Values

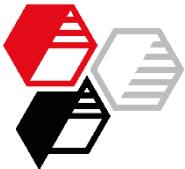
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Filler comparison  
• Color  
SUMMARY

— RAL 9002

■ ■ crystalline ▨ ▨ amorphous





# Filler Comparison Summary

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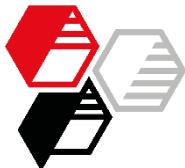
RESULTS

## SUMMARY

Filler comparison

### Silfit Z 91:

- ✓ meets the tested requirements of RAL GZ 716/1, B II with a filler loading of 200 phr without any problems in combination with a crystalline polymer.  
With an amorphous polymer, an optimization is only necessary regarding tensile strength.
- ✓ results in very neutral color in comparison to **Sillitin Z 89**.
- ✓ leads to a reduced conversion time  $t_{90}$ , a slightly higher tensile strength by tendency and to a lower compression set in comparison to calcined clay at a filler loading of 200 phr.



# Summary Part 1

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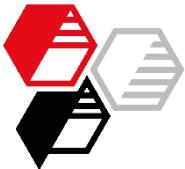
RESULTS

SUMMARY

Filler comparison

## Aktifit VM:

- ✓ meets the tested requirements of RAL GZ 716/1, B II with a filler loading of 200 phr.  
Even with an amorphous polymer all tested requirements can be fulfilled.
- ✓ results in a very neutral color in comparison to Aktisil **VM 56/89**.
- ✓ leads to a reduced conversion time  $t_{90}$  and to improved mechanical properties in comparison to calcined clay and **Aktisil VM 56/89**, especially to a higher tensile strength and a lower compression set at a filler loading of 200 phr.



# Filler Comparison Conclusion

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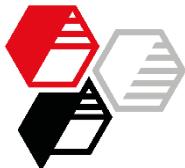
RESULTS

SUMMARY

Filler comparison

- The very good properties leave a margin for further optimization:
  - a higher filler loading (e.g. 300 to 400 phr **Aktifit VM** with 70 to 95 phr plasticizer) and / or
  - a reduction of the peroxide dosage





# Formulation

INTRODUCTION

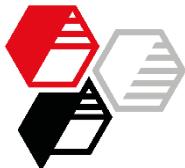
EXPERIMENTAL

Formulation optimization

RESULTS

SUMMARY

	<b>in phr</b>
Keltan 778 Z – crystalline or Keltan 8340 A – amorphous	100.00
Zinkoxyd aktiv	5.00
Stearic acid	0.50
Vulkanox HS/LG	0.75
Lipoxol 3000	2.00
Kronos 2222	15.00
Kezadol GR	10.00
Aflux 42	2.00
Trigonox 29/40 B pd	2 or 3
Perkadox 14/40 B pd	2 or 3
Rhenofit TRIM/S	1.00
Primol 352	45 - 95
<b>Mineral filler</b>	<b>200 - 400</b>



# Formulation Variations

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INTRODUCTION

## EXPERIMENTAL

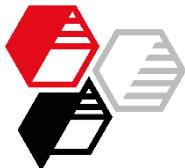
Formulation optimization

RESULTS

SUMMARY

Loadings in phr

		crystalline		amorphous	
Polestar 200 R (Reference)	200				
Aktifit VM		400	300	300	300
Primol 352	45	95	70	70	70
Trigonox 29/40 B pd	3	3	2	3	2
Perkadox 14/40 B pd	3	3	2	3	2



# Mooney-Viscosity

DIN 53 523 Part3, ML 1+4, 100 °C

INTRODUCTION

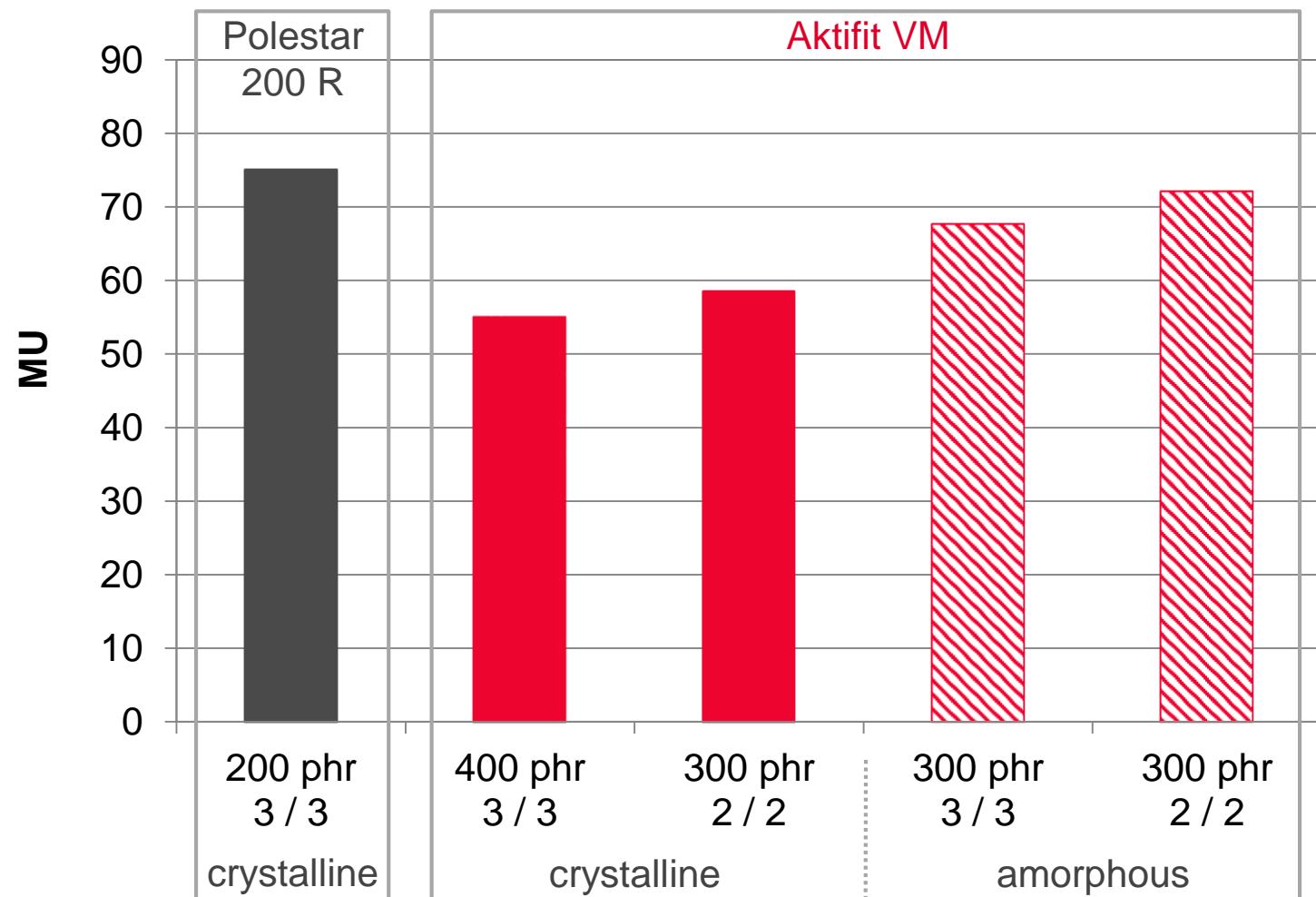
EXPERIMENTAL

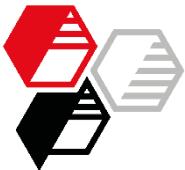
## RESULTS

Formulation optimization

• Rheology

SUMMARY





# Mooney Scorch Time

DIN 53 523 Part 4, ML +5, 120 °C

INTRODUCTION

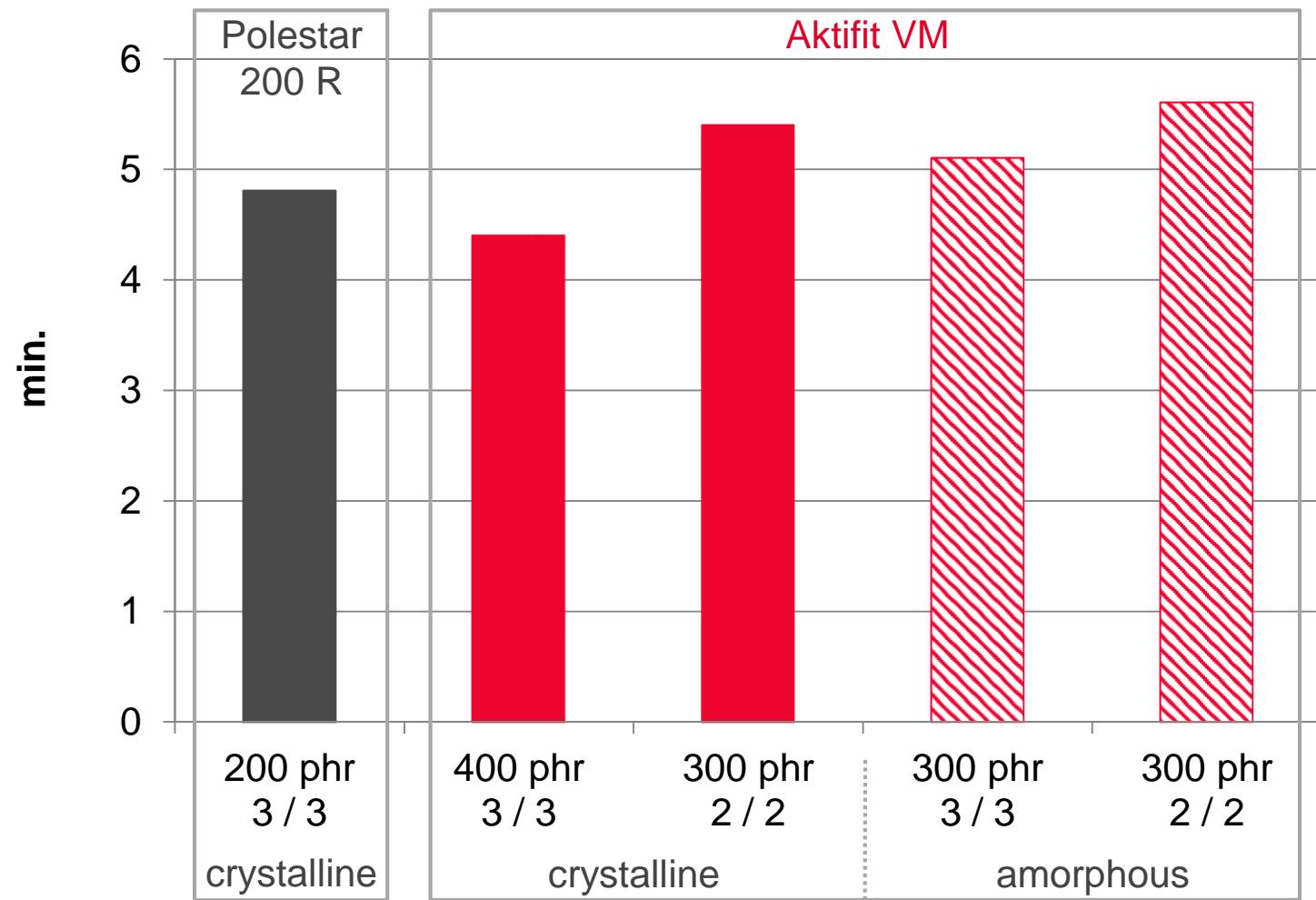
EXPERIMENTAL

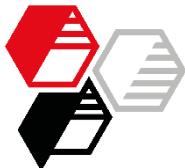
## RESULTS

Formulation optimization

- Rheology

SUMMARY





# Torque Maximum

DIN 53 529-A3, 180 °C, 0,2° deflection – Göttfert Elastograph

INTRODUCTION

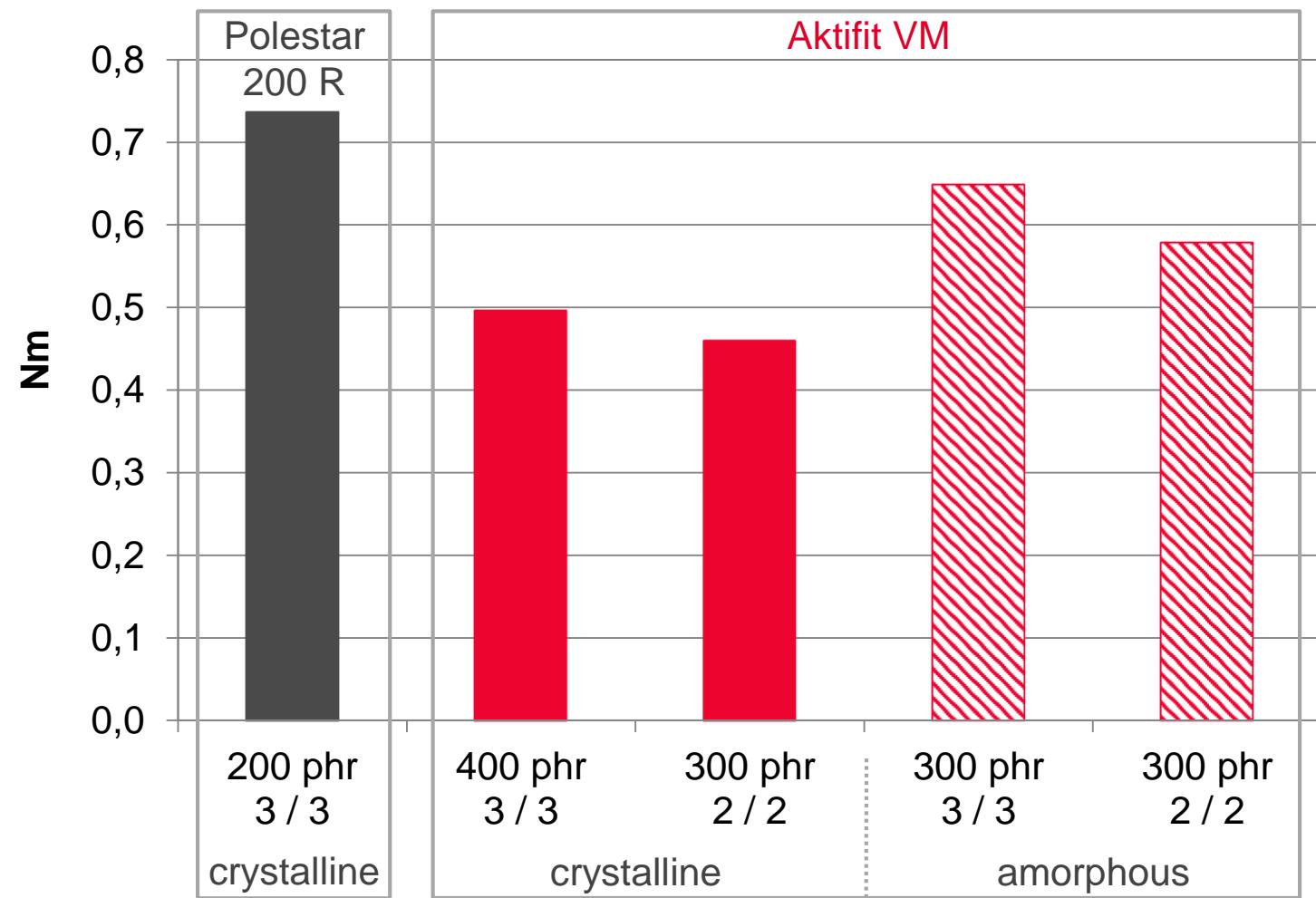
EXPERIMENTAL

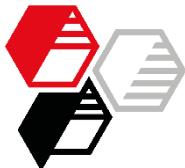
## RESULTS

Formulation optimization

• Rheology

SUMMARY





# Conversion Time $t_{90}$

DIN 53 529-A3, 180 °C, 0,2° deflection – Göttfert Elastograph

INTRODUCTION

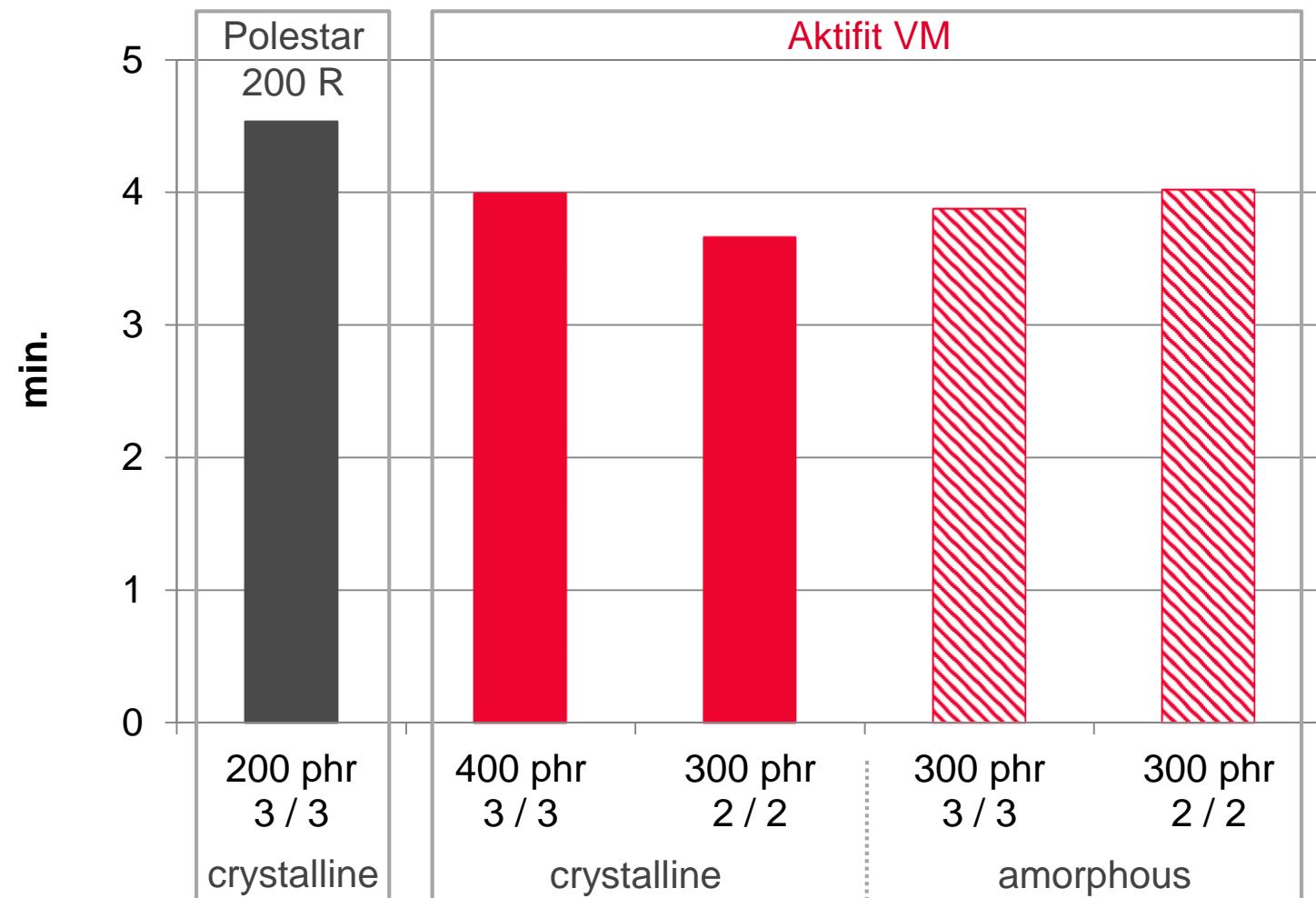
EXPERIMENTAL

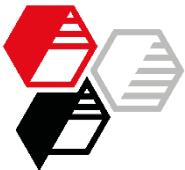
## RESULTS

Formulation optimization

- Rheology

SUMMARY





# Hardness

DIN 53 505-A, piled-up S2-dumbbells

— RAL GZ 716/1, B II

INTRODUCTION

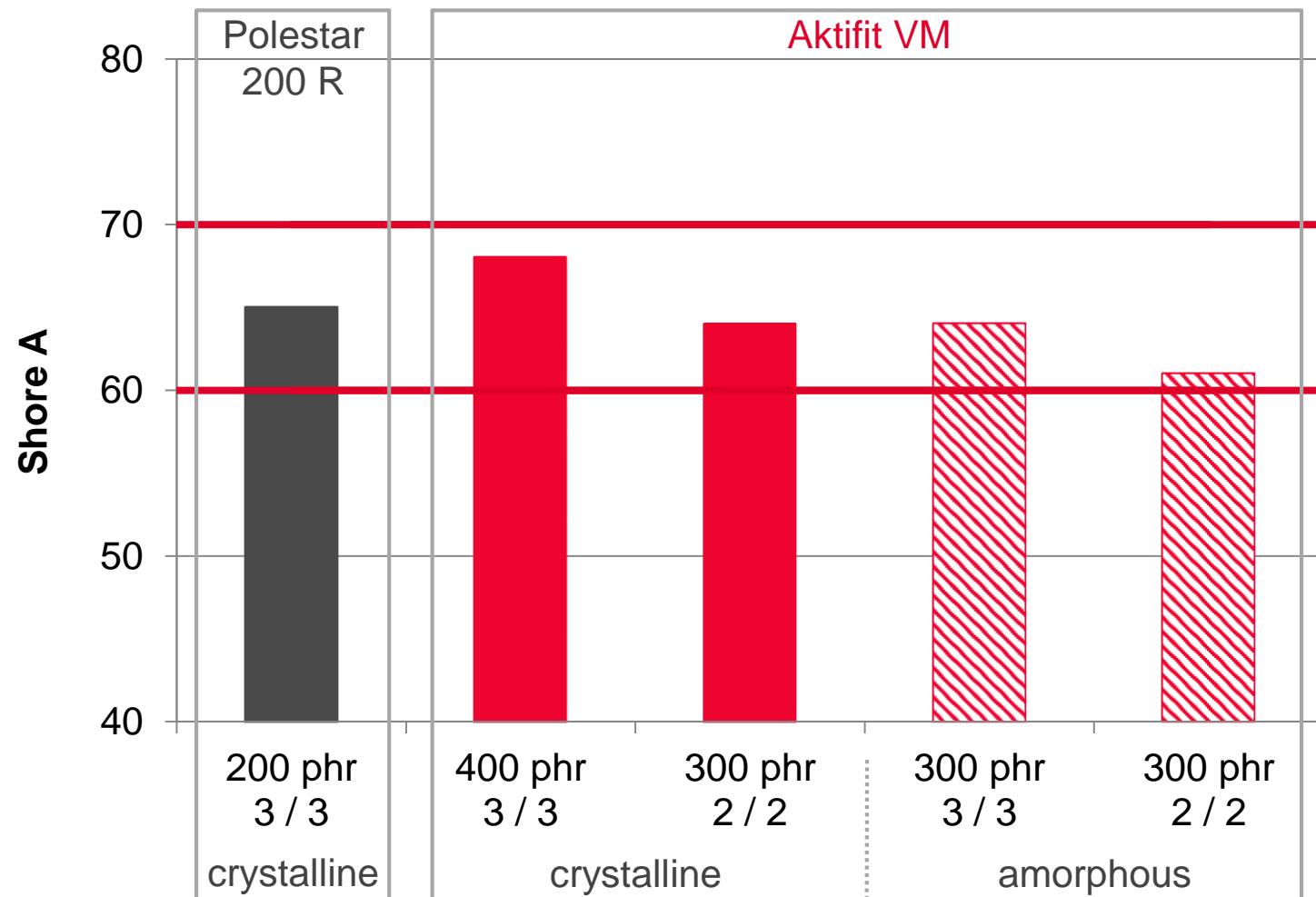
EXPERIMENTAL

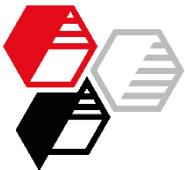
## RESULTS

Formulation optimization

- Base properties

SUMMARY





# Tensile Strength

DIN 53 504, S2

— RAL GZ 716/1, B II

INTRODUCTION

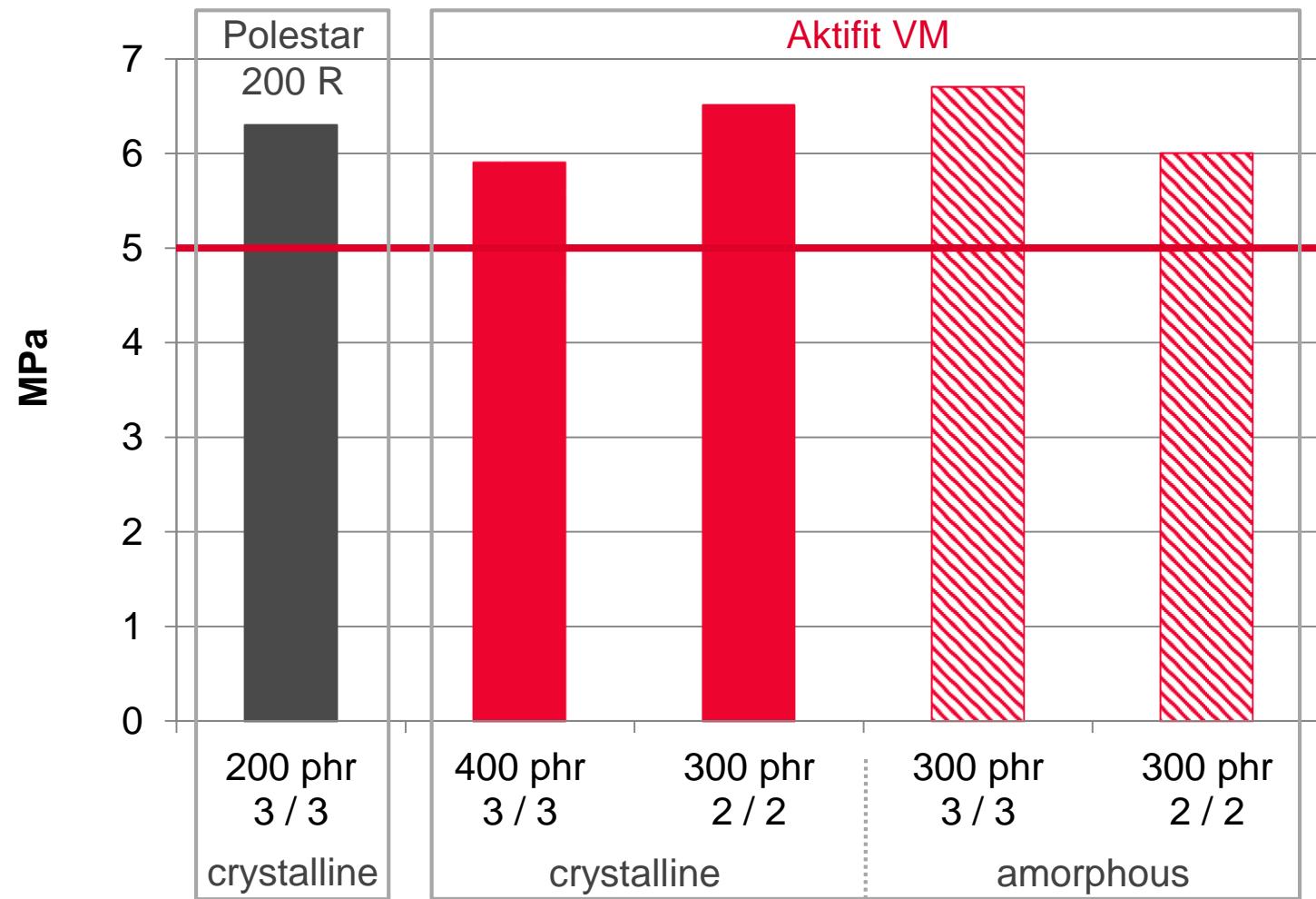
EXPERIMENTAL

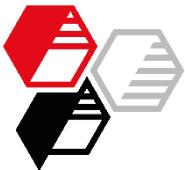
## RESULTS

Formulation optimization

- Base properties

SUMMARY





# Elongation at Break

DIN 53 504, S2

— RAL GZ 716/1, B II

INTRODUCTION

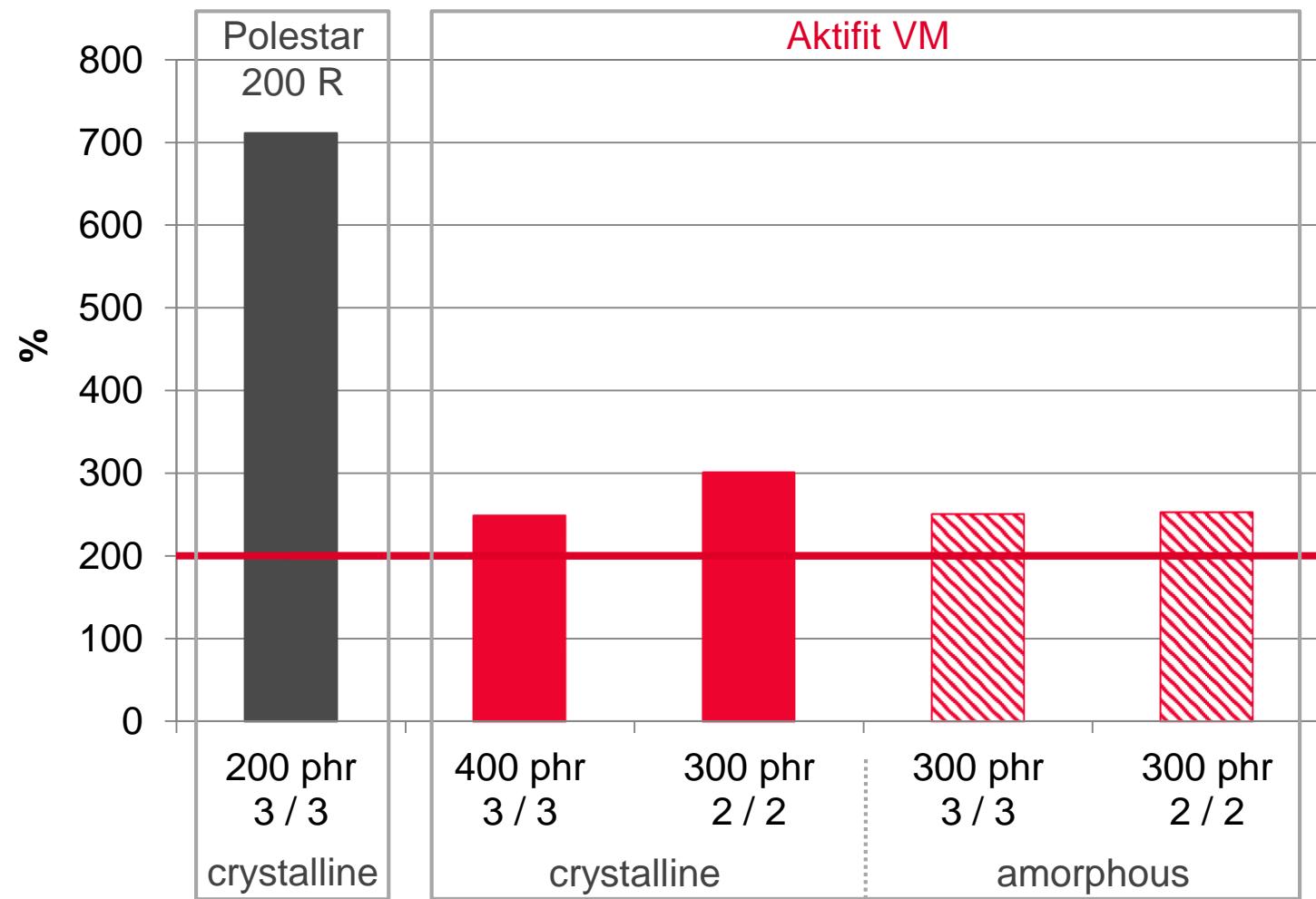
EXPERIMENTAL

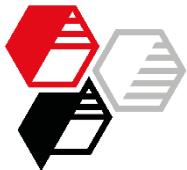
## RESULTS

Formulation optimization

- Base properties

SUMMARY





# Modulus 100 %

DIN 53 504, S2

INTRODUCTION

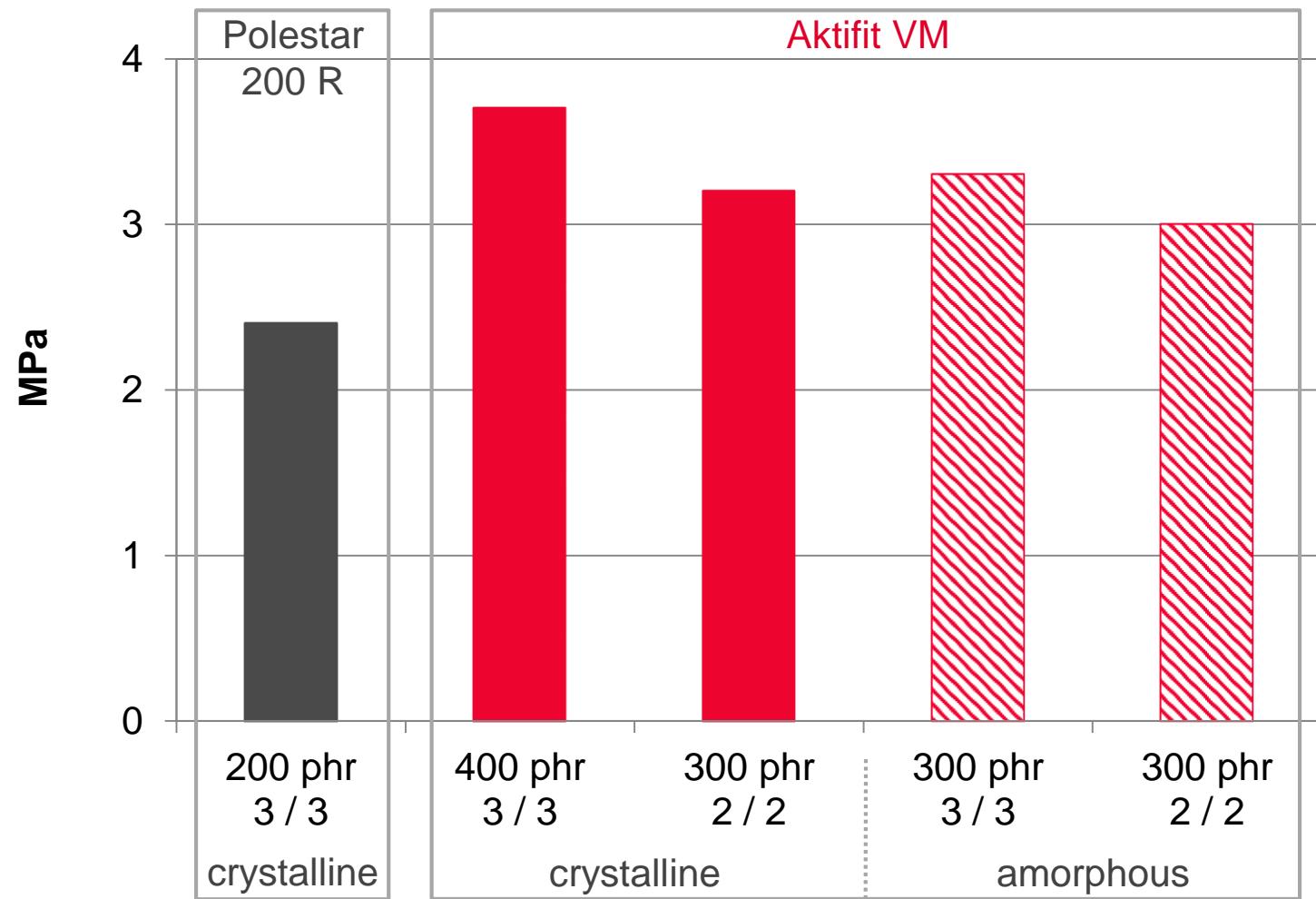
EXPERIMENTAL

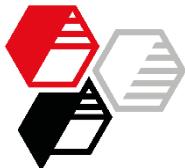
## RESULTS

Formulation optimization

- Base properties

SUMMARY





# Compression Set

## 24 h / 125 °C

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INTRODUCTION

EXPERIMENTAL

### RESULTS

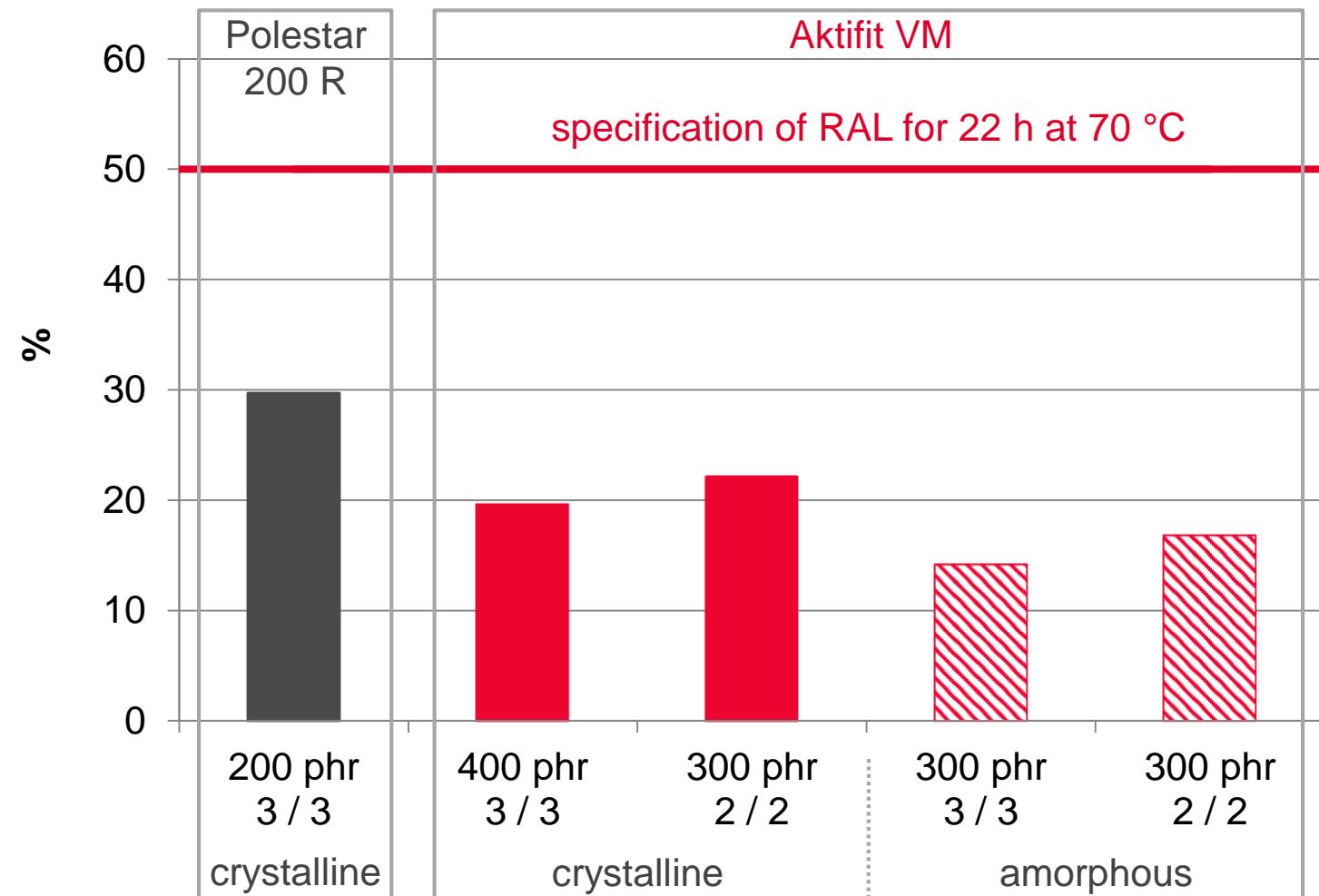
Formulation optimization

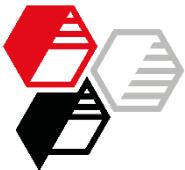
- Base properties

SUMMARY

DIN ISO 815-1 B, cooling method A , 25 % defl.

— RAL GZ 716/1, B II





# Compression Set

## 22 h / 23 °C

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INTRODUCTION

EXPERIMENTAL

### RESULTS

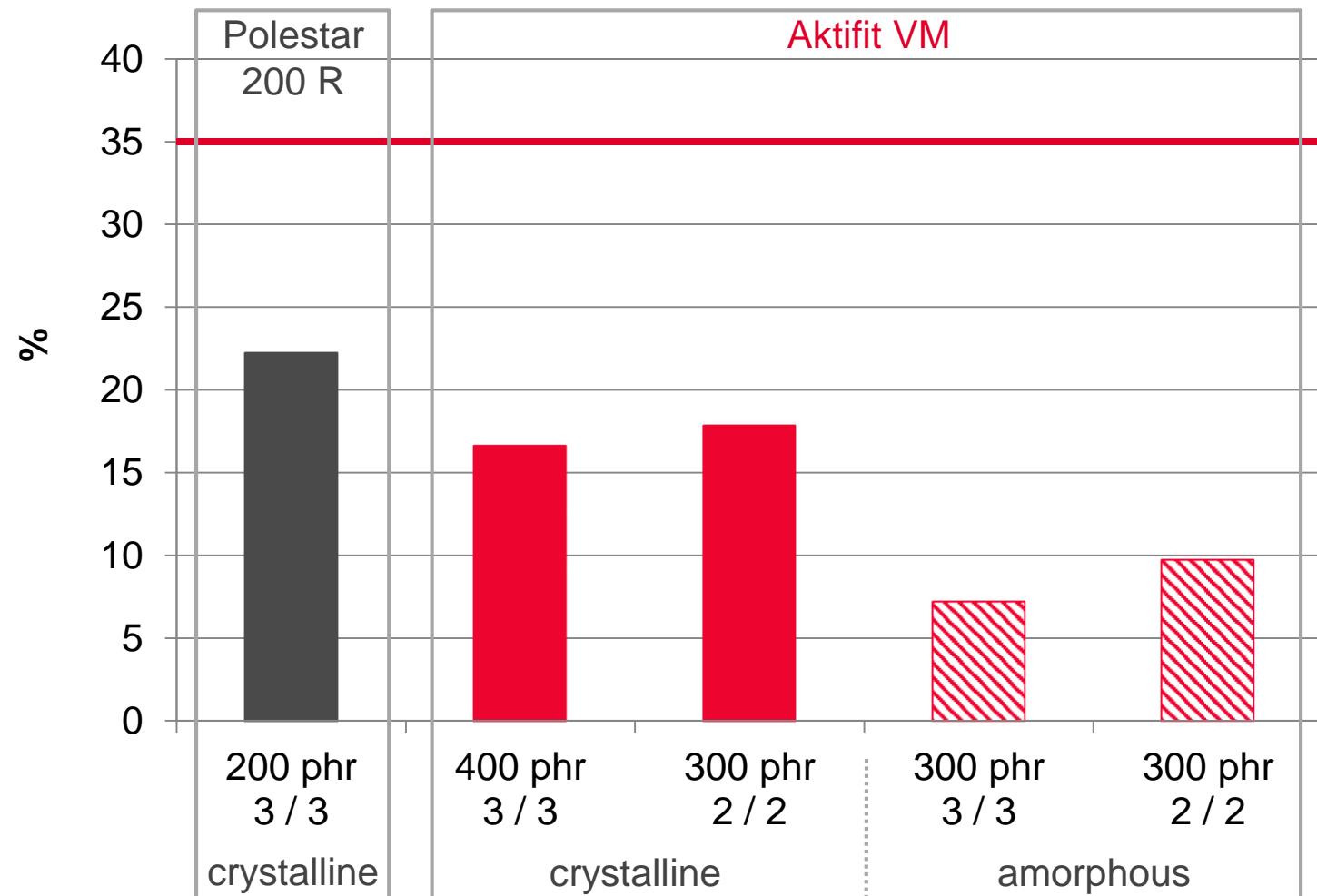
Formulation optimization

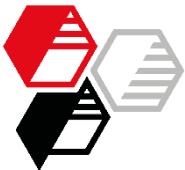
- Base properties

SUMMARY

DIN ISO 815-1 B, cooling method A , 25 % defl.

— RAL GZ 716/1, B II





# Low Temperature Exposure Change of Hardness

**HOFFMANN**  
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INTRODUCTION

EXPERIMENTAL

## RESULTS

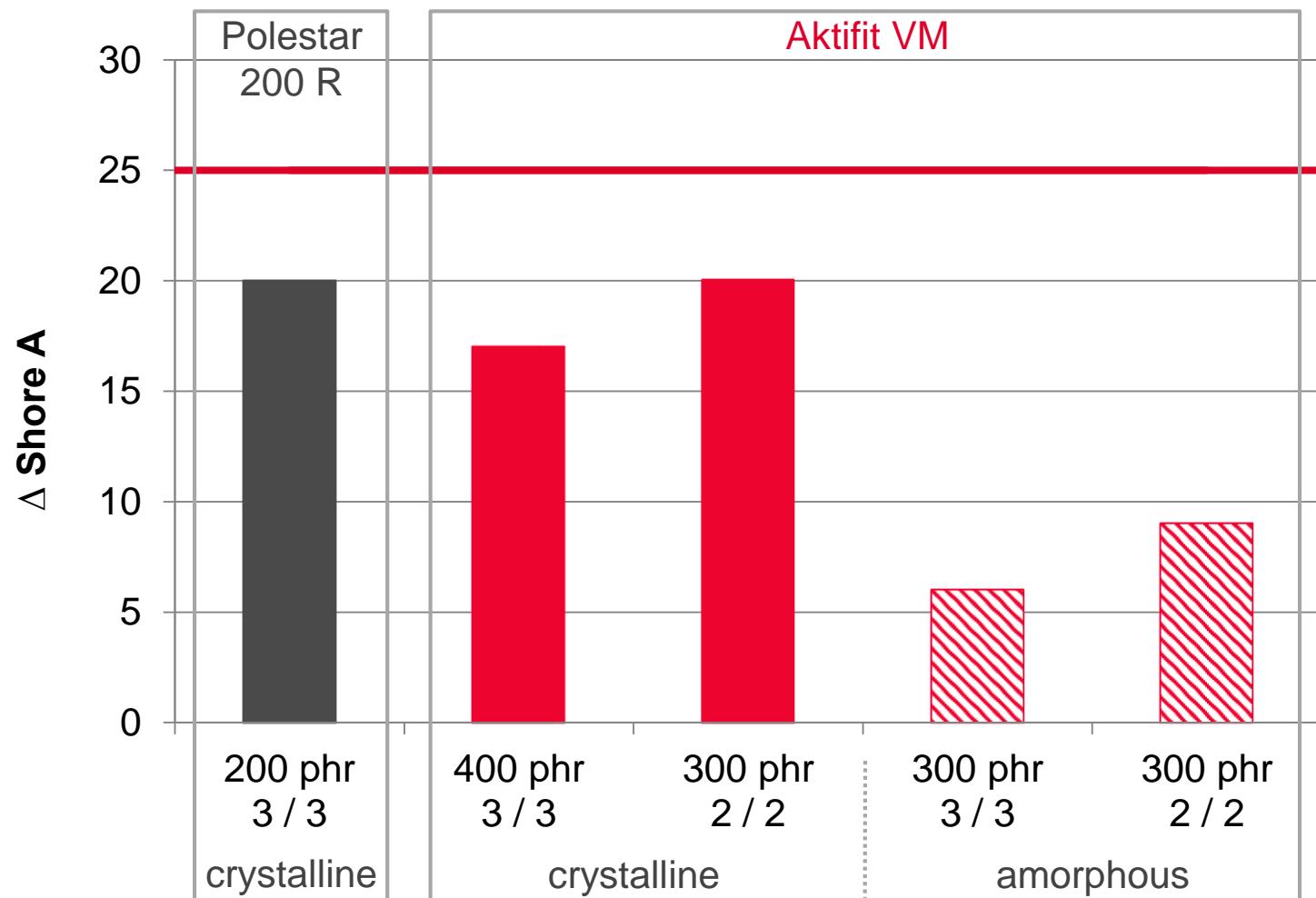
Formulation optimization

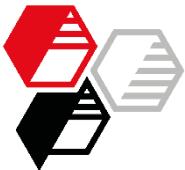
- Base properties

SUMMARY

22 h / -10 °C

— RAL GZ 716/1, B II





# Hot Air Aging Change of Hardness

**HOFFMANN**  
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DIN 53 508, 168 h / 100 °C

— RAL GZ 716/1, B II

INTRODUCTION

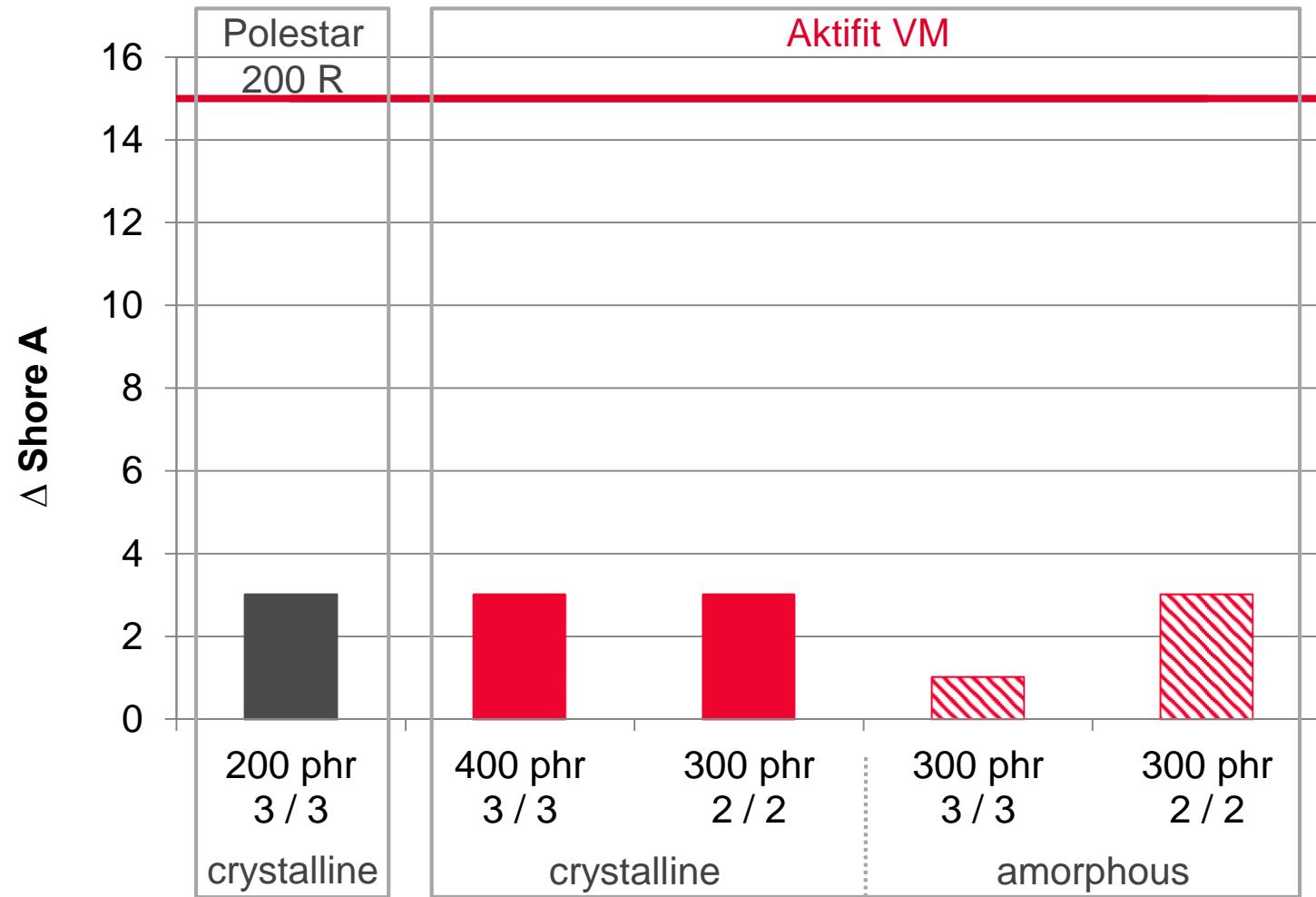
EXPERIMENTAL

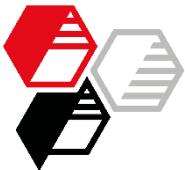
## RESULTS

Formulation optimization

- Base properties

SUMMARY





# Hot Air Aging Change of Tensile Strength

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INTRODUCTION

EXPERIMENTAL

## RESULTS

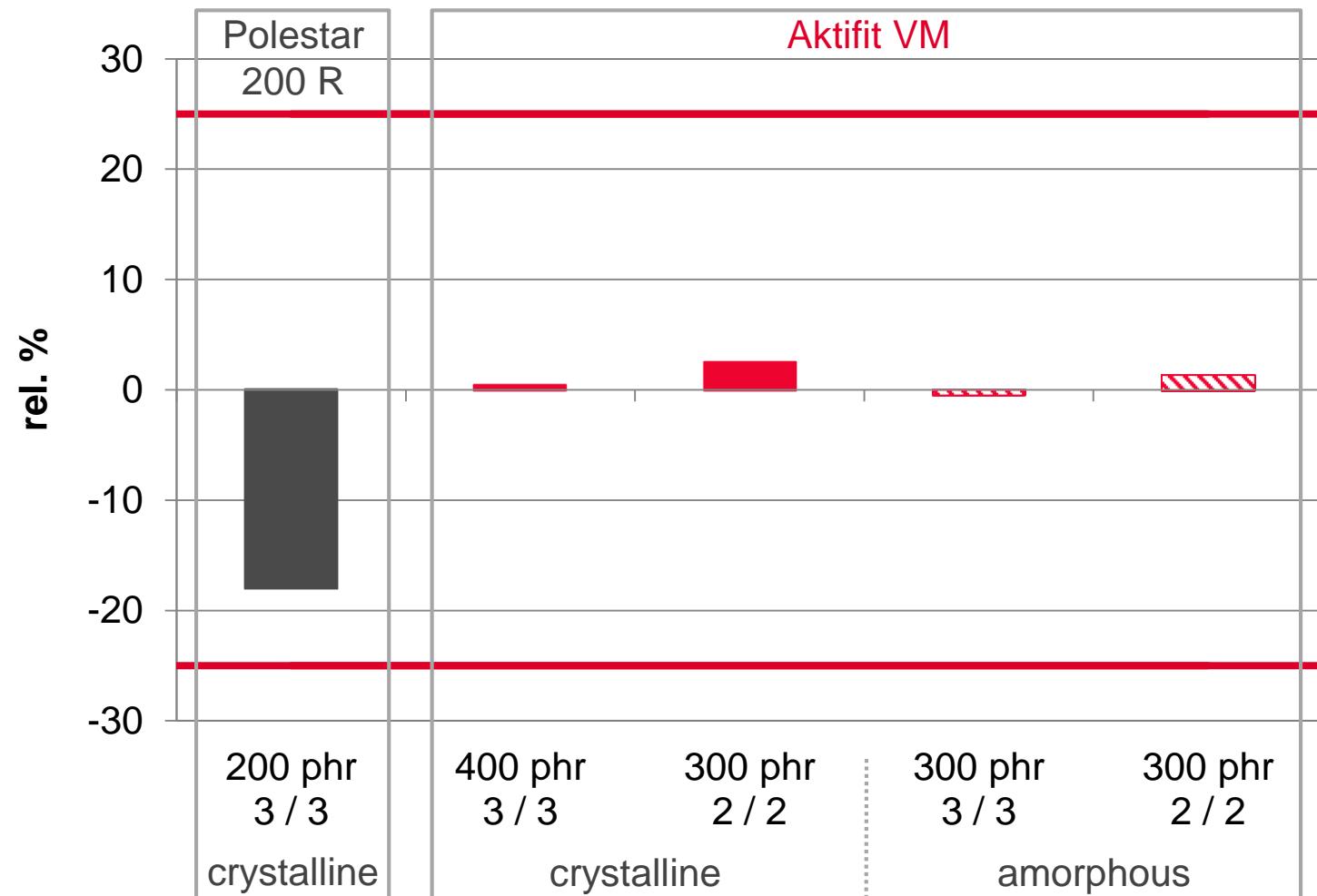
Formulation optimization

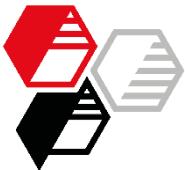
- Base properties

SUMMARY

DIN 53 508, 168 h / 100 °C

— RAL GZ 716/1, B II





# Hot Air Aging Remaining Elongation at B.

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INTRODUCTION

EXPERIMENTAL

## RESULTS

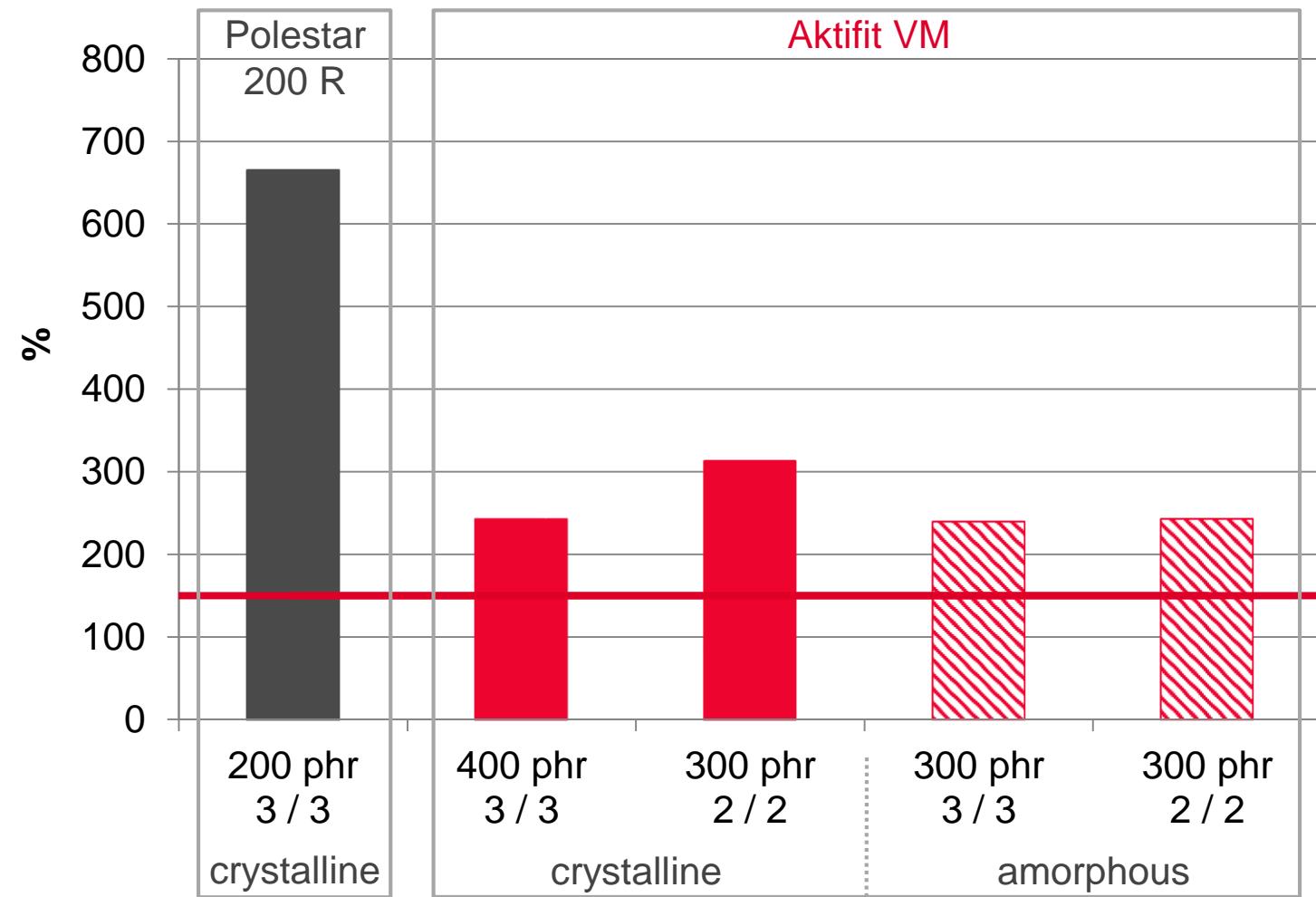
Formulation optimization

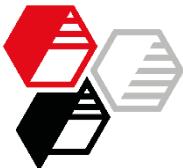
- Base properties

SUMMARY

DIN 53 504, S2

— RAL GZ 716/1, B II





# Color Values

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INTRODUCTION

EXPERIMENTAL

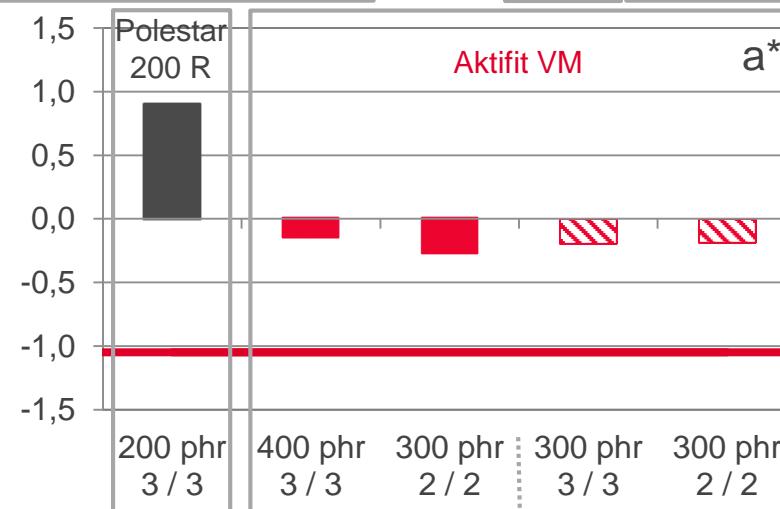
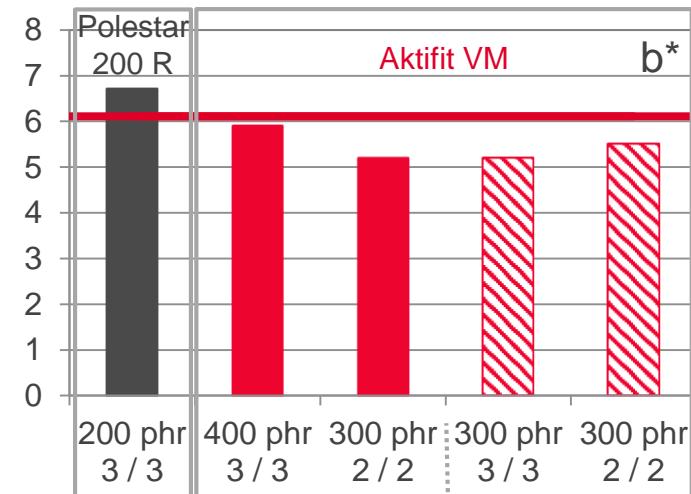
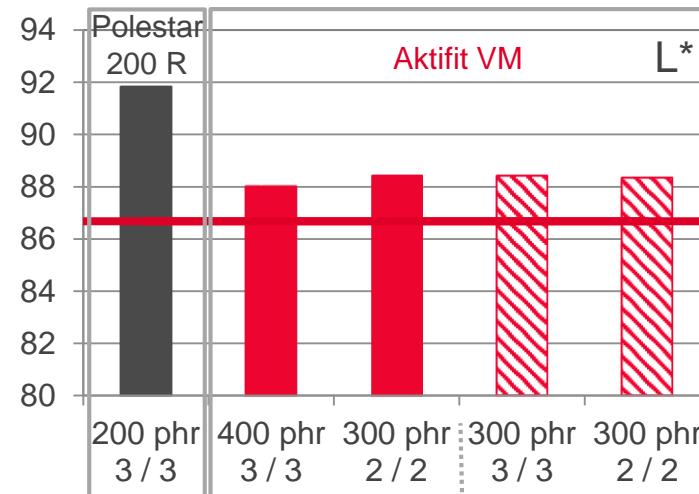
## RESULTS

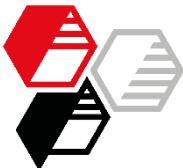
Formulation optimization

• Color

SUMMARY

ISO 7724





# Garvey Extrusion

ASTM D 2230, rating 4 4 4 4

INTRODUCTION

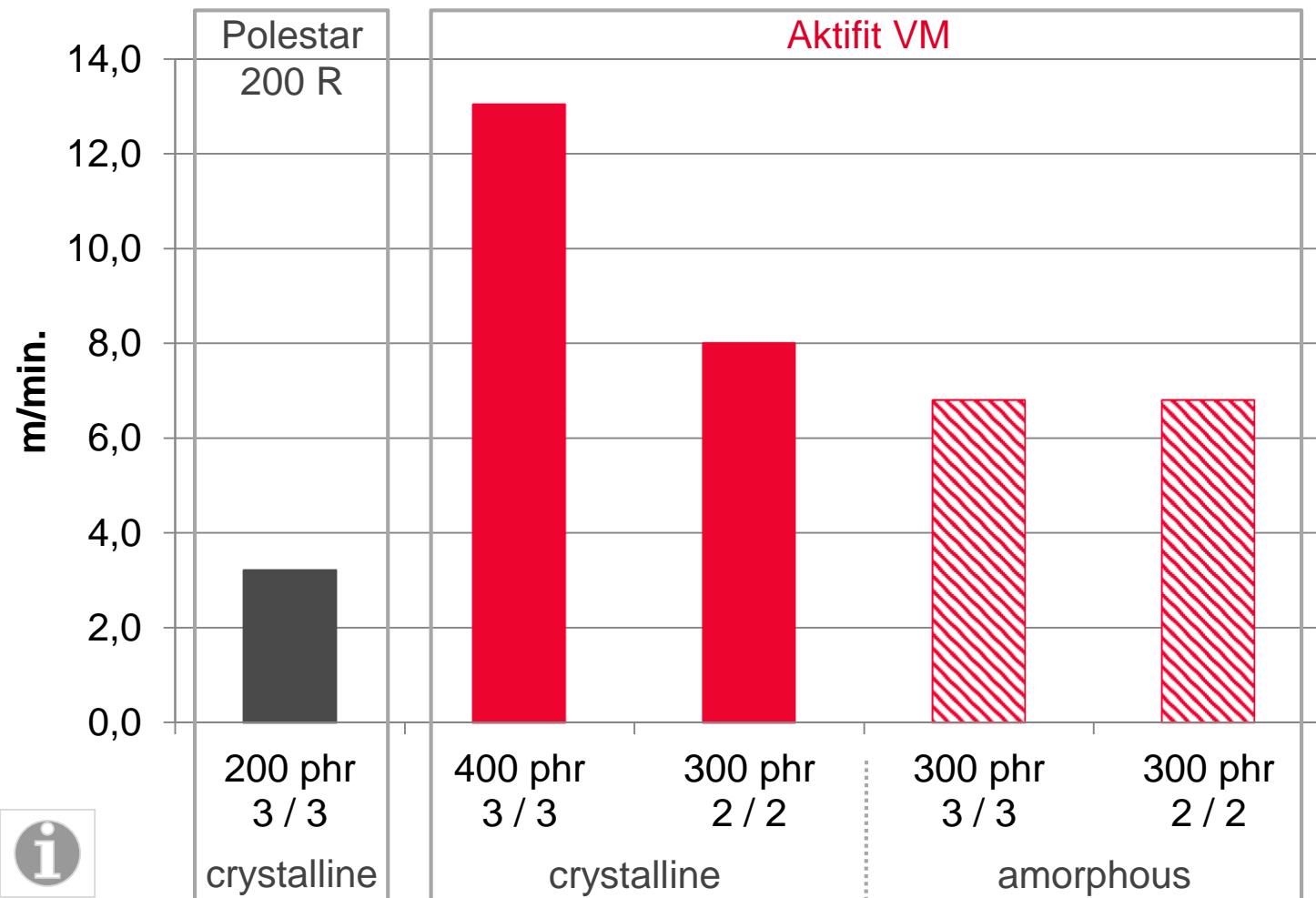
EXPERIMENTAL

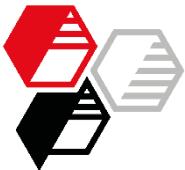
## RESULTS

Formulation optimization

• Extrusion

SUMMARY





# Cost Index

Volume related, Germany 2011

INTRODUCTION

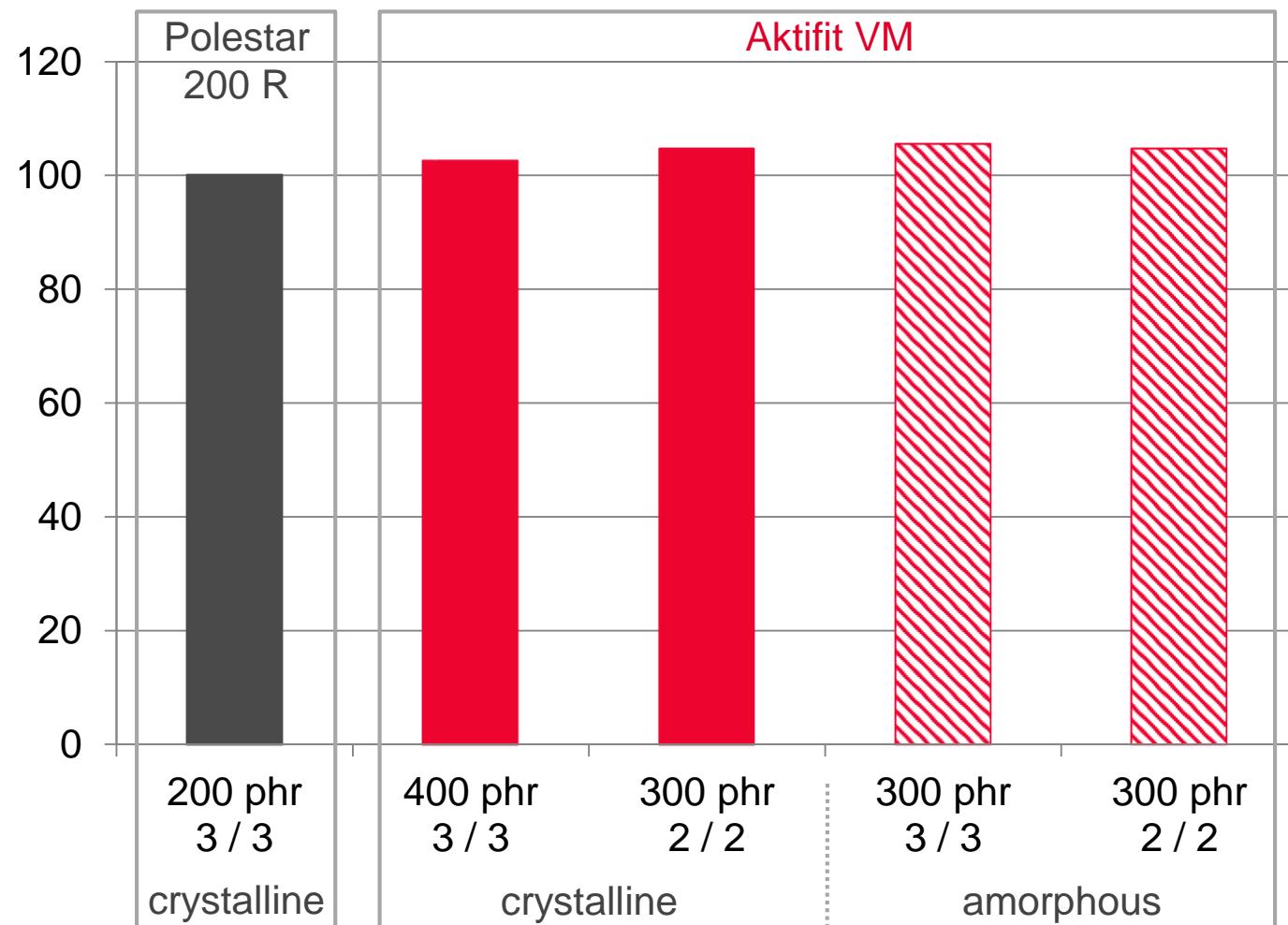
EXPERIMENTAL

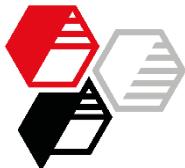
## RESULTS

Formulation optimization

- Compound costs

SUMMARY





## Summary Part 2

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INTRODUCTION

EXPERIMENTAL

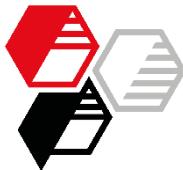
RESULTS

### SUMMARY

Formulation optimization

**Aktifit VM** 300 phr, peroxide combination 2 / 2, both polymers

- ✓ compared to reference compound with Polestar 200 R improved mechanical properties, especially compression set
- ✓ higher approach to color values of RAL 9002 with lower red and yellow tint than reference compound with Polestar 200 R
- ✓ in comparison to reference compound with Polestar 200 R doubled haul-off speed
- ✓ increased volume related compound costs only of approx. 5 % in comparison to reference compound with Polestar 200 R



## Summary Part 2

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INTRODUCTION

EXPERIMENTAL

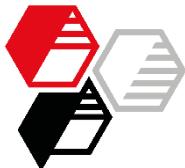
RESULTS

SUMMARY

Formulation optimization

Aktifit VM 300 phr, peroxide combination 3 / 3, amorphous polymer

- ✓ best compression set
- ✓ lowest change of hardness after storage at low temperature
- ✓ higher approach to color values of RAL 9002 with lower red and yellow tint than reference compound with Polestar 200 R
- ✓ in comparison to reference compound with Polestar 200 R doubled haul-off speed
- ✓ increased volume related compound costs only of approx. 5 % in comparison to reference compound with Polestar 200 R



## Summary Part 2

INTRODUCTION

EXPERIMENTAL

RESULTS

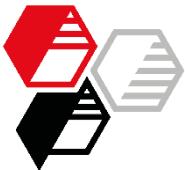
SUMMARY

Formulation optimization

Aktifit VM 400 phr, peroxide combination 3 / 3, crystalline polymer

- ✓ better compression set than reference compound with Polestar 200 R
- ✓ slight improvement of change of hardness after storage at low temperature compared to reference compound with Polestar 200 R
- ✓ higher approach to color values of RAL 9002 with lower red and yellow tint than reference compound with Polestar 200 R despite of doubled filler loading
- ✓ haul-off speed increase fourfold compared to reference compound with Polestar 200 R
- ✓ increased volume related compound costs only of approx. 2.5 % in comparison to reference compound with Polestar 200 R





## Final Conclusion

INTRODUCTION

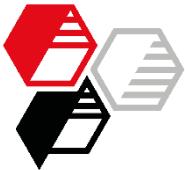
EXPERIMENTAL

RESULTS

SUMMARY

For the replacement of Polestar 200 R by **Aktifit VM** speak:

- ✓ improved mechanical properties
  - ✓ higher color neutrality
  - ✓ markedly improved extrusion behavior
- **Calcined Neuburg Siliceous Earth is excellently suitable for the application in grey-white colored building profiles.**



## We supply material for good ideas!

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