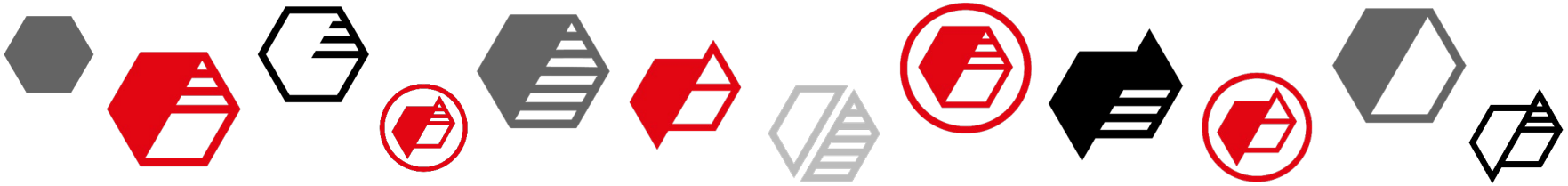

# Calcined Neuburg Siliceous Earth in Polypropylene (PP)

**Author: Petra Zehnder**

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We supply material for good ideas

# Contents

- Introduction
- Experimental
- Results
- Summary
- Appendix: summary tables

# Status Quo



- Polypropylene (PP) as a plastic which is produced in huge quantities goes into a multitude of application areas. Typical uses for injection molded articles are found in households and in the electricity sector, but the biggest amount in car body parts and molded products for car interiors.
- PP here meets many essential requirements for a technical plastic along with offering a favorable price-performance ratio.
- As a typical filler for PP talc is widely used.
- Important properties along with easy processing are balanced stiffness and toughness.
- In addition, for automotive interiors scratch resistance is of high importance.

# Objective



Assessment of the performance of

Calcined Neuburg Siliceous Earth

versus

**an especially for good scratch resistance recommended talc**

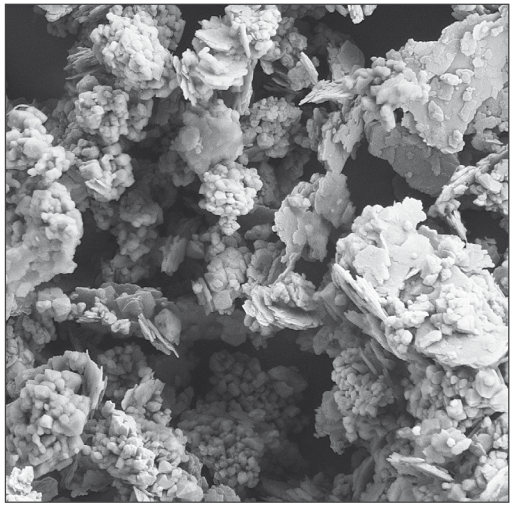
in PP copolymer and homopolymer regarding

- » Flow
- » Heat deflection
- » Mechanical properties
  - Tensile test
  - Impact strength
- » Scratch resistance and resistance to intendation by writing

# Calcined Neuburg Siliceous Earth

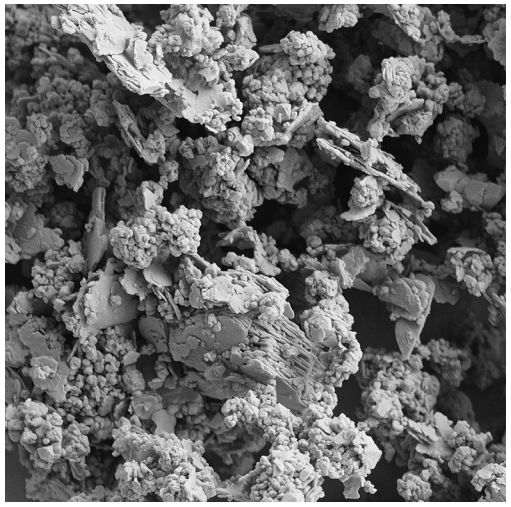


A downstream thermal process leads to the calcined products Silfit and Aktifit, based on SILLITIN Z 86.



Neuburg Siliceous Earth

**Calcination Process**



Calcined Neuburg Siliceous Earth

Additional application benefits, as well as the removing of crystal water included in the kaolinite. The silica part remains inert.

# Fillers and Characteristics



| | | Talc ultrafine* | Calcined Neuburg Siliceous Earth | |
|---|---|---|---|---|
| | | | Silfit Z 91 | Aktifit AM |
| Color value L* (CIELAB) | | 90.2 | 95.1 | 95.1 |
| Color value a* (CIELAB) | | -0.5 | -0.2 | -0.2 |
| Color value b* (CIELAB) | | 0.3 | 1.0 | 1.0 |
| Particle size $d_{50}$ | [µm] | 3.6 | 1.9 | 1.9 |
| Particle size $d_{97}$ | [µm] | 11 | 10 | 10 |
| Oil absorption | [g/100g] | 47 | 60 | 61 |
| Specific surface area BET | [m²/g] | 12 | 7.4 | 6.6 |
| Sieve residue > 40 µm | [mg/kg] | 2 | 8 | 21 |
| Functionalization | | none | none | Amino |

*manufacturer information: $d_{50}$: 1 µm, $d_{95}$: 3 µm;
special grade for good impact strength and scratch resistance

# Compounding
# Injection Molding

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| | | |
|---|---|---|
| PP grade | Copolymer: | MFR 16 g/10 min |
| | Homopolymer: | MFR 17 g/10 min |
| Filler content | PP Copolymer: | 40 % and 20 % |
| | PP Homopolymer: | 40 % |
| Compounding | Twin screw extruder ZSK 30 | |
| | Melt temperature: | approx. 250 °C |
| Injection molding of test specimens acc. to ISO 1874 | Mold acc. to ISO 294 | |
| | Mold temperature: | 40 °C |
| | Melt temperature: | 230 °C |

# Selection

| Copolymer | Homopolymer |
| --- | --- |
| • 20 % Filler | |
| • 40 % Filler | • 40 % Filler |

*forward to summary*

# Formulations PP Copo 20 % Filler

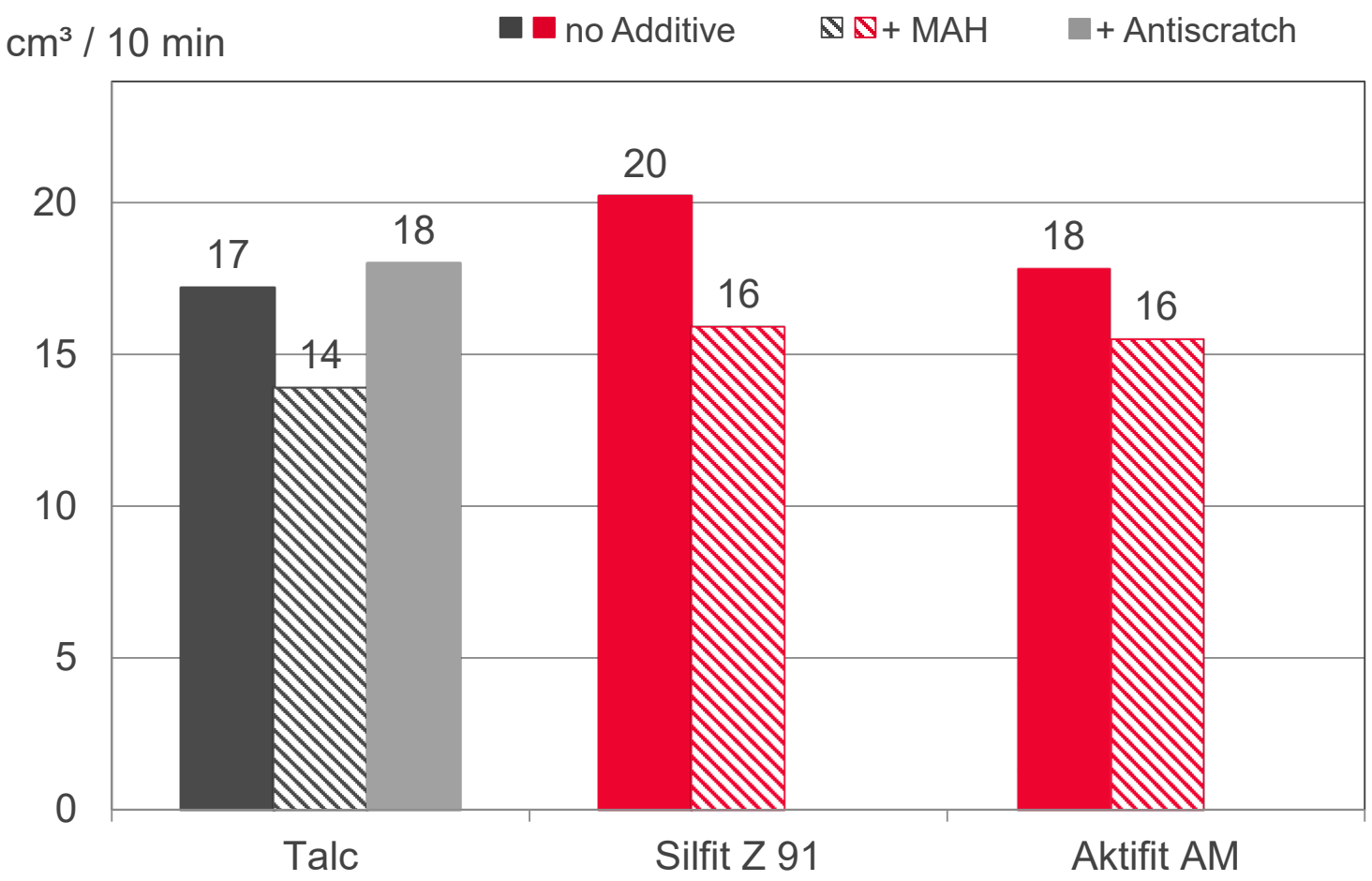


| | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|
| | --- | MAH | Anti-scratch | --- | MAH | --- | MAH |
| **PP Copolymer** MFR 16 g/10 min (230 °C, 2.16 kg) | 77.7 | 74.7 | 76.7 | 77.7 | 74.7 | 77.7 | 74.7 |
| **Stabilizer and black color batch** | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 |
| **Talc** ultrafine | 20 | 20 | 20 | --- | --- | --- | --- |
| **Silfit Z 91** | --- | --- | --- | 20 | 20 | --- | --- |
| **Aktifit AM** | --- | --- | --- | --- | --- | 20 | 20 |
| **Scratch additive** Siloxane based | --- | --- | 1 | --- | --- | --- | --- |
| **MAH-modified PP** (1 % MAH) | --- | 3 | --- | --- | 3 | --- | 3 |
| **Total** | **100** | **100** | **100** | **100** | **100** | **100** | **100** |

# Melt Volume-flow Rate

DIN ISO 1133; 230 °C, 2.16 kg





# Charpy
# Notched Impact Strength 23 °C

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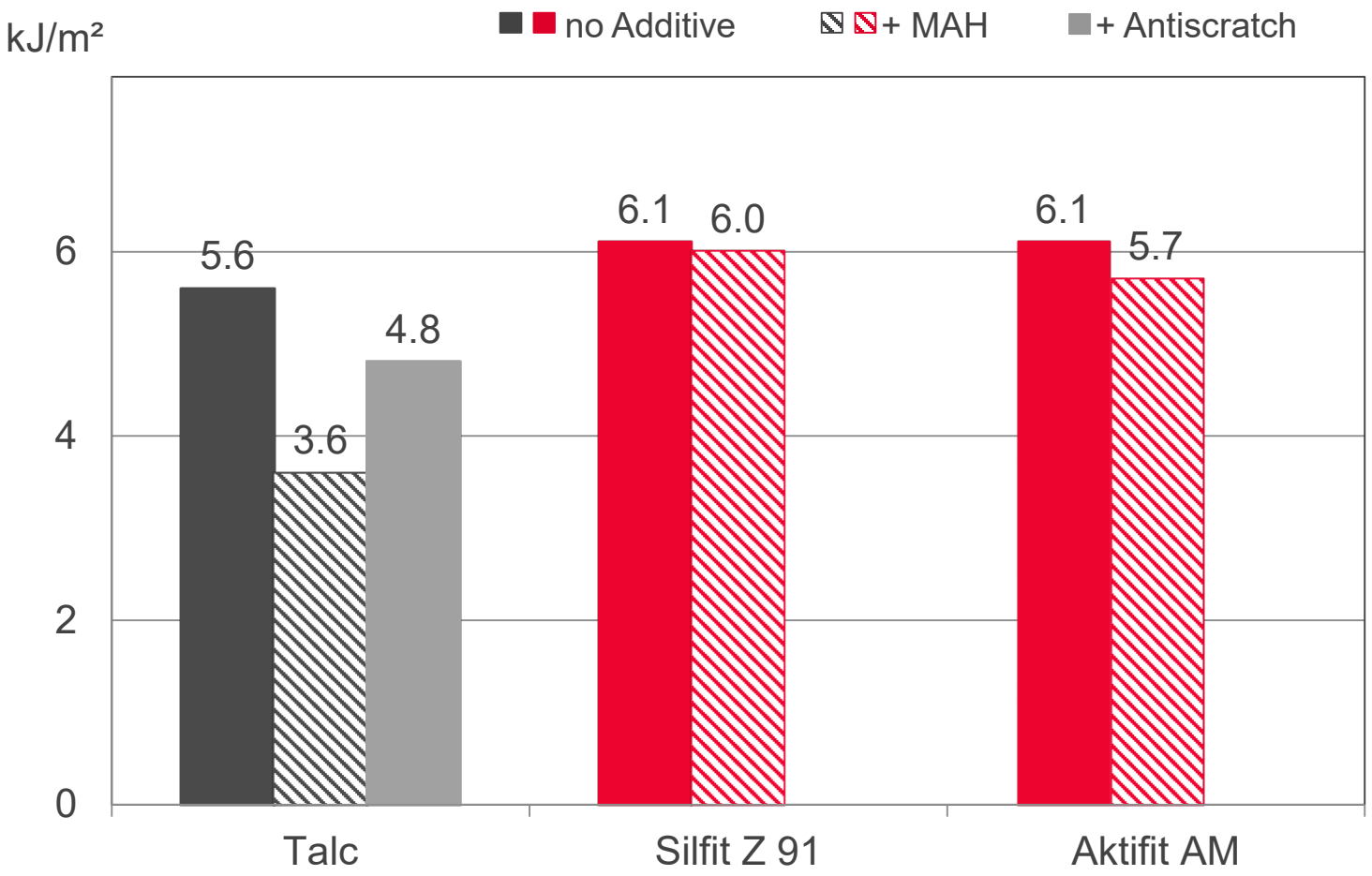
DIN EN ISO 179-1/1eA

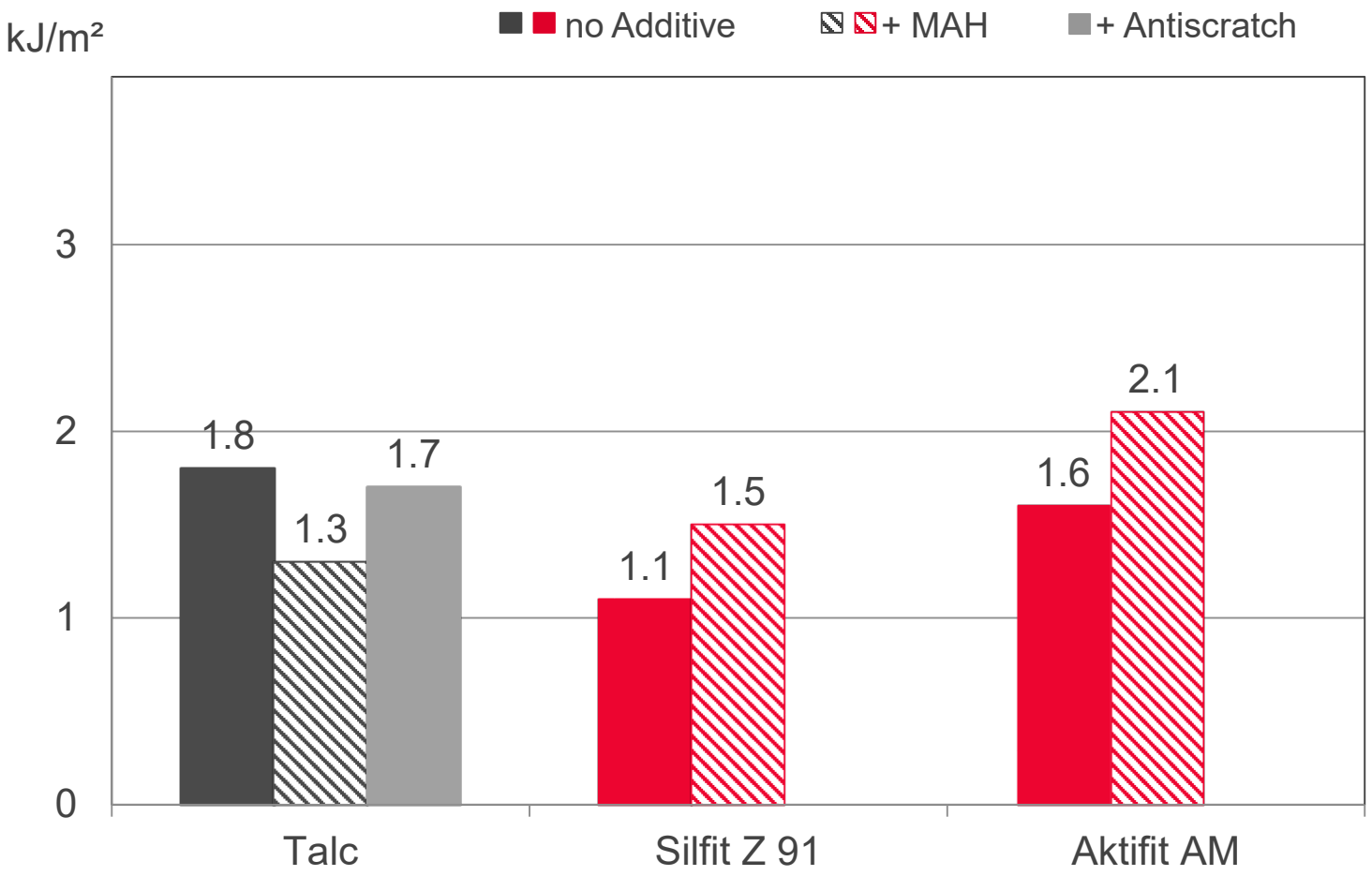




# Charpy
# Notched Impact Strength -30 °C

DIN EN ISO 179-1/1eA

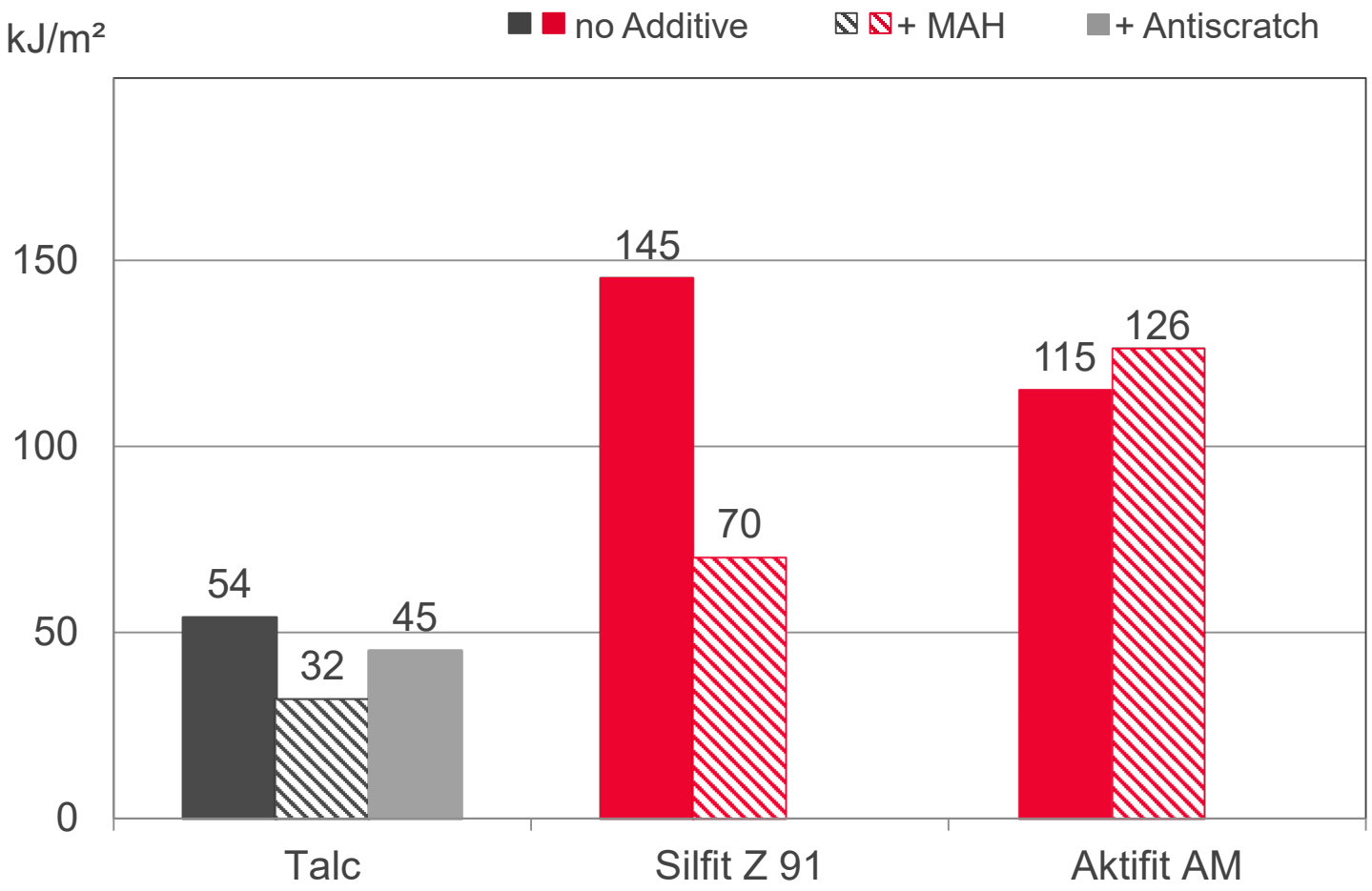




# Charpy
# Impact Strength 23 °C

DIN EN ISO 179-1/1eU

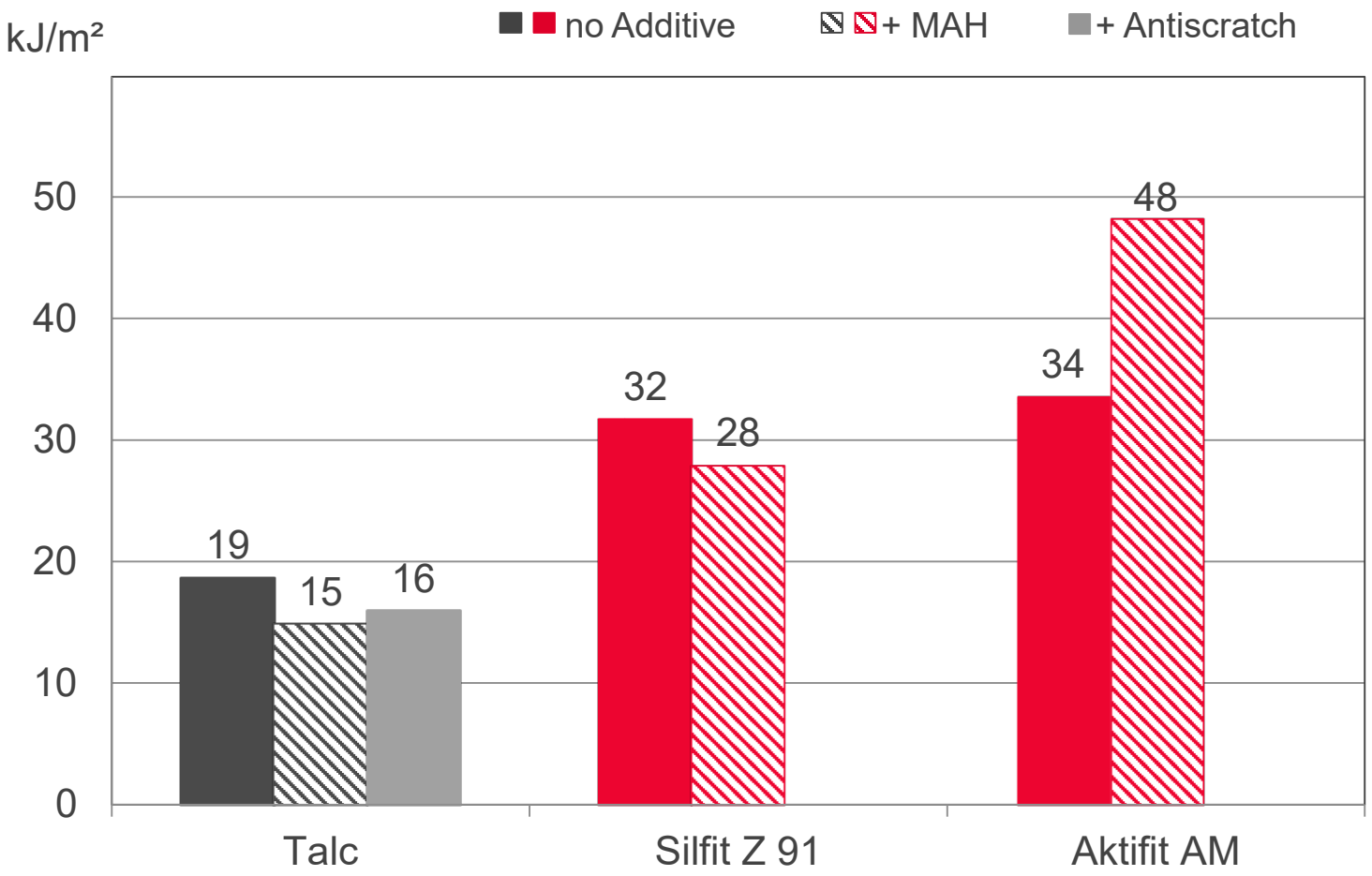




# Charpy
# Impact Strength -30 °C

DIN EN ISO 179-1/1eU

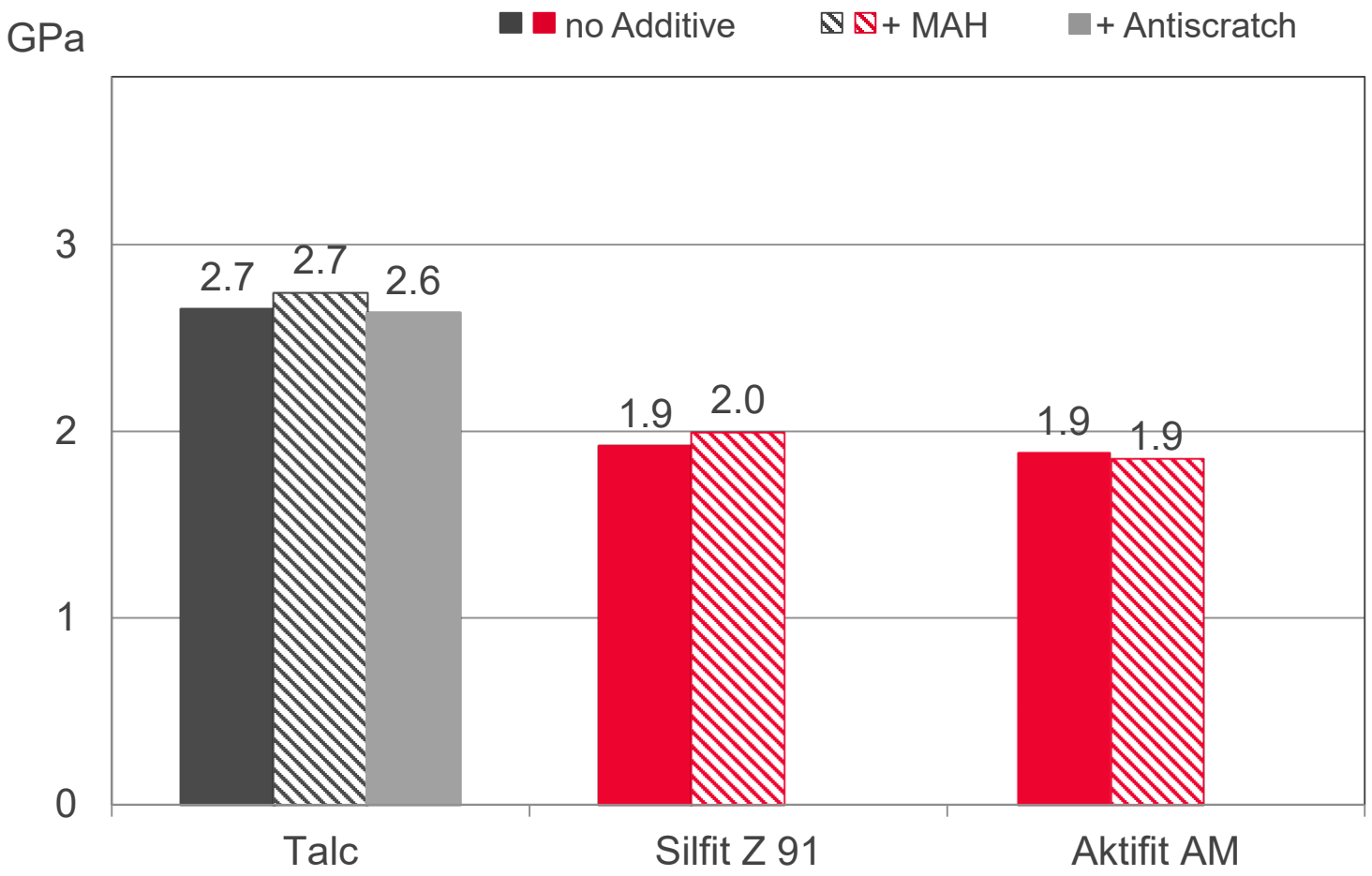




# Tensile Modulus

DIN EN ISO 527-1,-2; 1 mm/min

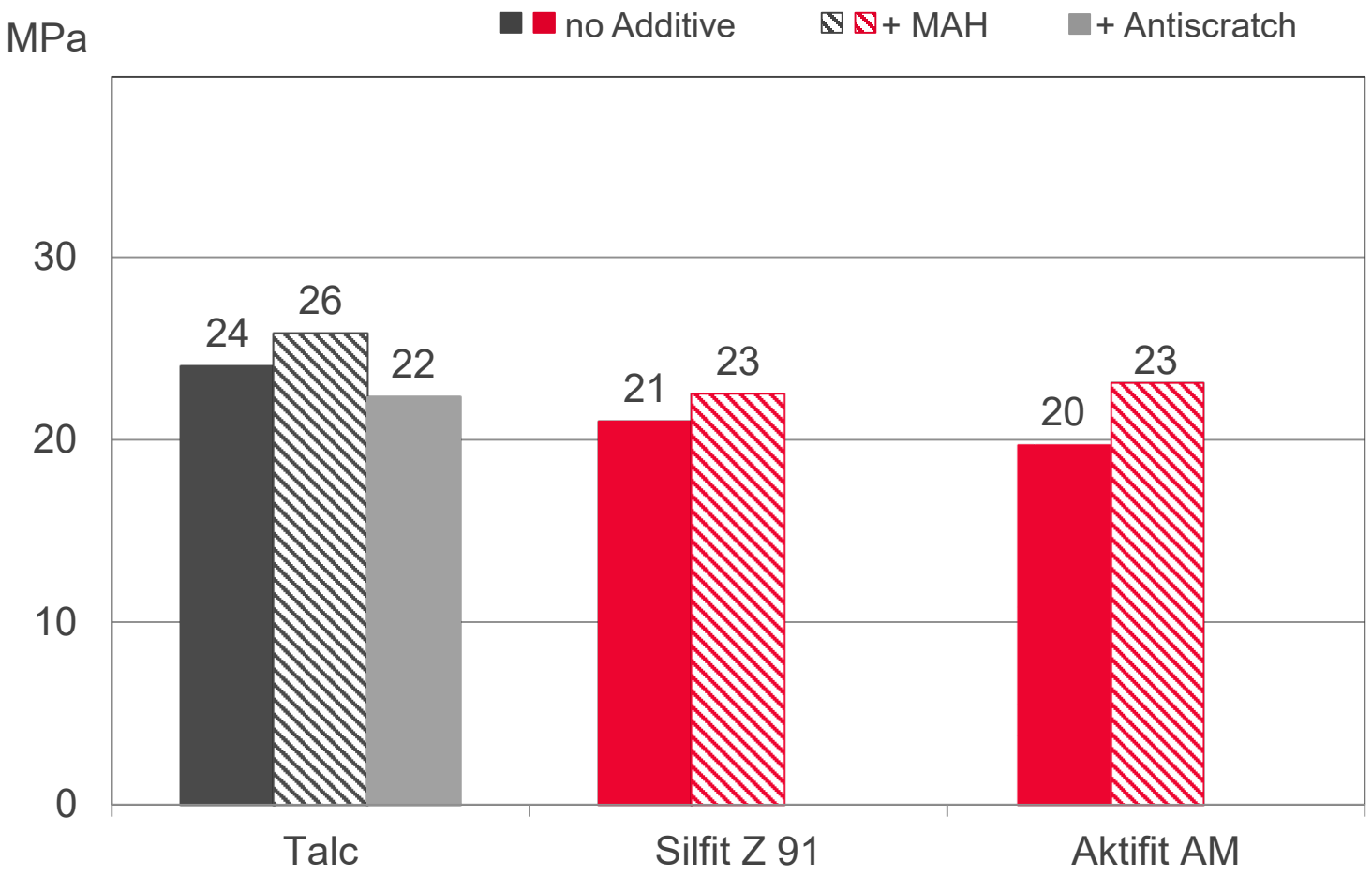




# Tensile Strength

DIN EN ISO 527-1,-2; 5 mm/min

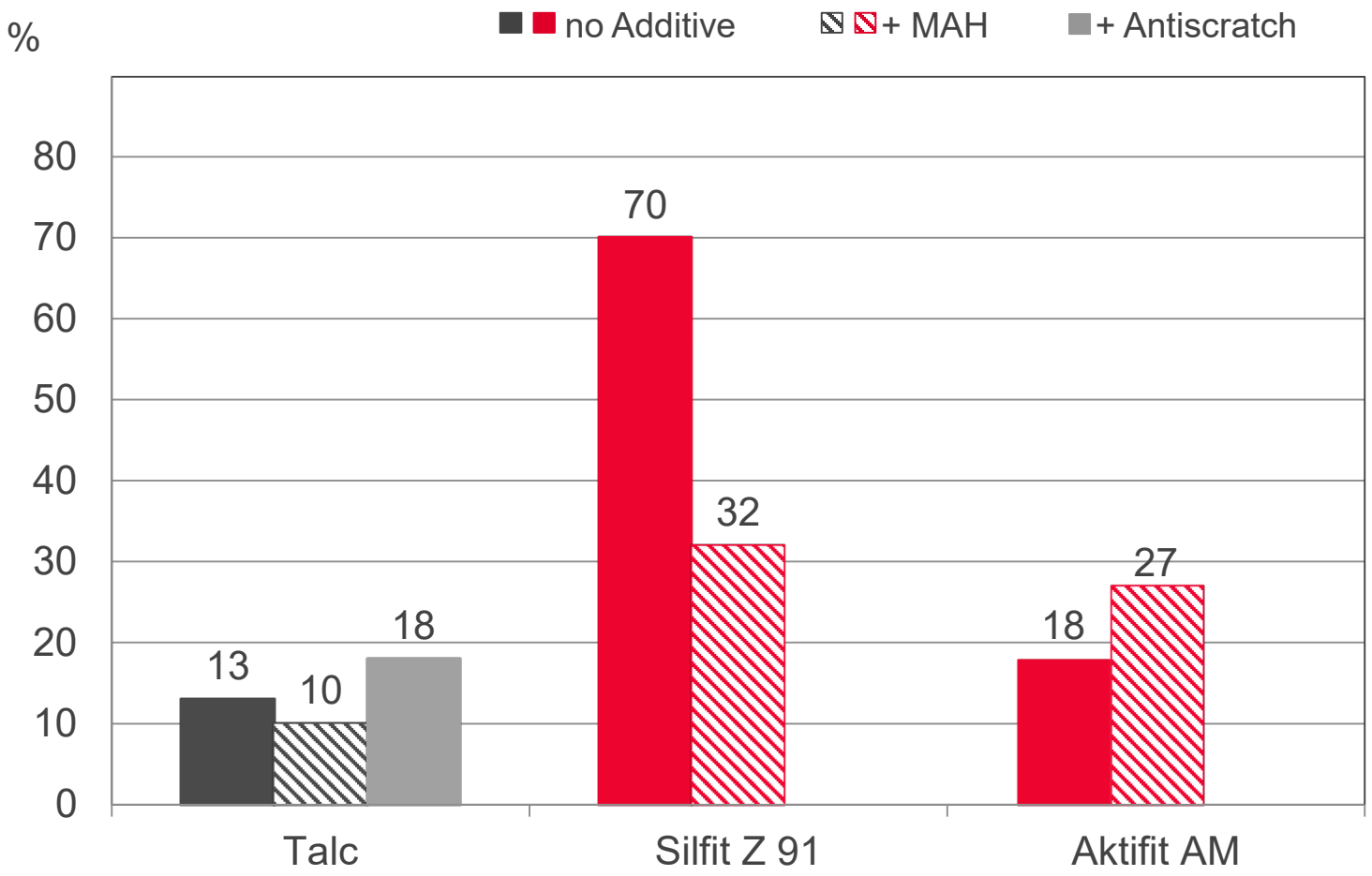




# Tensile Strain at Break

DIN EN ISO 527-1,-2; 5 mm/min

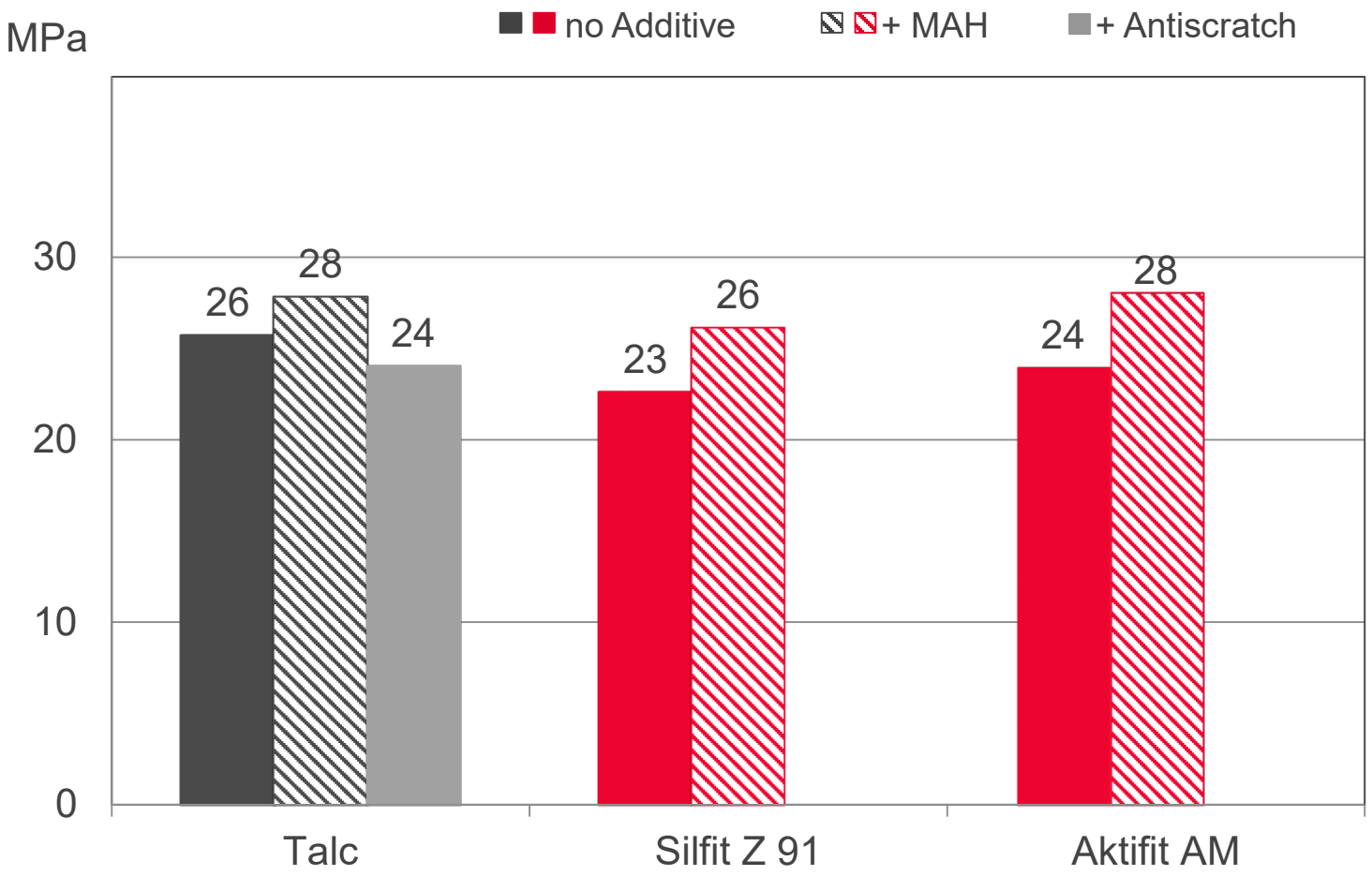




# Yield Stress

DIN EN ISO 527-1,-2; 50 mm/min

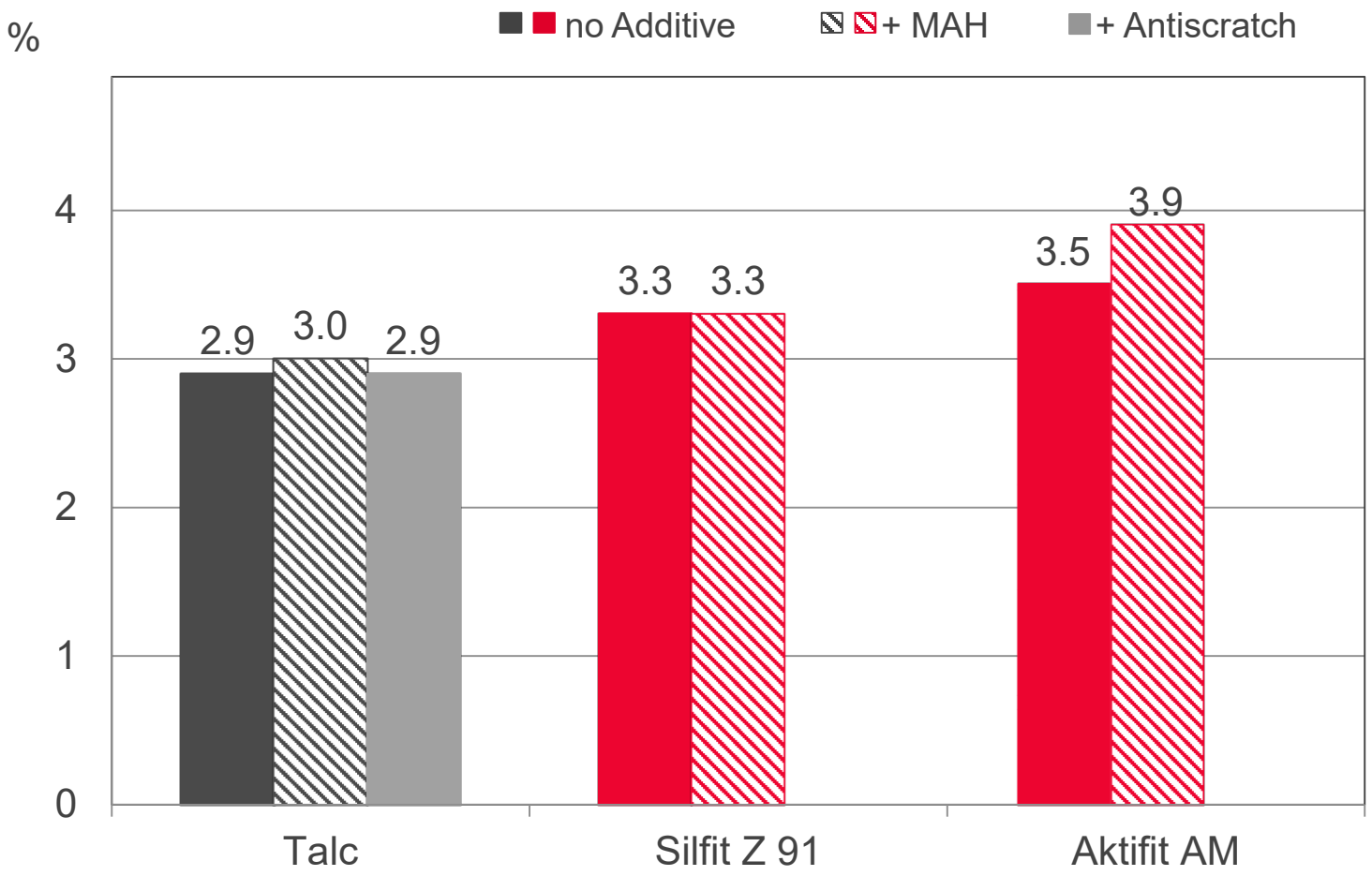




# Yield Strain

DIN EN ISO 527-1,-2; 50 mm/min

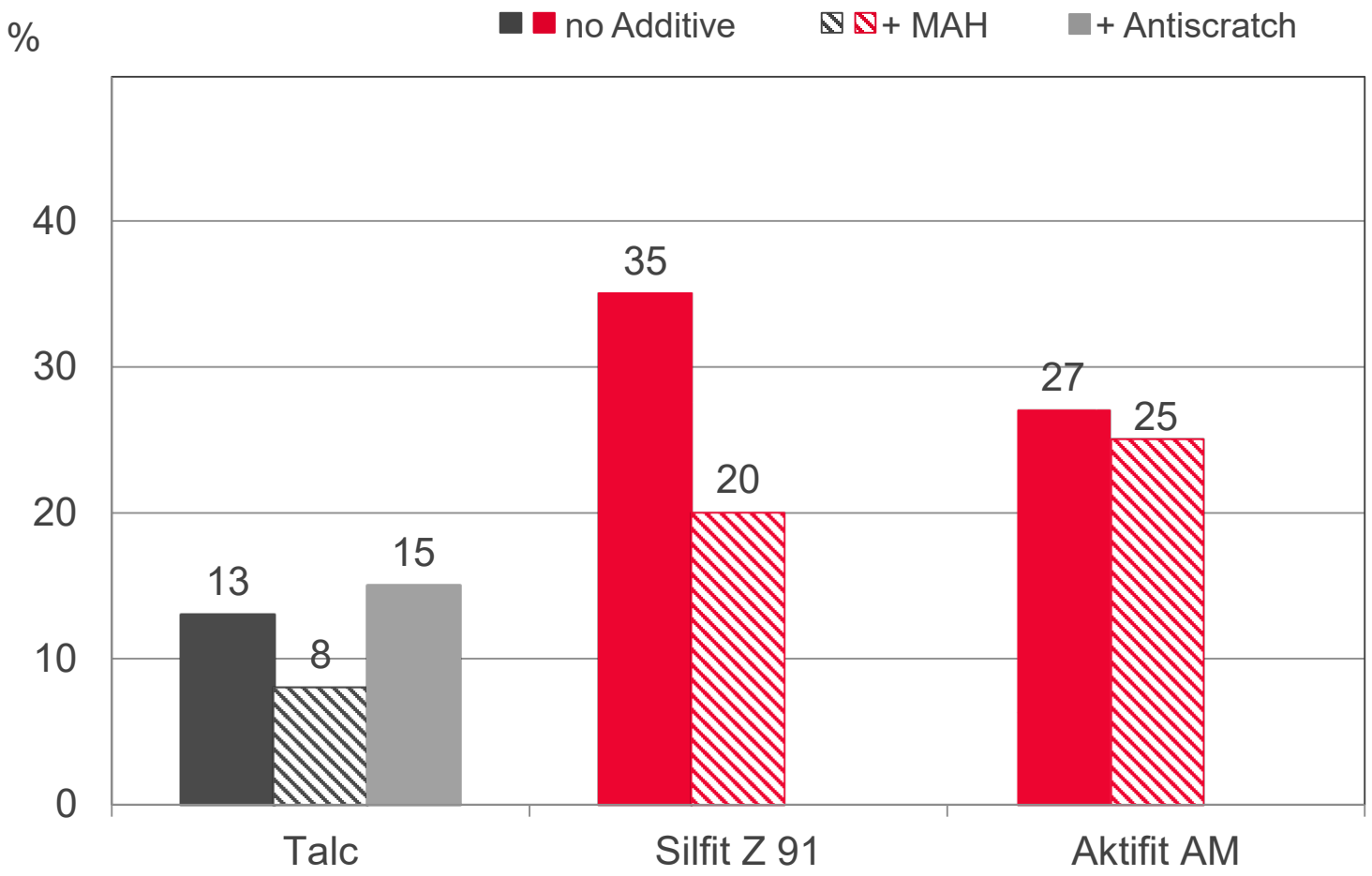




# Nominal Strain at Break

DIN EN ISO 527-1,-2; 50 mm/min

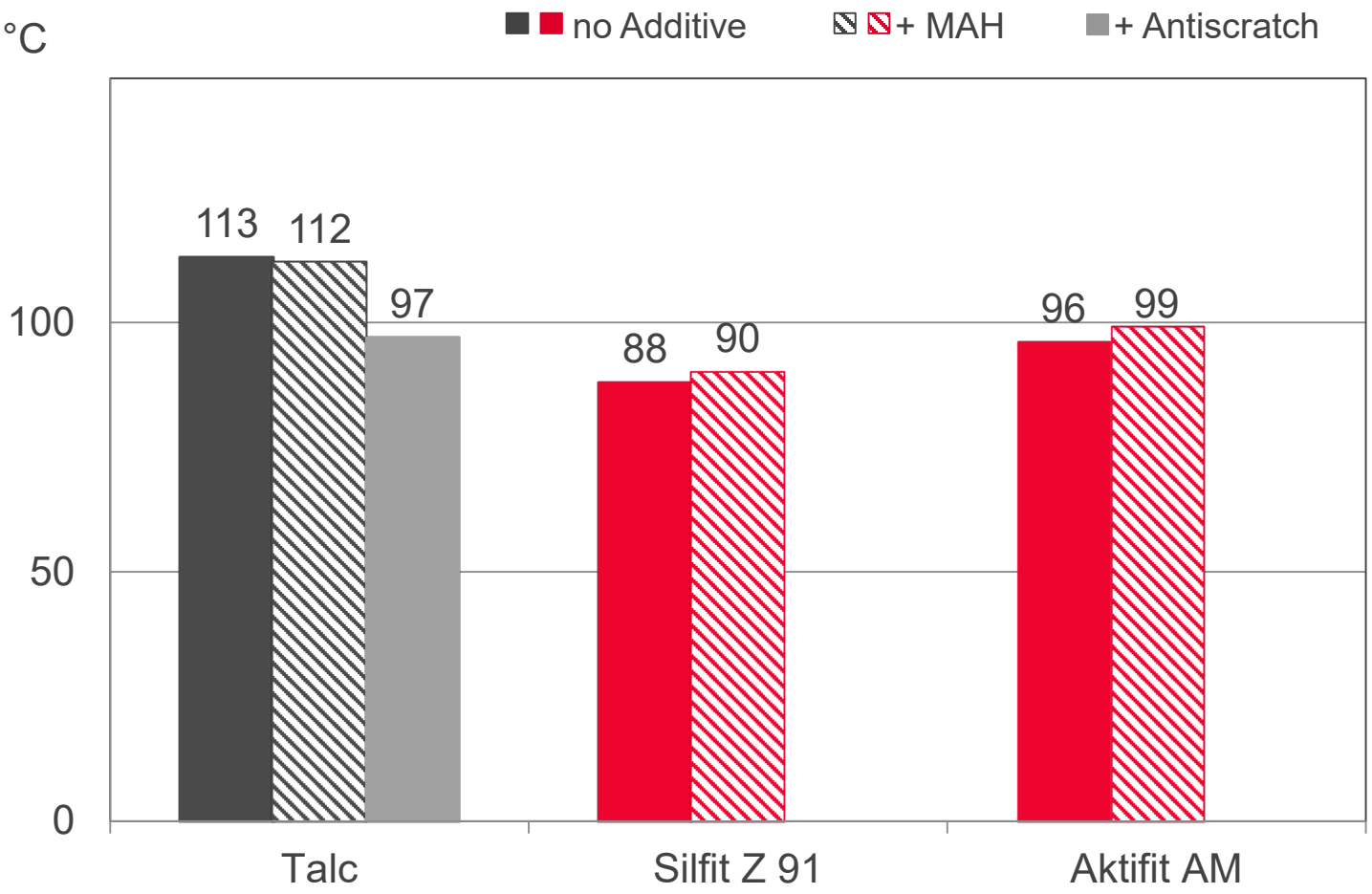




# HDT
# Heat Distortion Temperature

DIN EN ISO 75-1, -2; Method Bf (0.45 MPa)

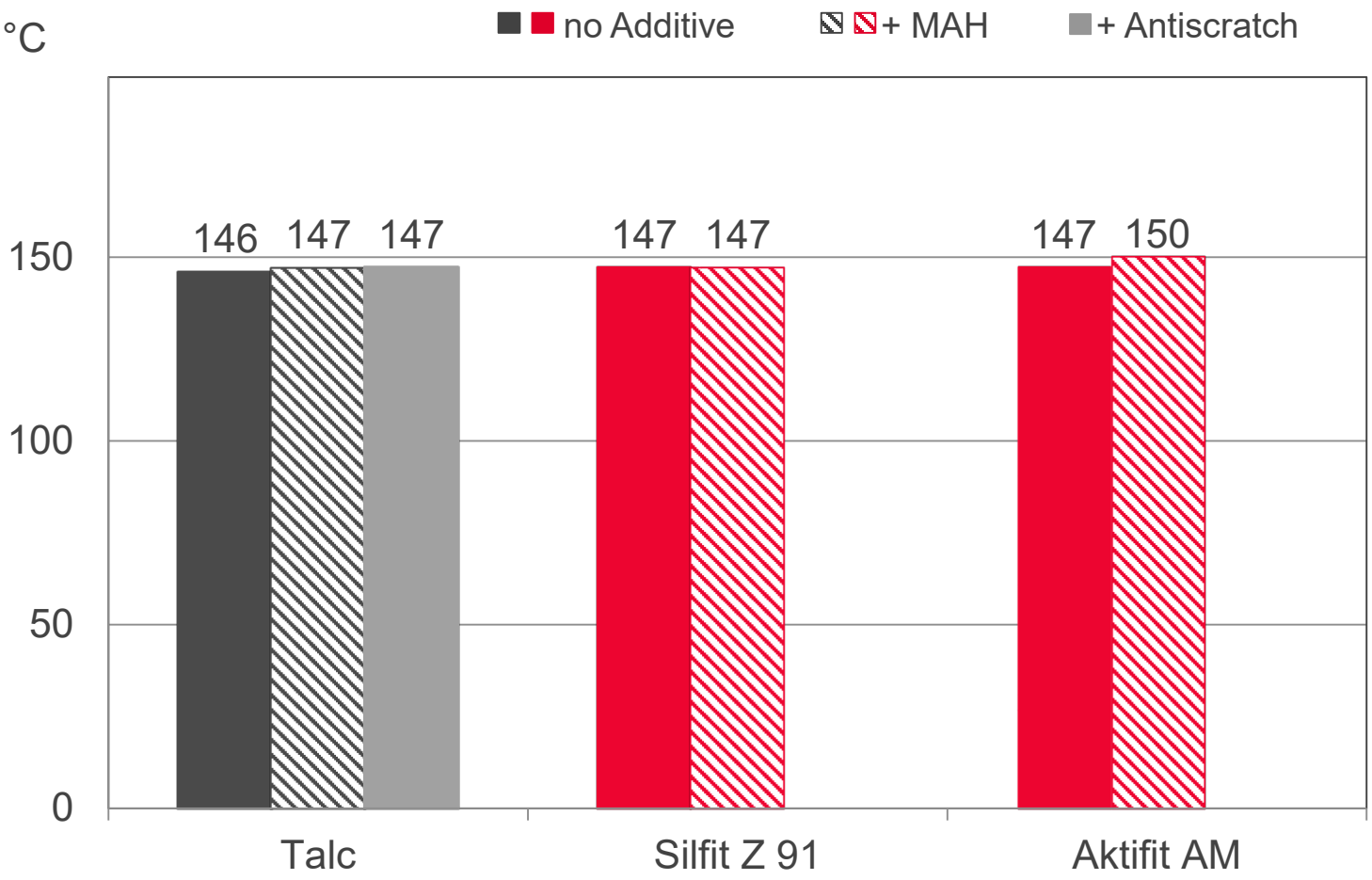




# Vicat Softening Point

DIN EN ISO 306; Method A50 (50 °C/h, 10 N)





# Indentation by Writing
# K 09 (coarse grain)

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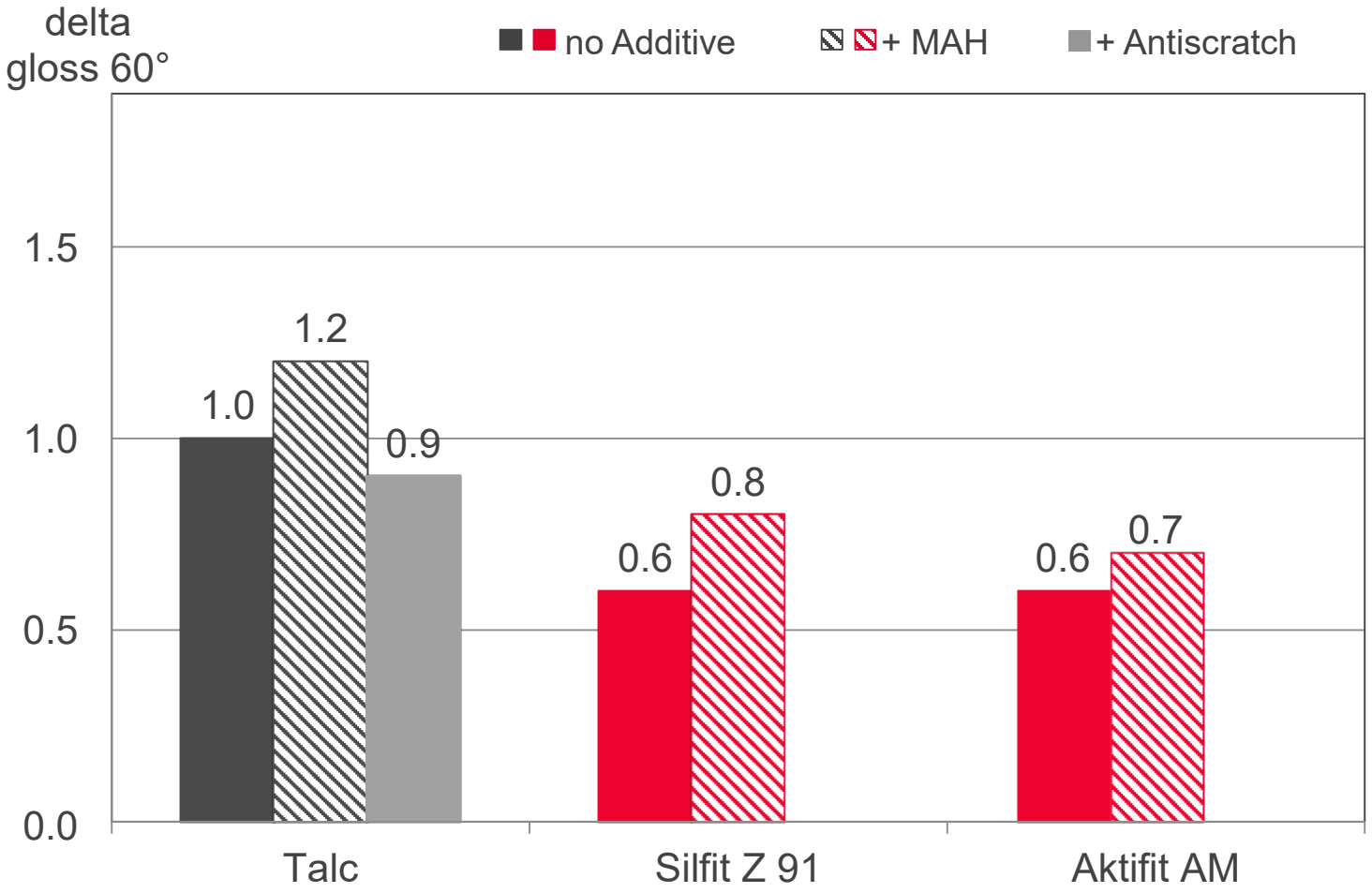
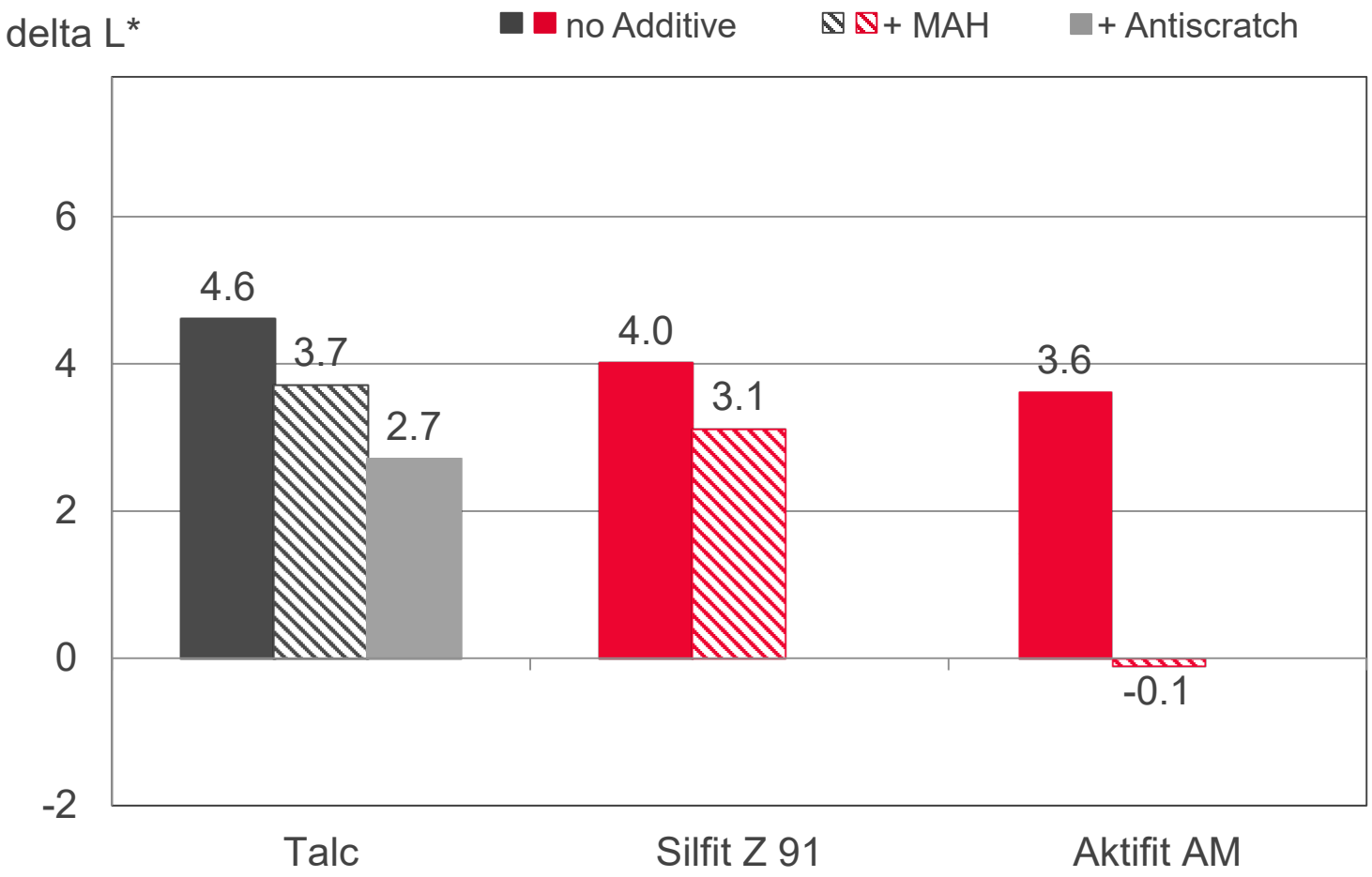
VW PV 3974



# Scratch Resistance
# K 09 (coarse grain)

VW PV 3952, 10 N



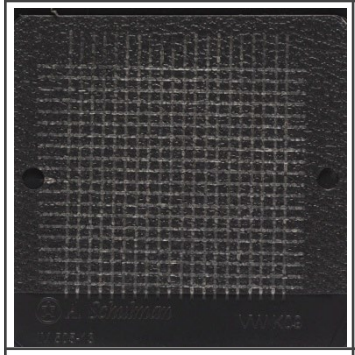


# Scratch Resistance
# K 09 (coarse grain)

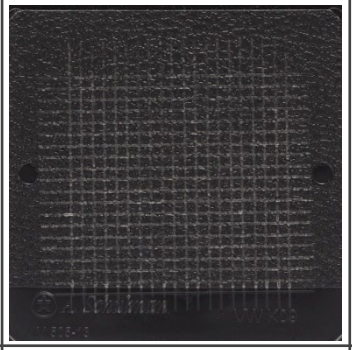
HOFFMANN MINERAL

VW PV 3952, 10 N

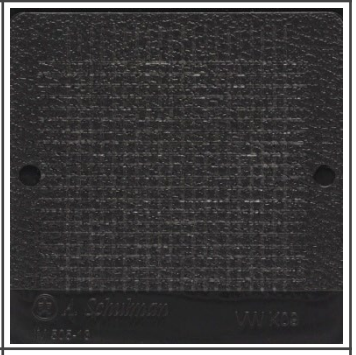




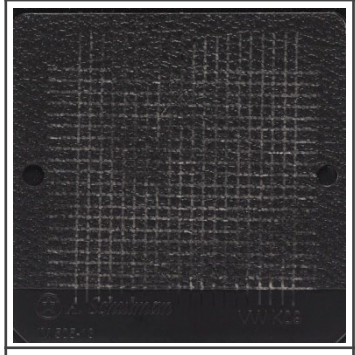
Talc



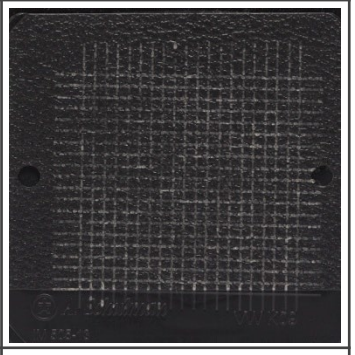
Talc
+ MAH



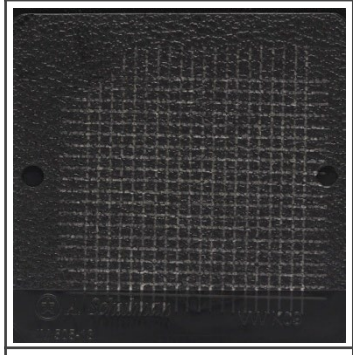
Talc
+ Antiscratch



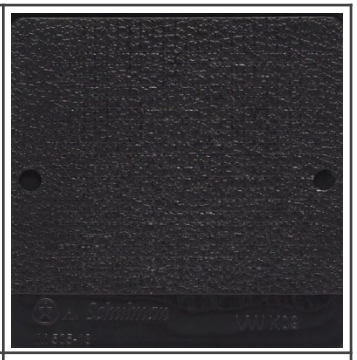
Silfit Z 91



Silfit Z 91
+ MAH



Aktifit AM



Aktifit AM
+ MAH

# Indentation by Writing
# K 31 (fine grain)

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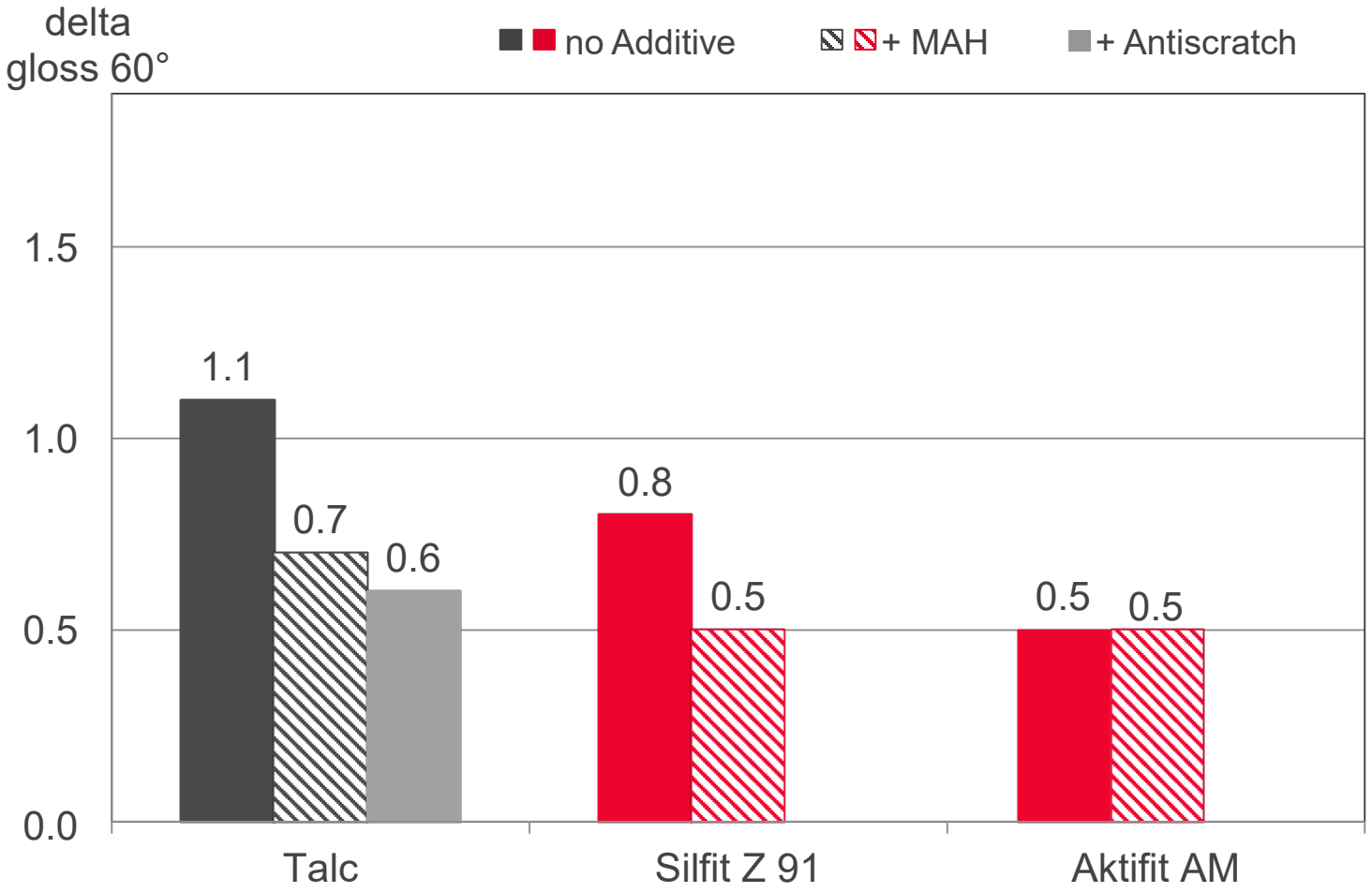
VW PV 3974

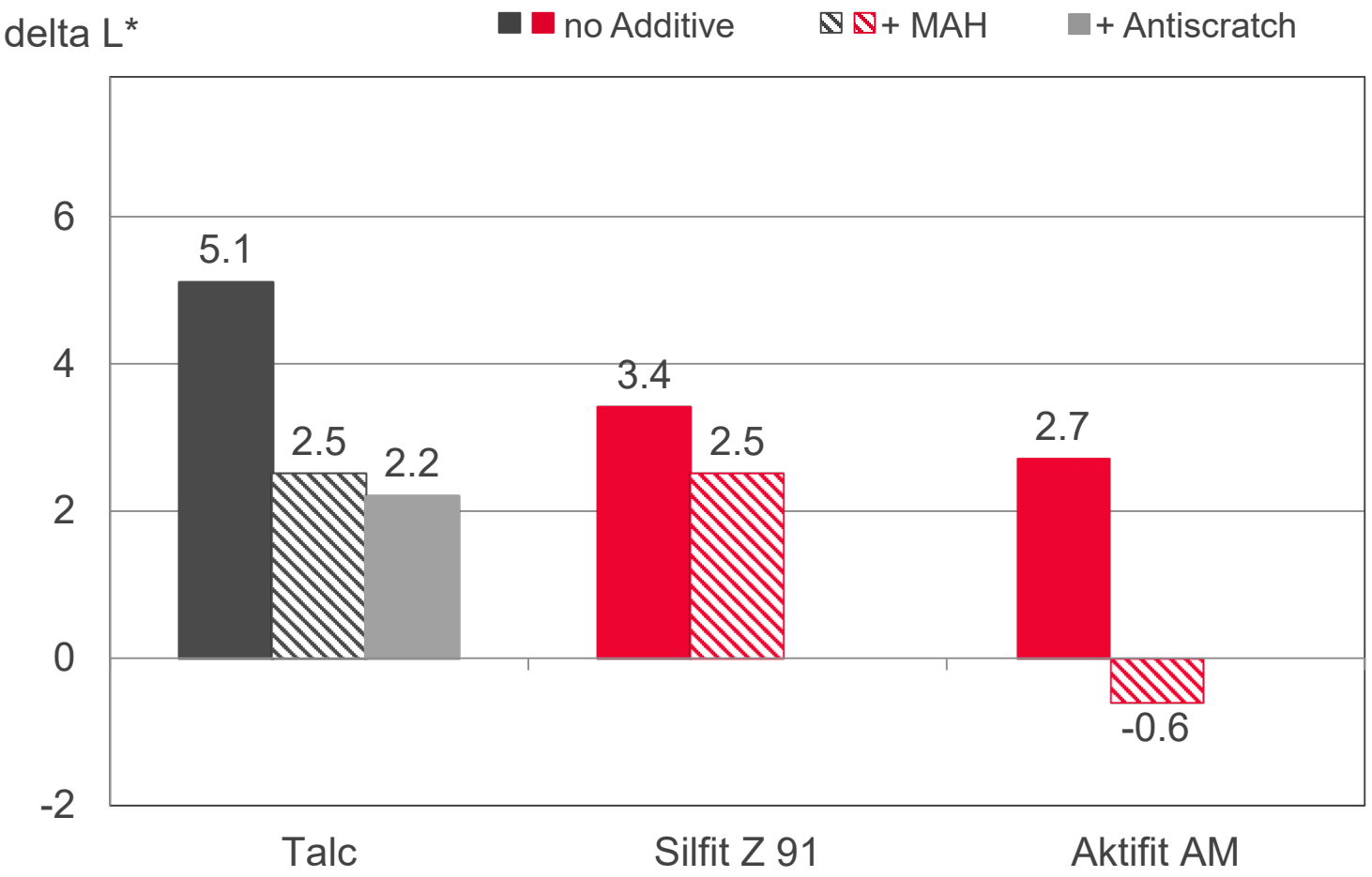


# Scratch Resistance
# K 31 (fine grain)

HOFFMANN MINERAL

VW PV 3952, 10 N





# Scratch Resistance
# K 31 (fine grain)

HOFFMANN MINERAL

VW PV 3952, 10 N



| | | |
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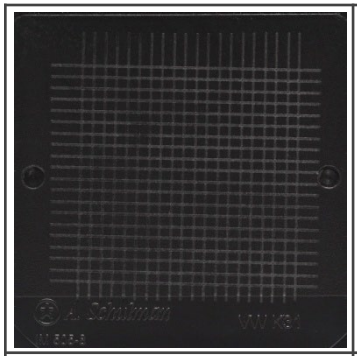
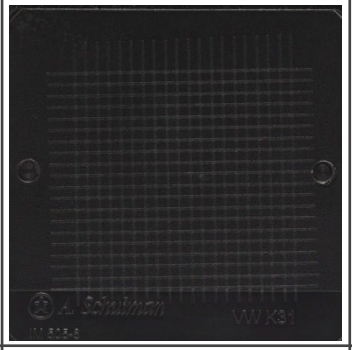
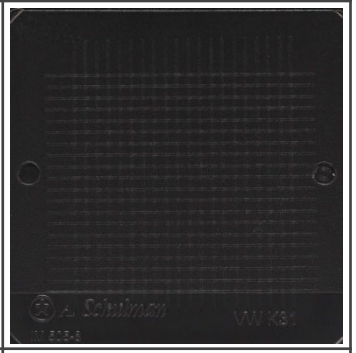
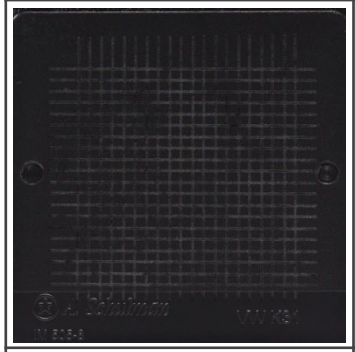
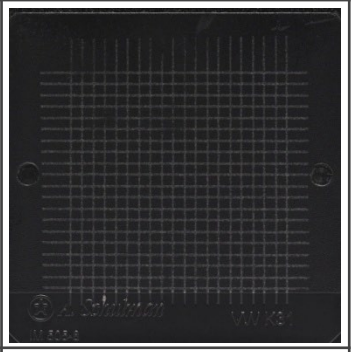
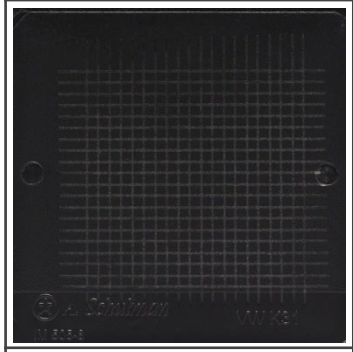
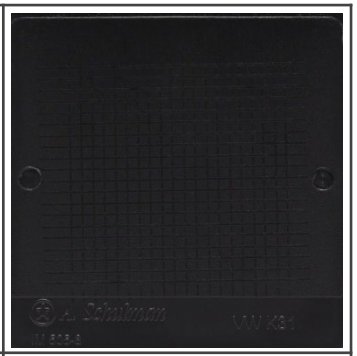
|  |  |  |
| Talc | Talc<br>+ MAH | Talc<br>+ Antiscratch |

| | | | |
|---|---|---|---|
|  |  |  |  |
| Silfit Z 91 | Silfit Z 91<br>+ MAH | Aktifit AM | Aktifit AM<br>+ MAH |

# Formulations PP Copo 40 % Filler

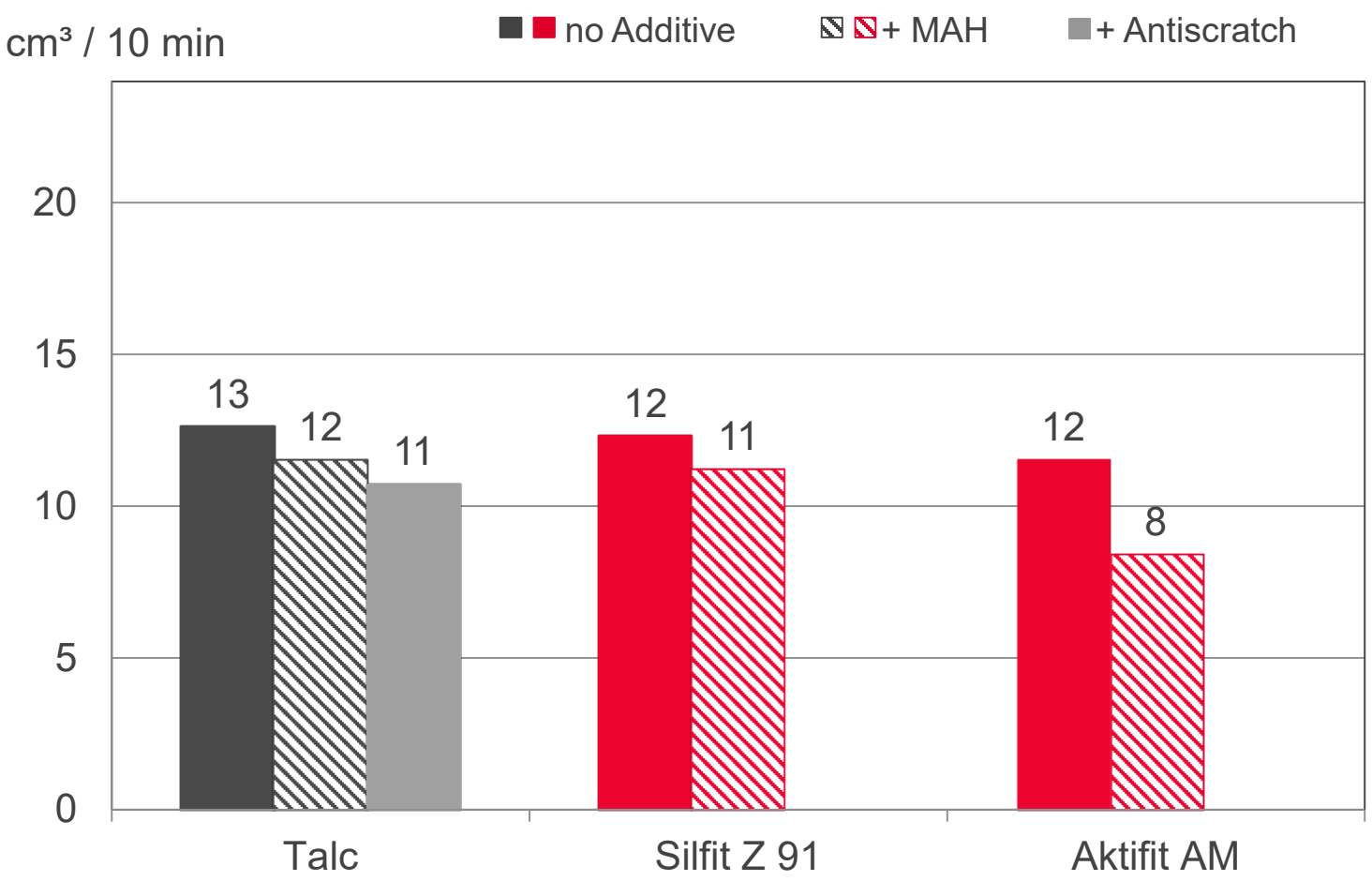


| | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|
| | --- | MAH | Anti-scratch | --- | MAH | --- | MAH |
| **PP Copolymer** MFR 16 g/10 min (230 °C, 2.16 kg) | 57.7 | 52.7 | 55.7 | 57.7 | 52.7 | 57.7 | 52.7 |
| **Stabilizer and black color batch** | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 |
| **Talc** ultrafine | 40 | 40 | 40 | --- | --- | --- | --- |
| **Silfit Z 91** | --- | --- | --- | 40 | 40 | --- | --- |
| **Aktifit AM** | --- | --- | --- | --- | --- | 40 | 40 |
| **Scratch additive** Siloxane based | --- | --- | 2 | --- | --- | --- | --- |
| **MAH-modified PP** (1 % MAH) | --- | 5 | --- | --- | 5 | --- | 5 |
| **Total** | **100** | **100** | **100** | **100** | **100** | **100** | **100** |

# Melt Volume-flow Rate

DIN ISO 1133; 230 °C, 2.16 kg

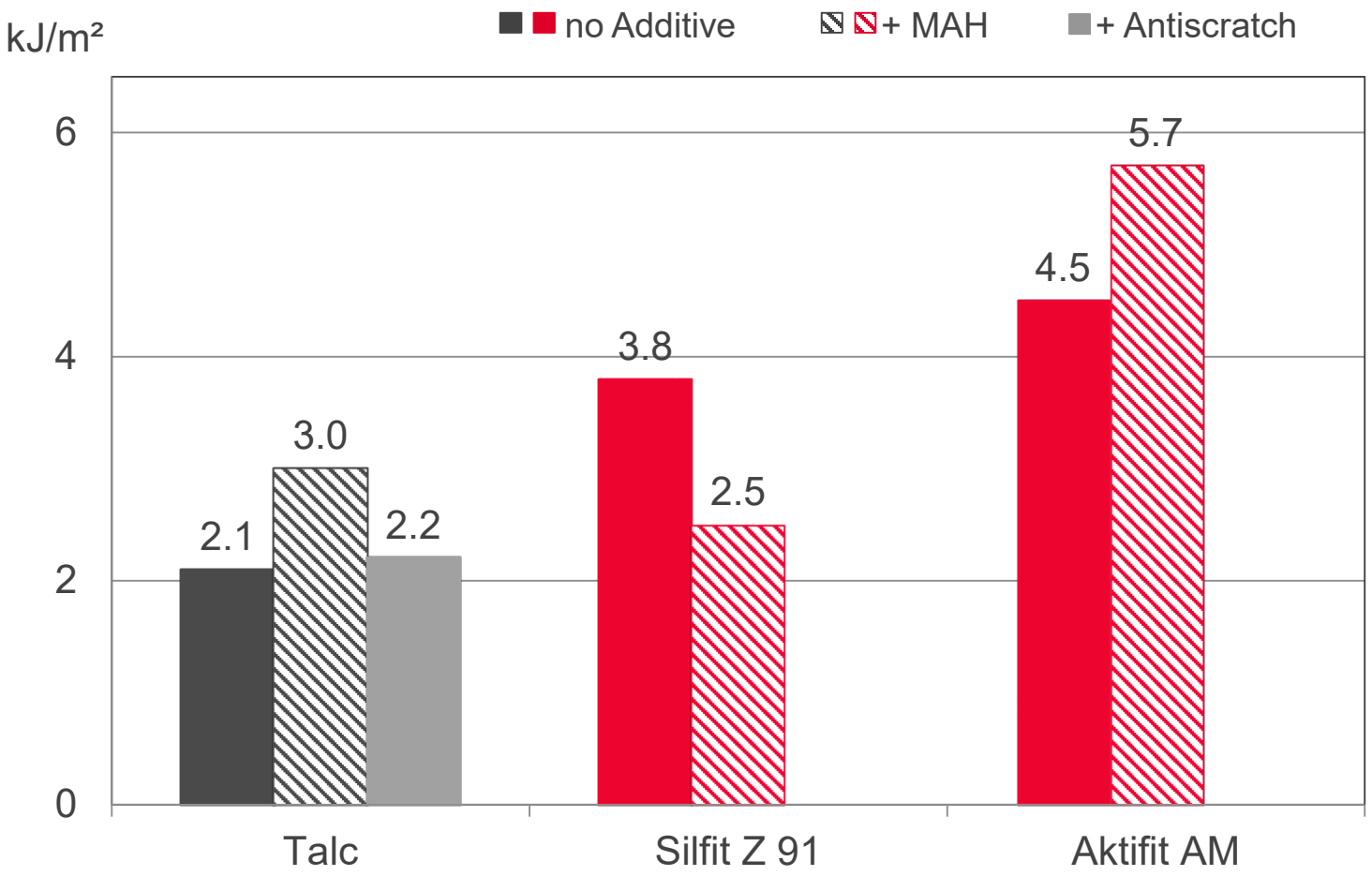




# Charpy
# Notched Impact Strength 23 °C

DIN EN ISO 179-1/1eA

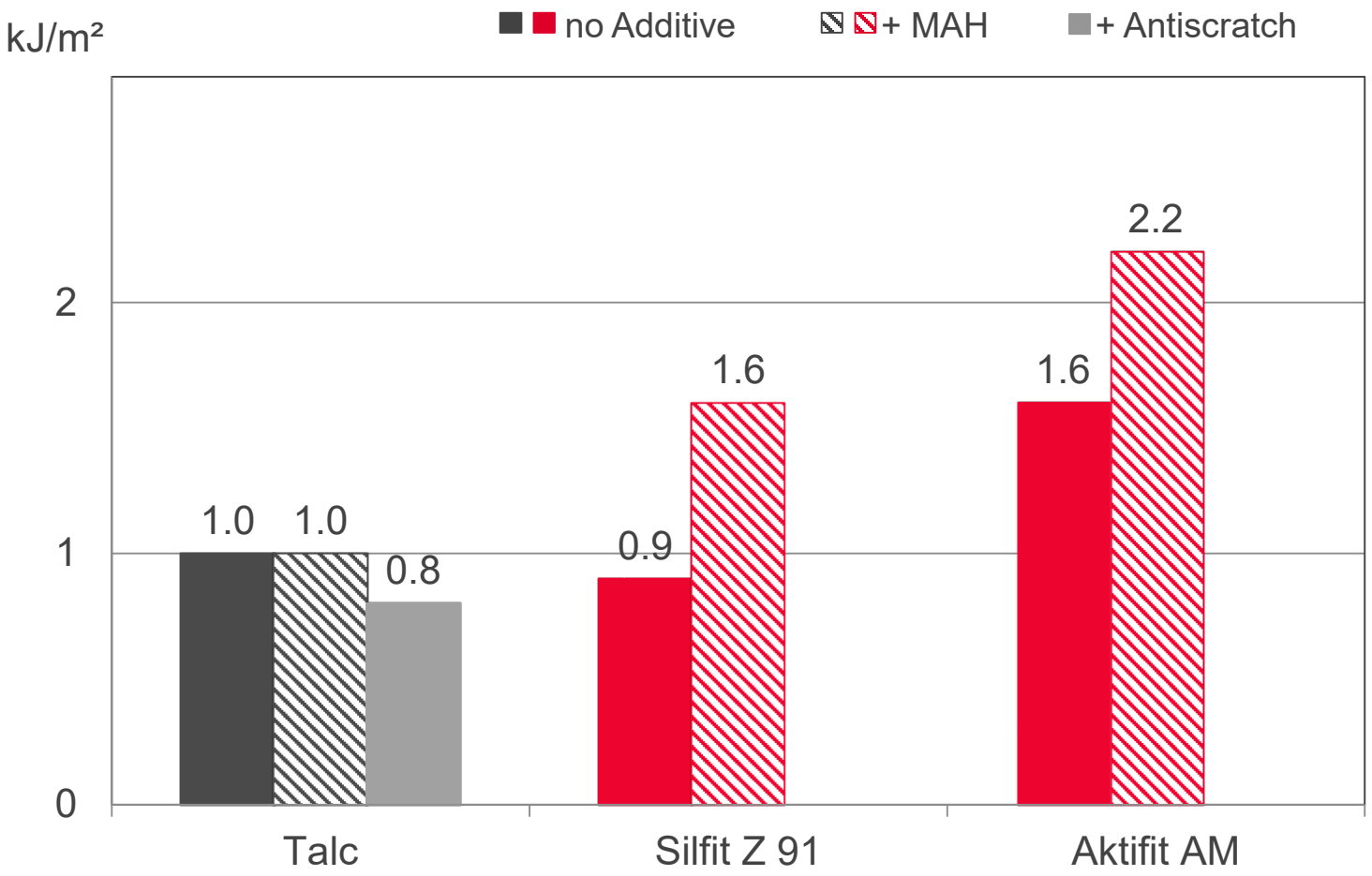




# Charpy
# Notched Impact Strength -30 °C

DIN EN ISO 179-1/1eA

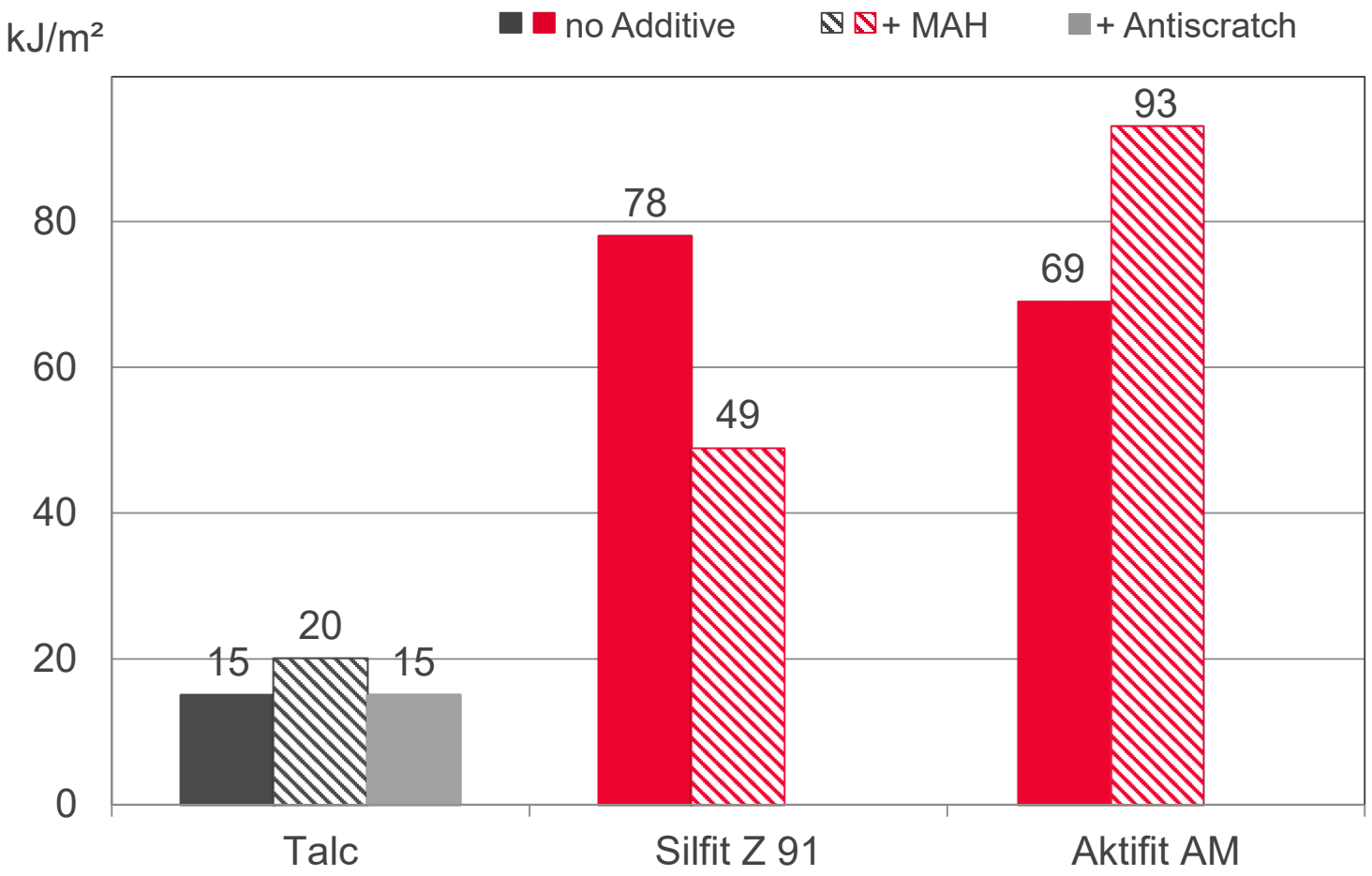




# Charpy
# Impact Strength 23 °C

DIN EN ISO 179-1/1eU

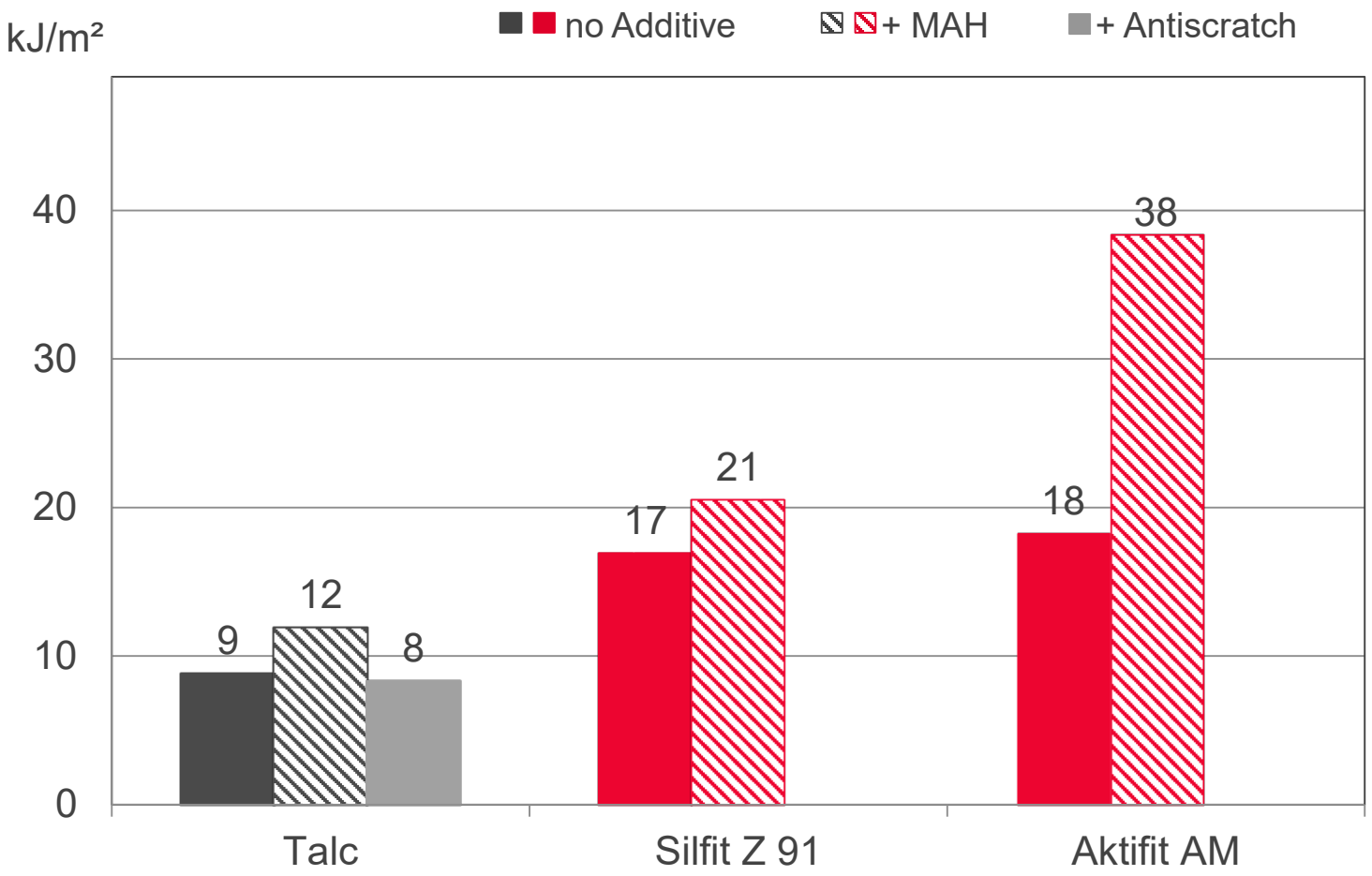




# Charpy
# Impact Strength -30 °C

DIN EN ISO 179-1/1eU

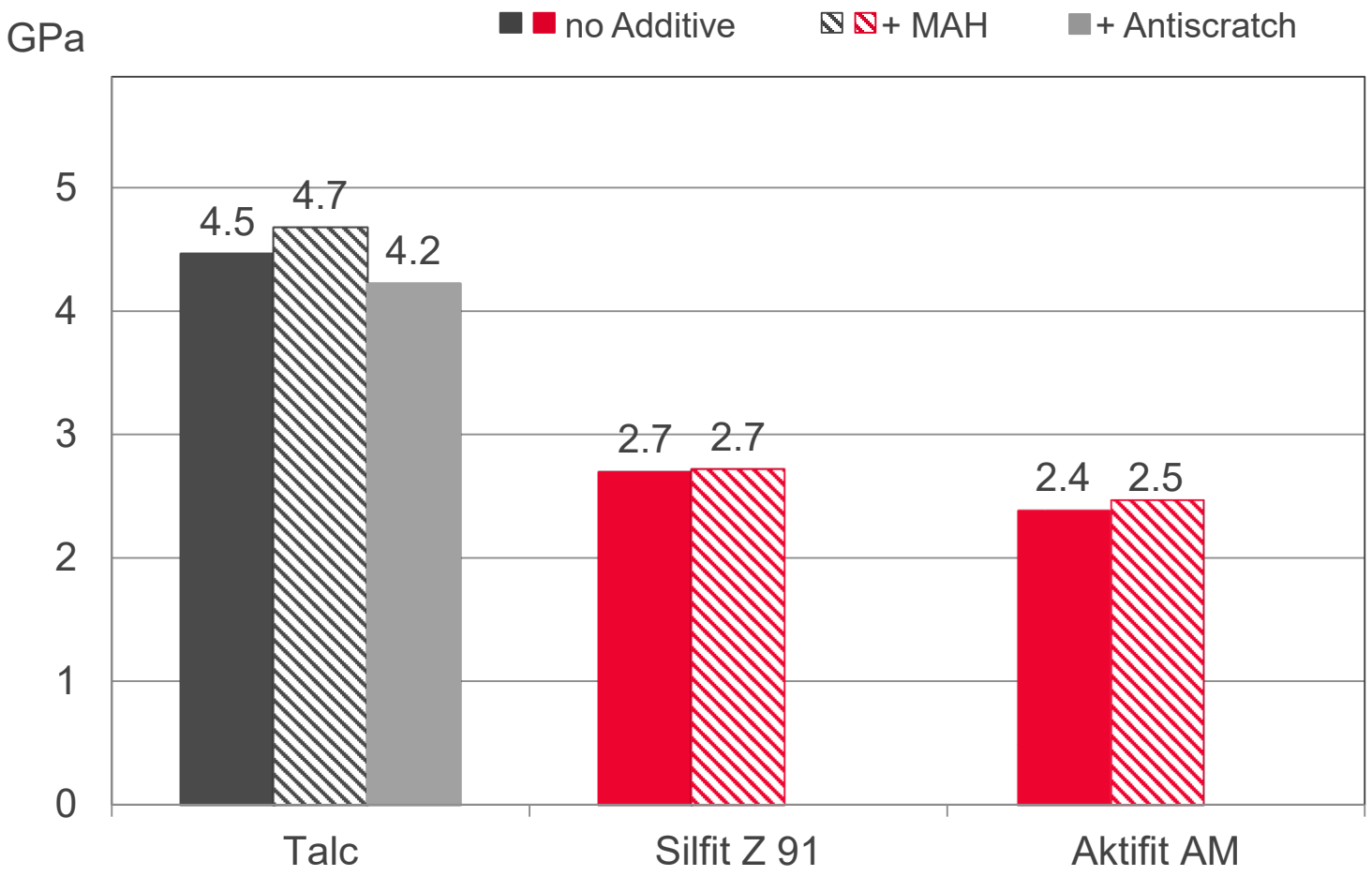




# Tensile Modulus

DIN EN ISO 527-1,-2; 1 mm/min

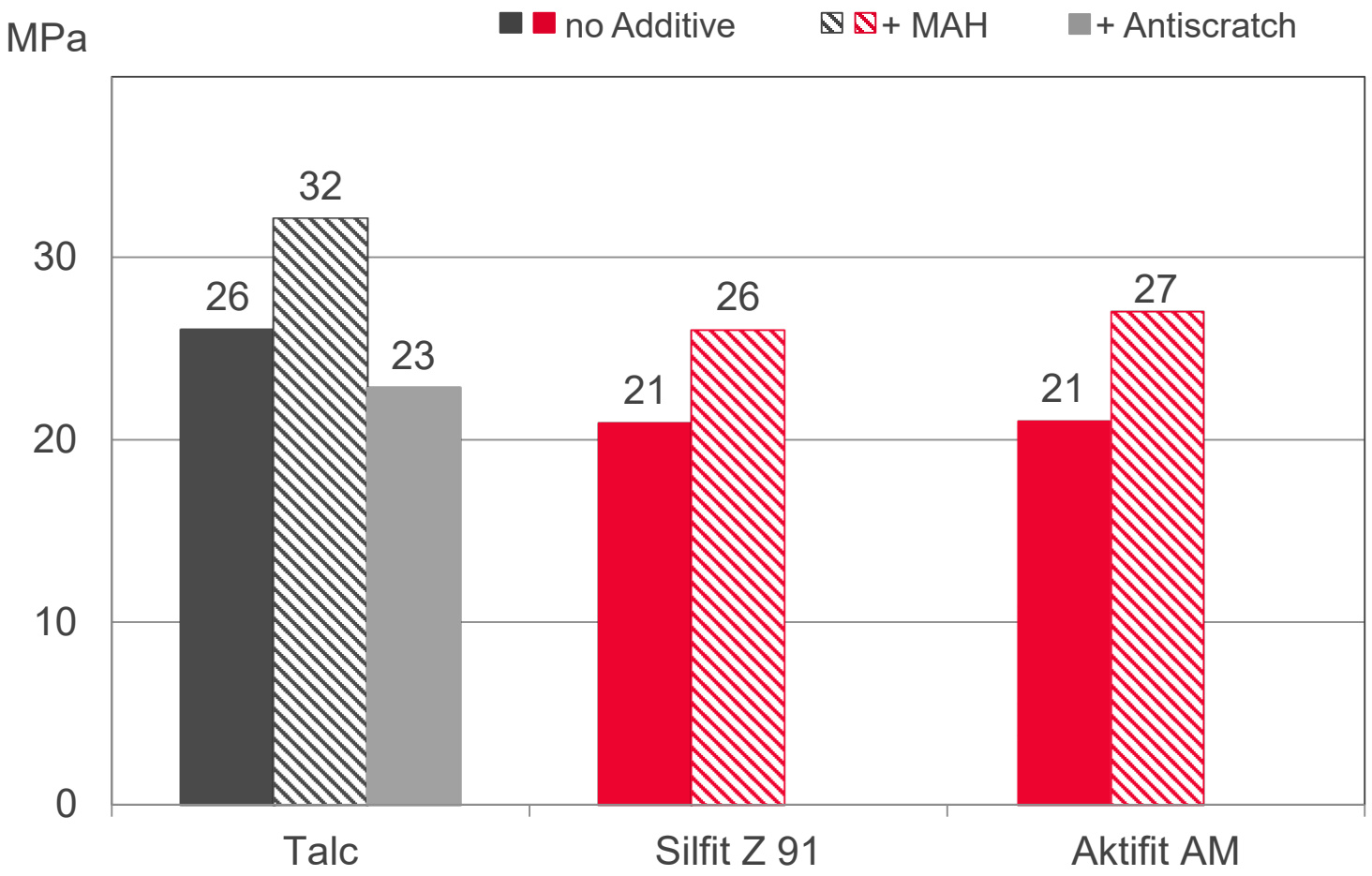




# Tensile Strength

DIN EN ISO 527-1,-2; 5 mm/min

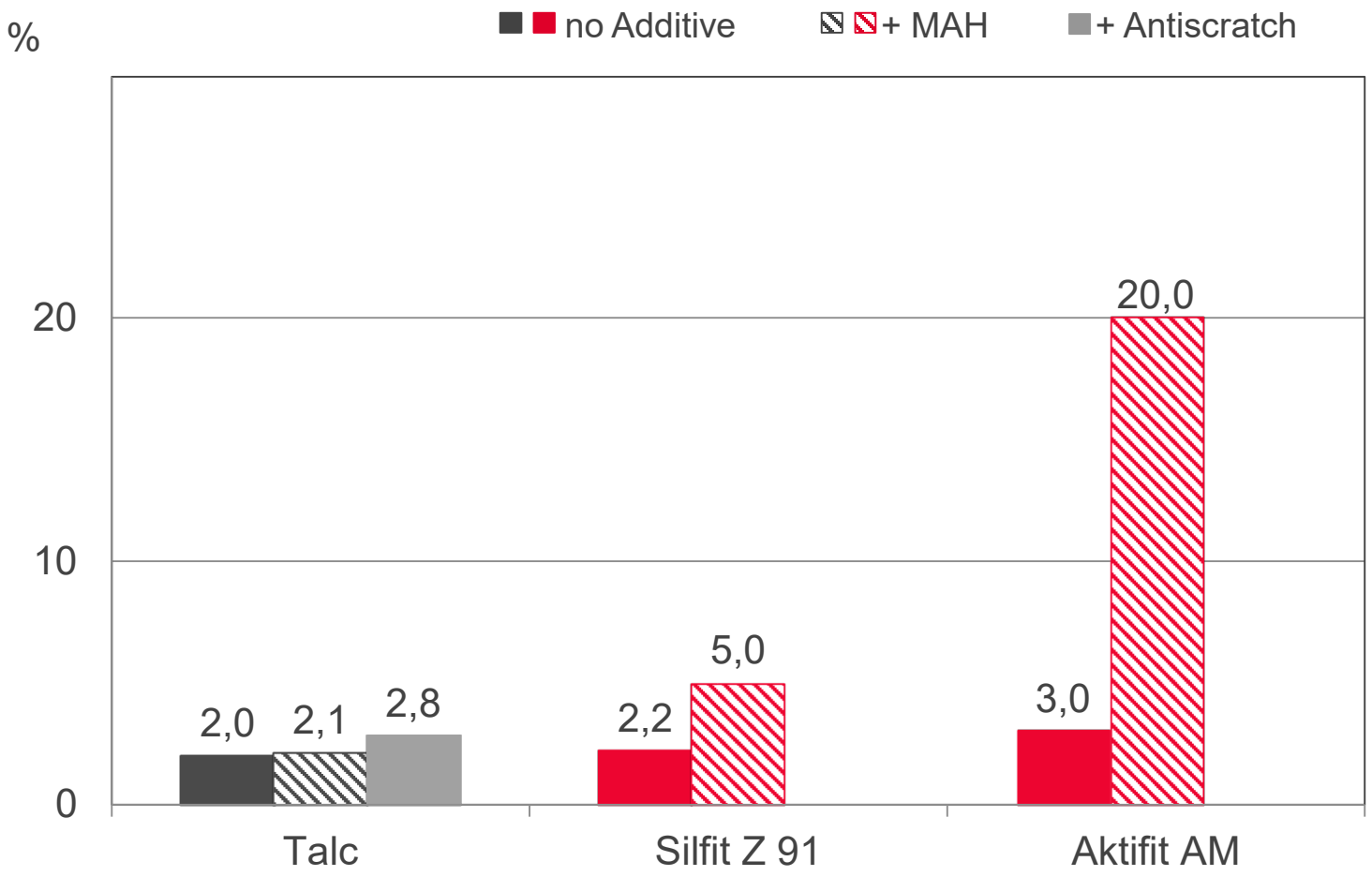




# Tensile Strain at Break

DIN EN ISO 527-1,-2; 5 mm/min

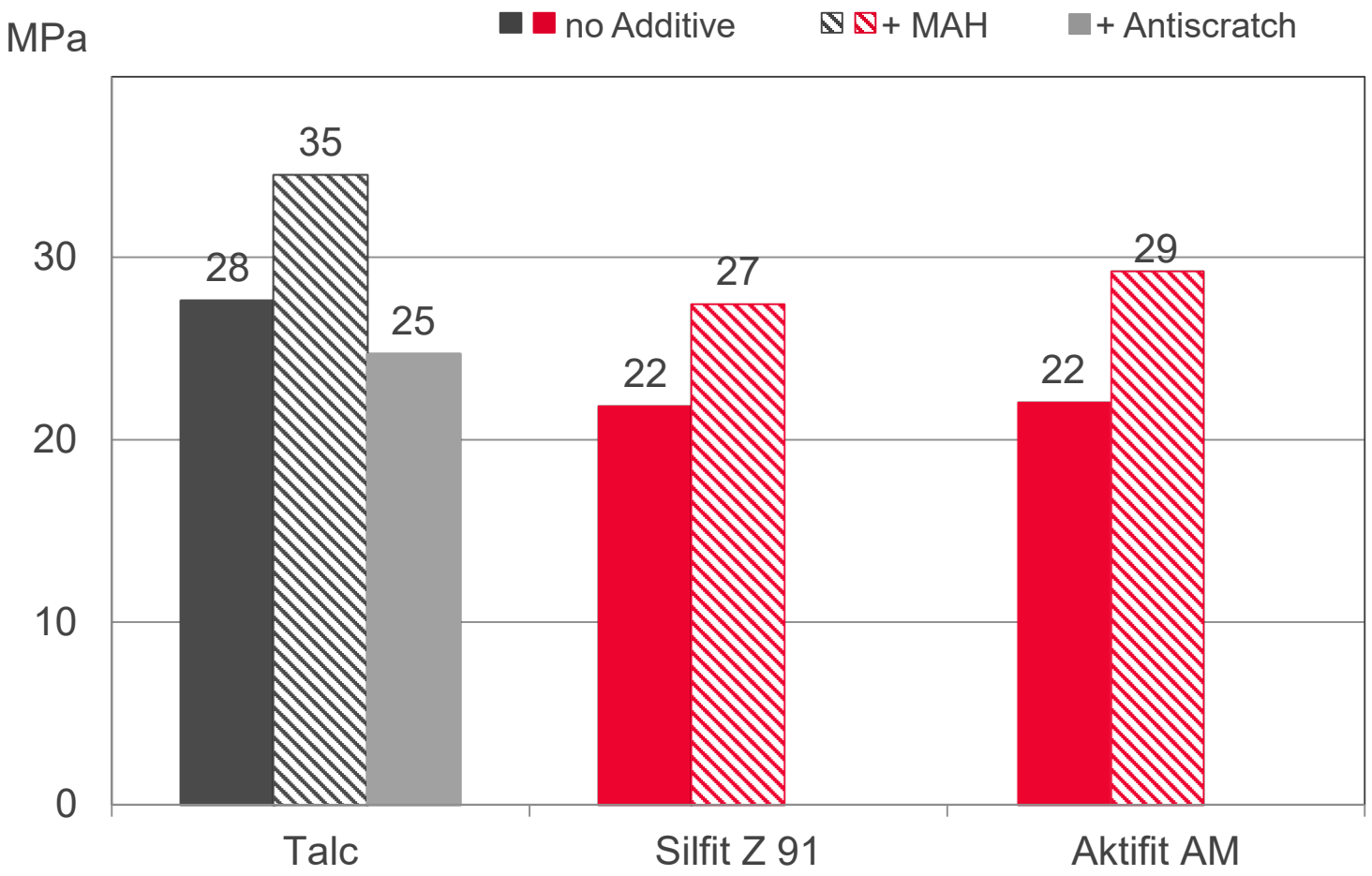




# Yield Stress

DIN EN ISO 527-1,-2; 50 mm/min

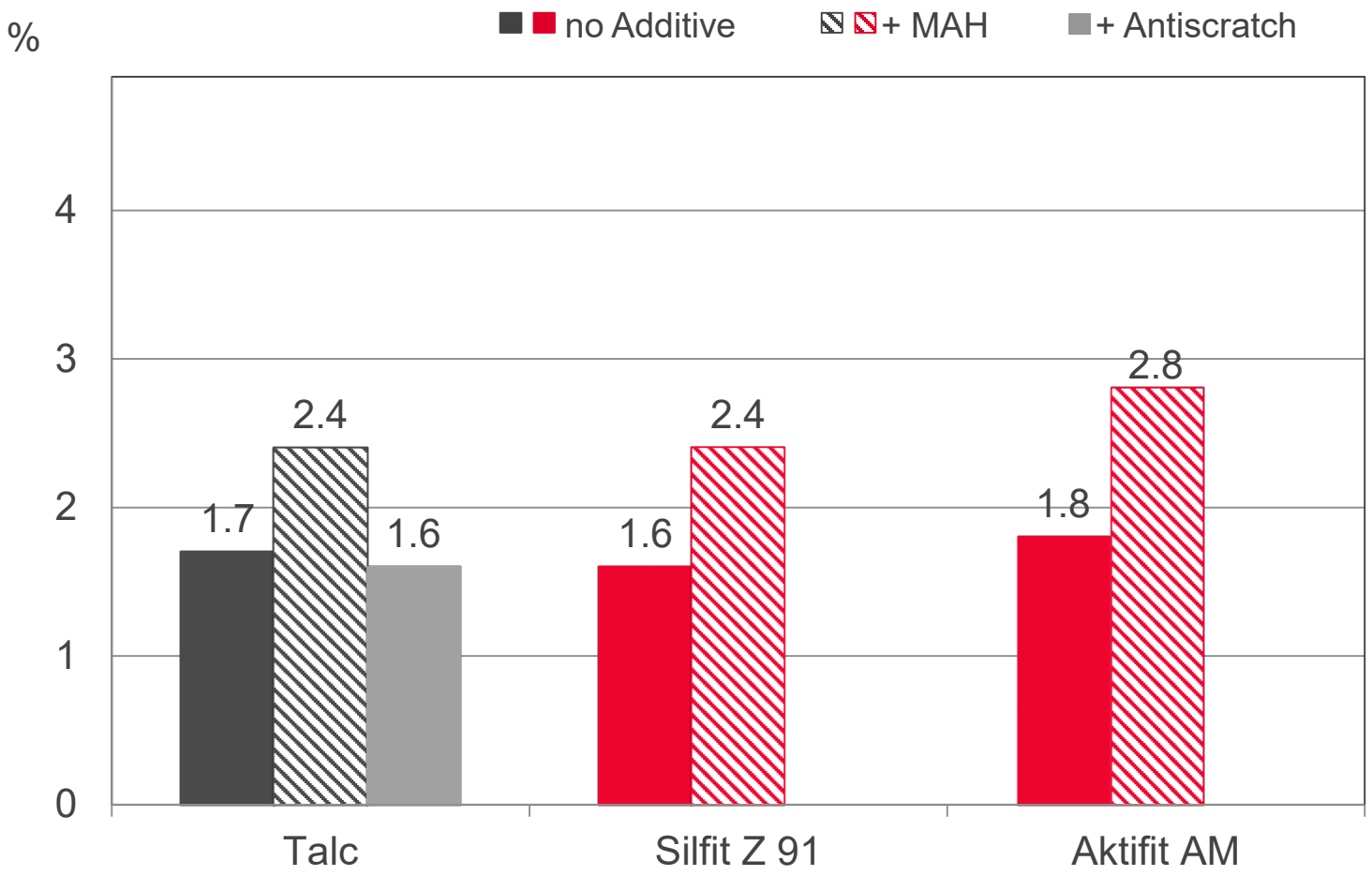




# Yield Strain

DIN EN ISO 527-1,-2; 50 mm/min

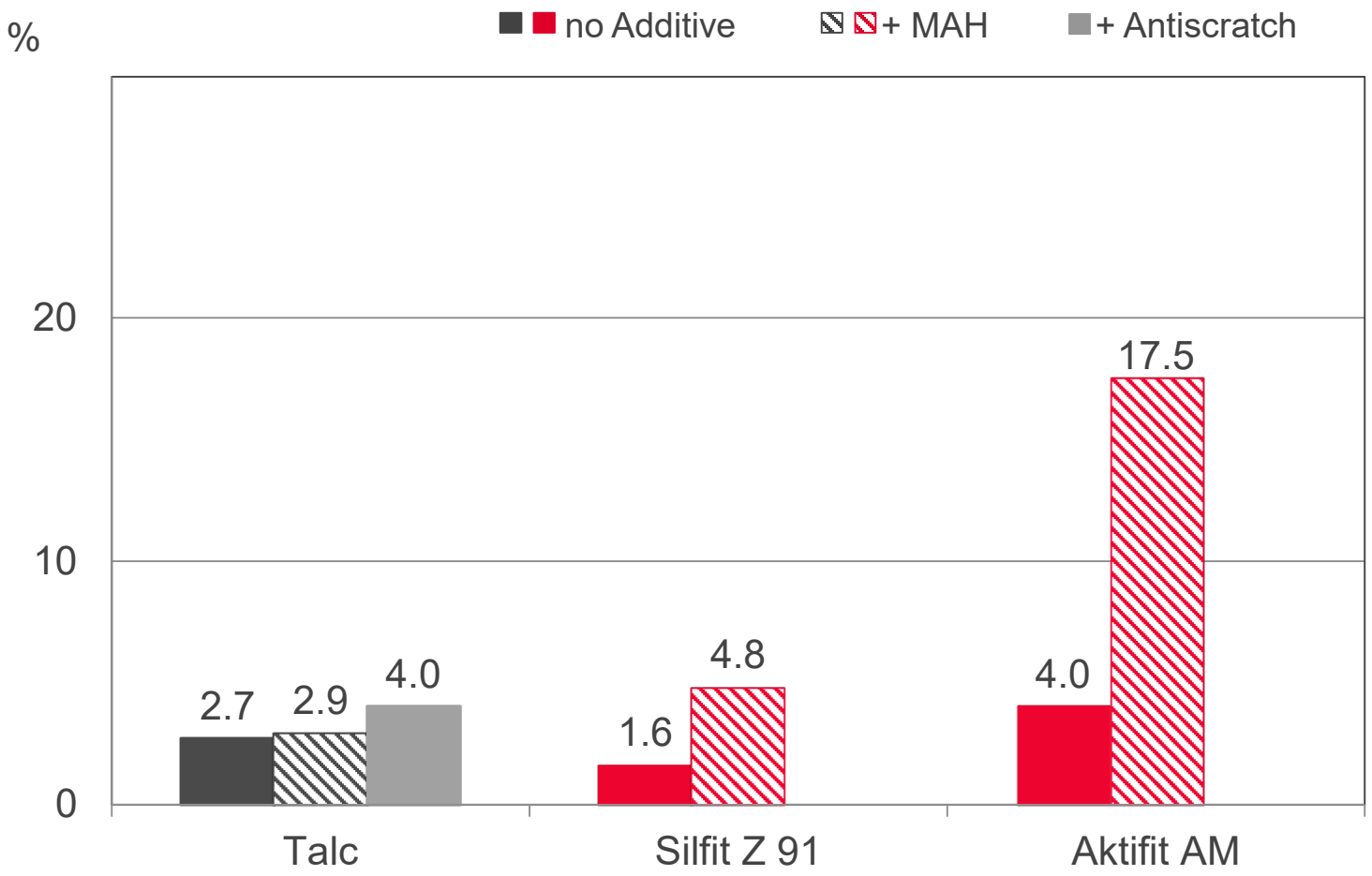




# Nominal Strain at Break

DIN EN ISO 527-1,-2; 50 mm/min

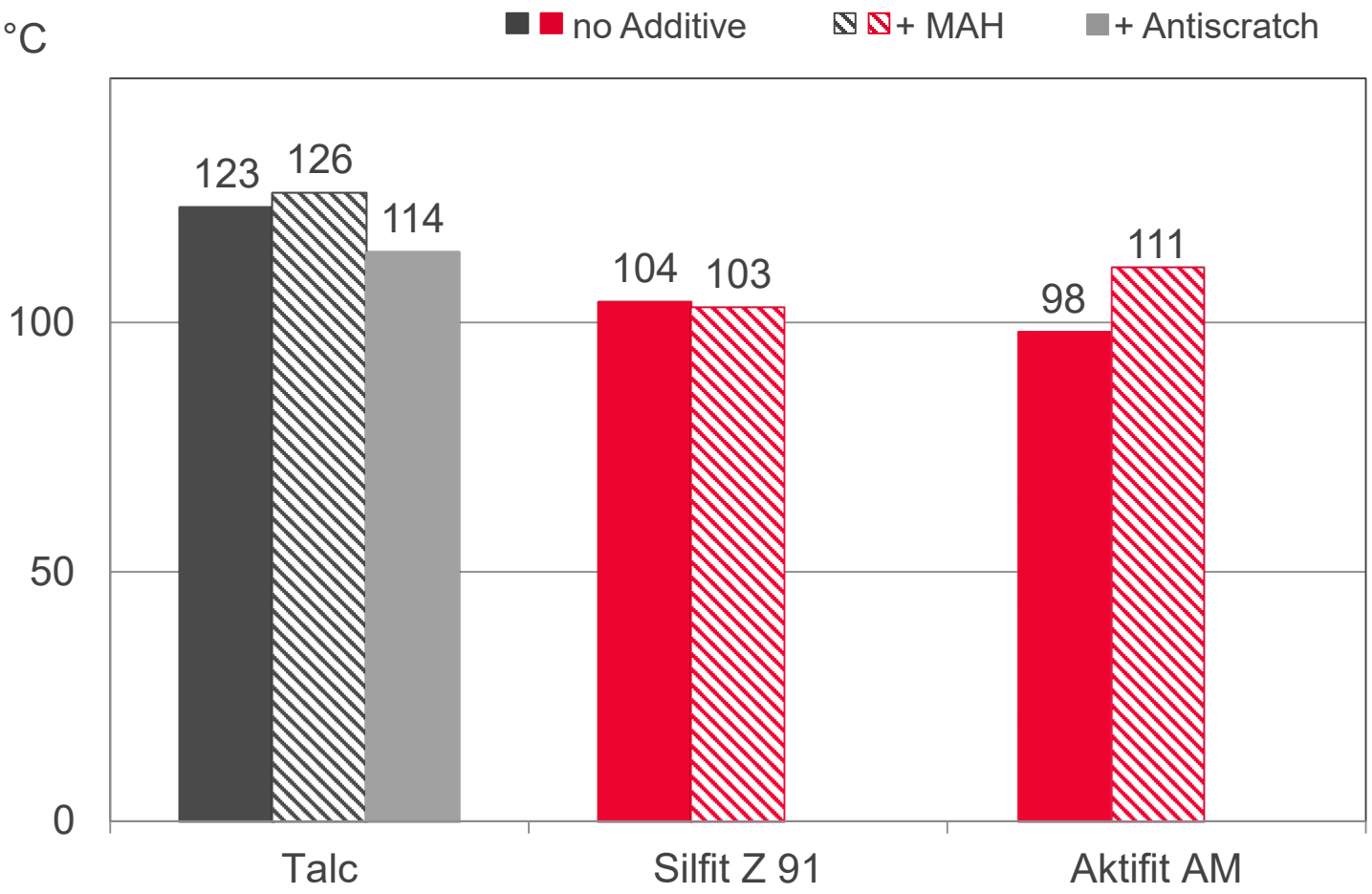




# HDT
# Heat Distortion Temperature

DIN EN ISO 75-1, -2; Method Bf (0.45 MPa)

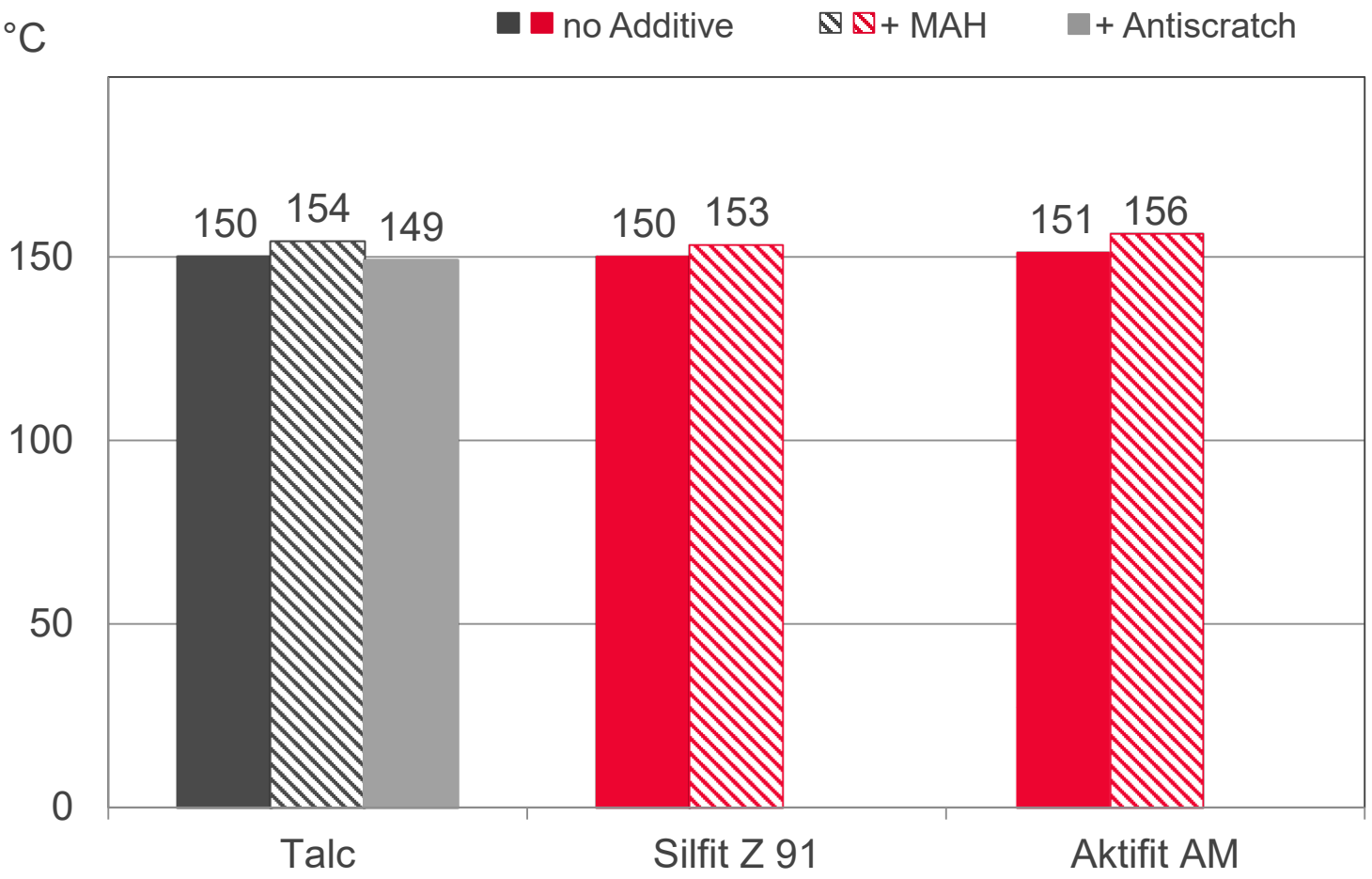




# Vicat Softening Point

DIN EN ISO 306; Method A50 (50 °C/h, 10 N)



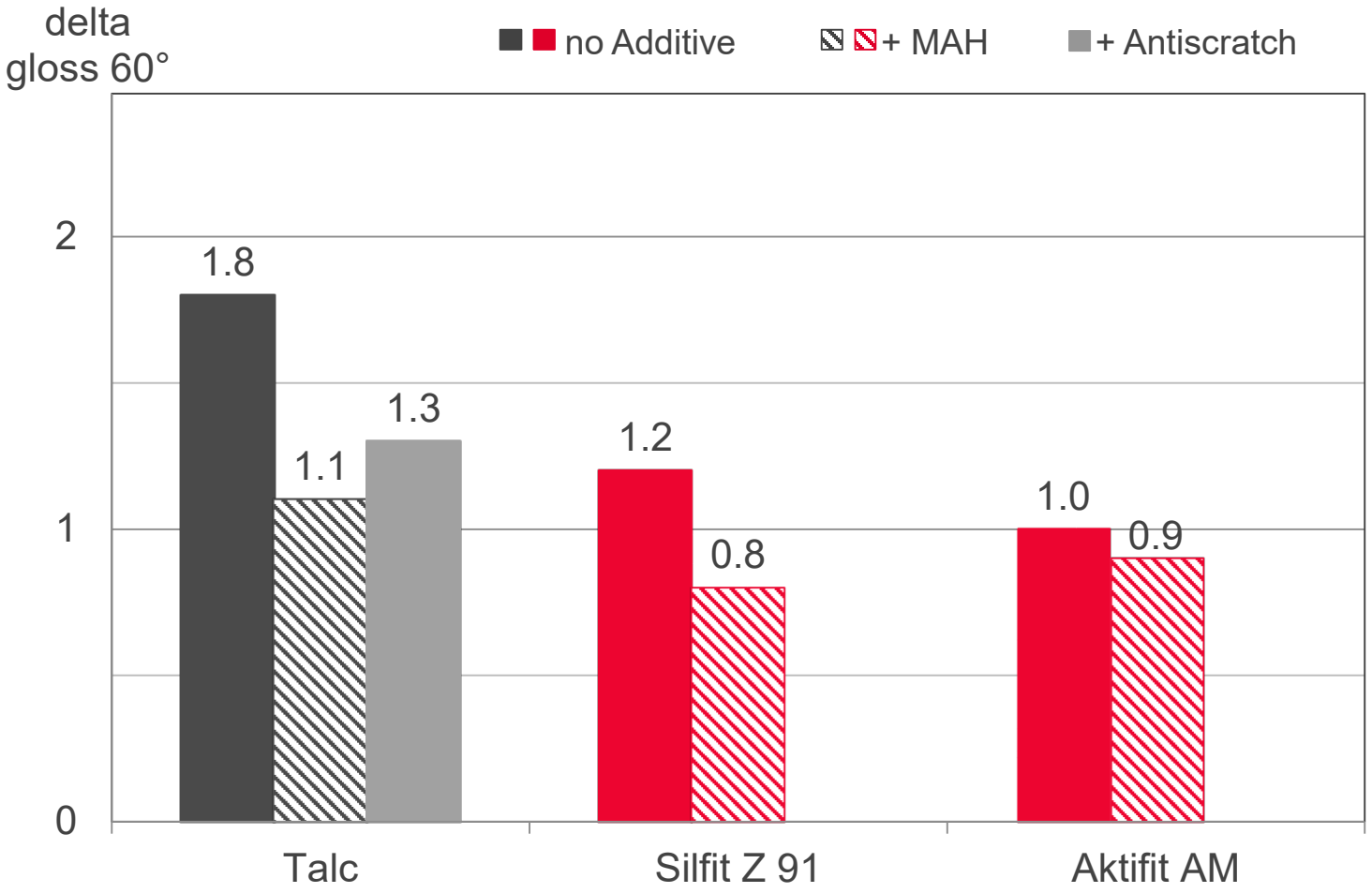


# Indentation by Writing
# K 09 (coarse grain)

HOFFMANN MINERAL

VW PV 3974

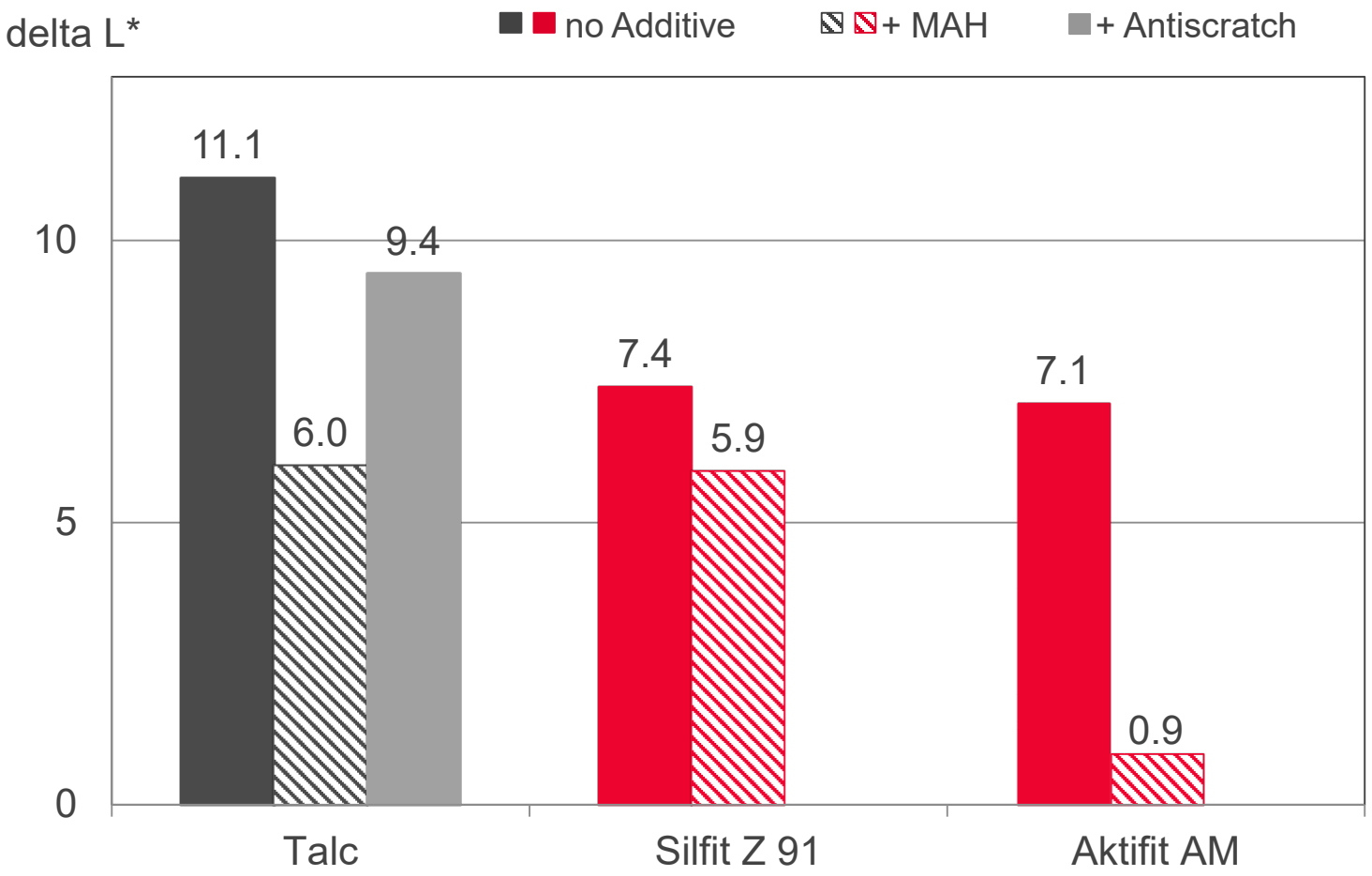




# Scratch Resistance
# K 09 (coarse grain)

VW PV 3952, 10 N



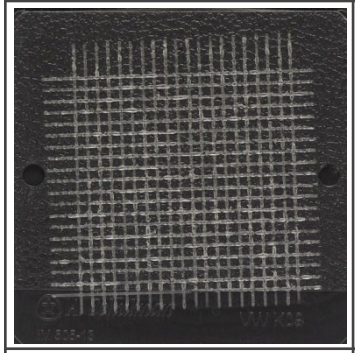
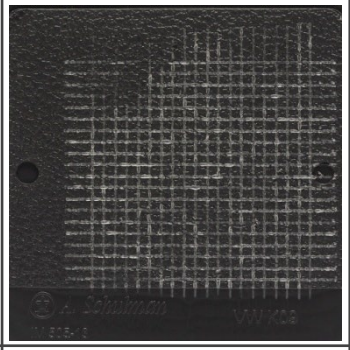
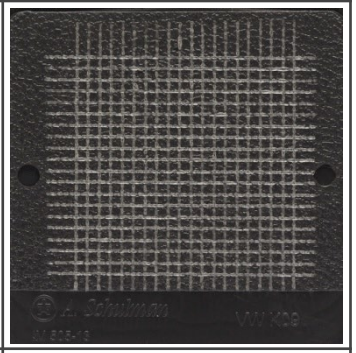


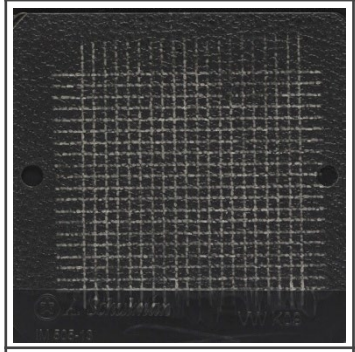
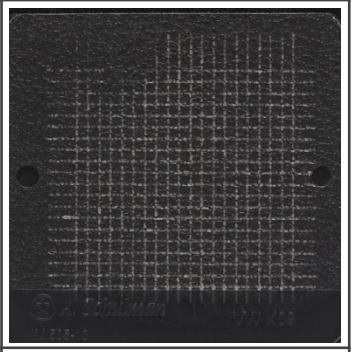
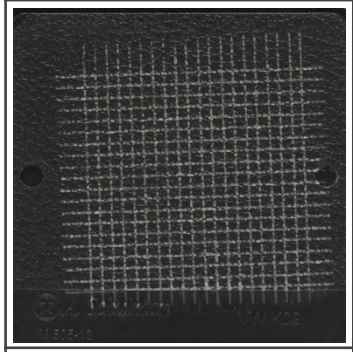
# Scratch Resistance
# K 09 (coarse grain)

HOFFMANN MINERAL

VW PV 3952, 10 N



| | | |
|---|---|---|
|  |  |  |
| Talc | Talc<br>+ MAH | Talc<br>+ Antiscratch |

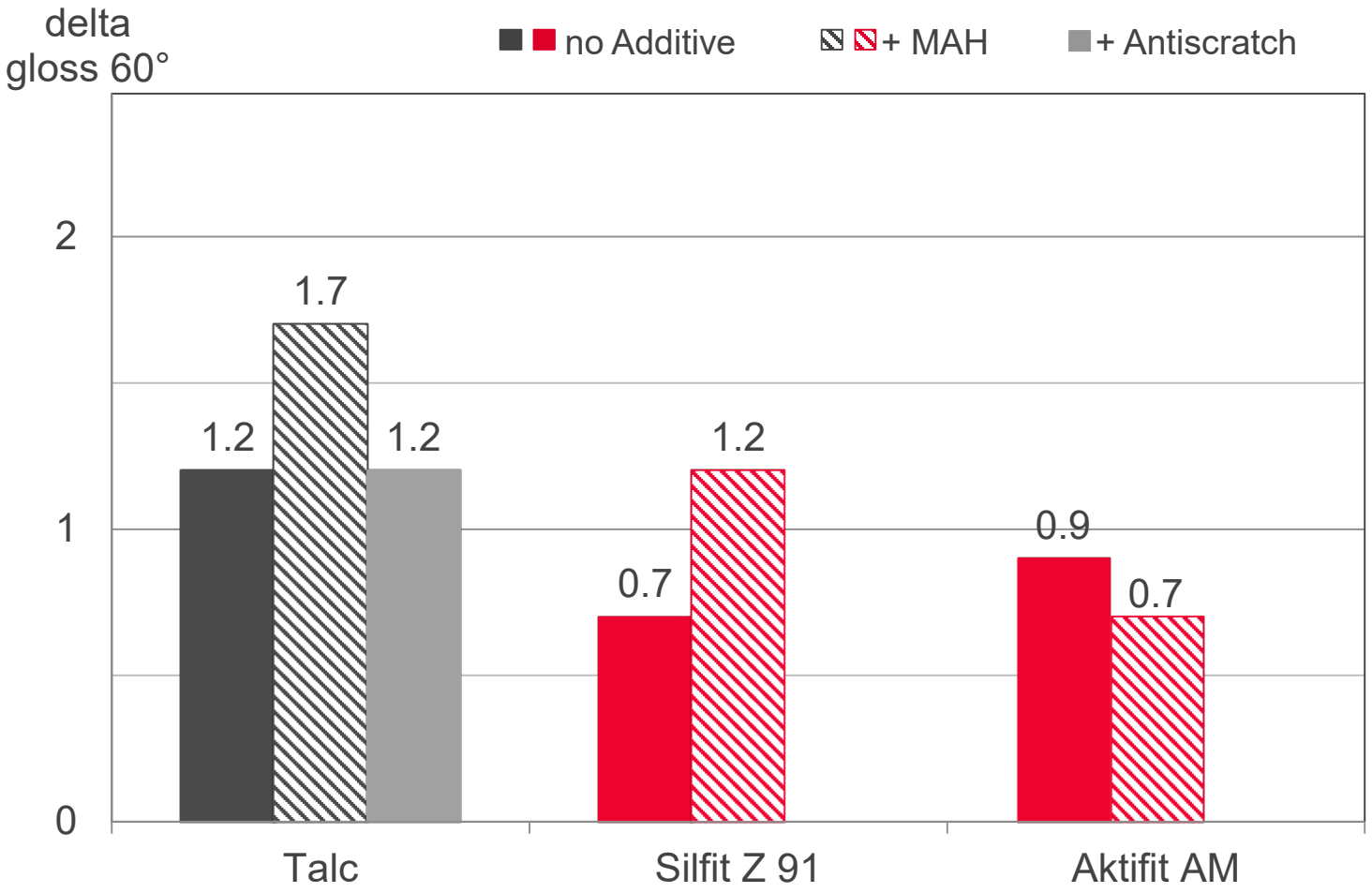
| | | | |
|---|---|---|---|
|  |  |  |  |
| Silfit Z 91 | Silfit Z 91<br>+ MAH | Aktifit AM | Aktifit AM<br>+ MAH |

# Indentation by Writing
# K 31 (fine grain)

HOFFMANN MINERAL



VW PV 3974

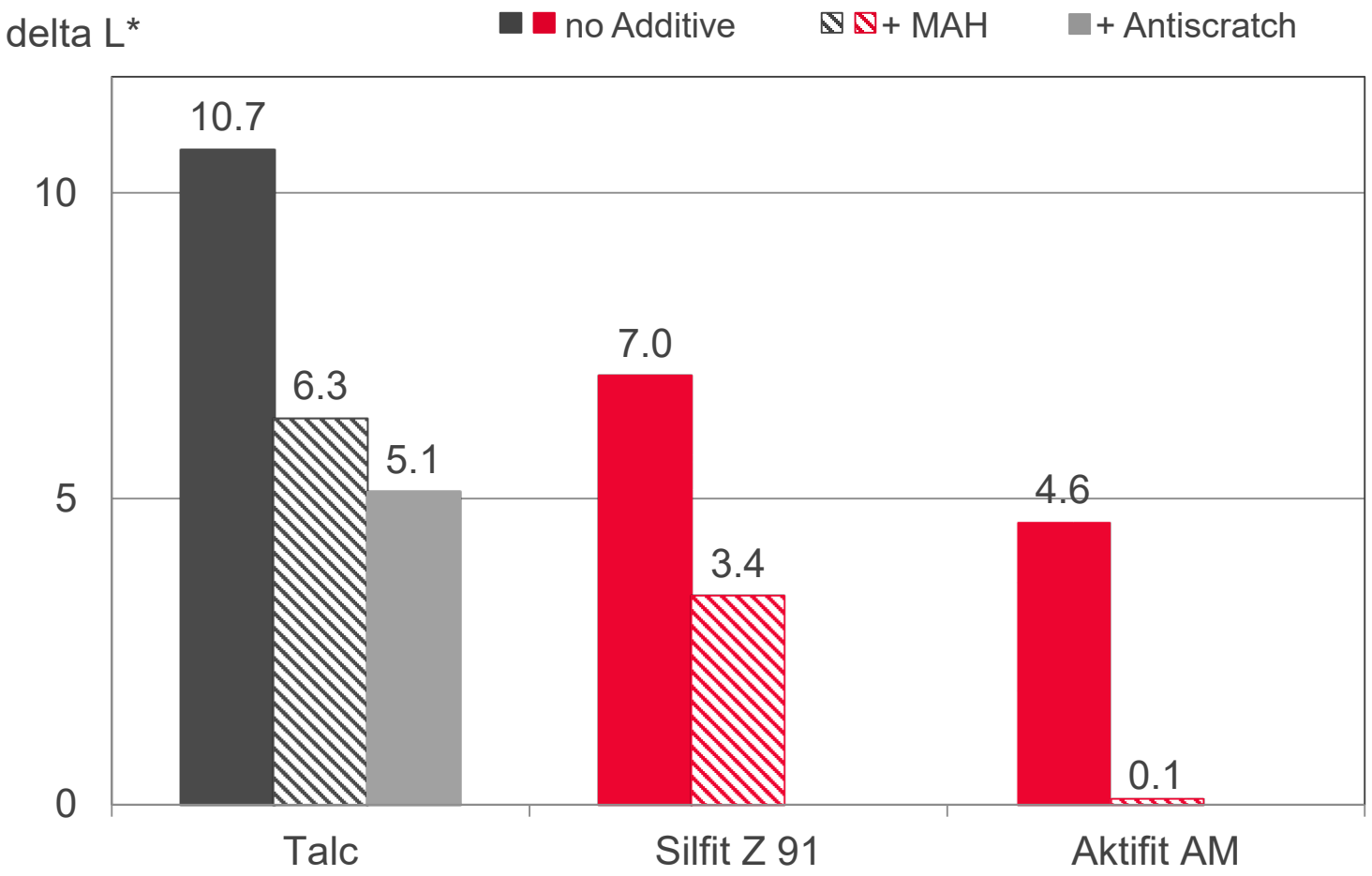


# Scratch Resistance
# K 31 (fine grain)

HOFFMANN MINERAL

VW PV 3952, 10 N



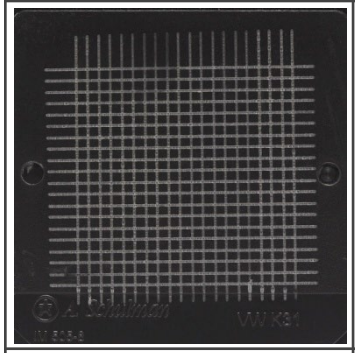
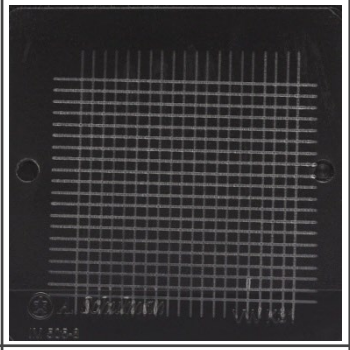
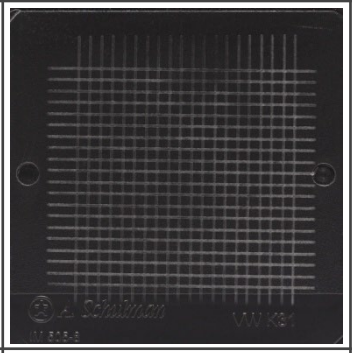


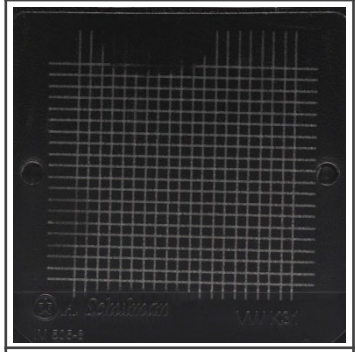
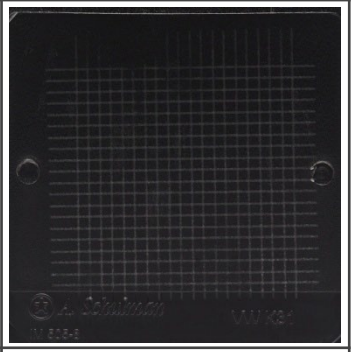
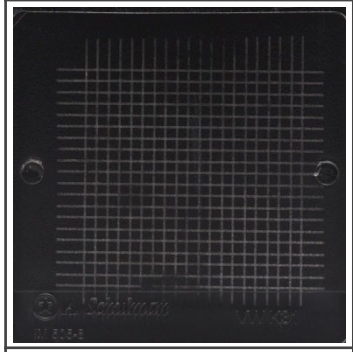
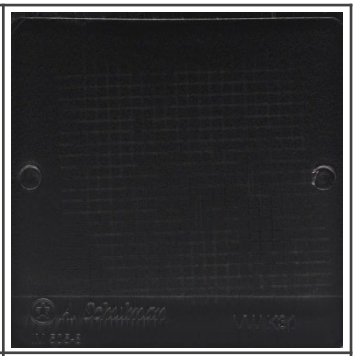
# Scratch Resistance
# K 31 (fine grain)

HOFFMANN MINERAL

VW PV 3952, 10 N



| | | |
|---|---|---|
|  |  |  |
| Talc | Talc<br>+ MAH | Talc<br>+ Antiscratch |

| | | | |
|---|---|---|---|
|  |  |  |  |
| Silfit Z 91 | Silfit Z 91<br>+ MAH | Aktifit AM | Aktifit AM<br>+ MAH |

# Formulations PP Homo 40 % Filler

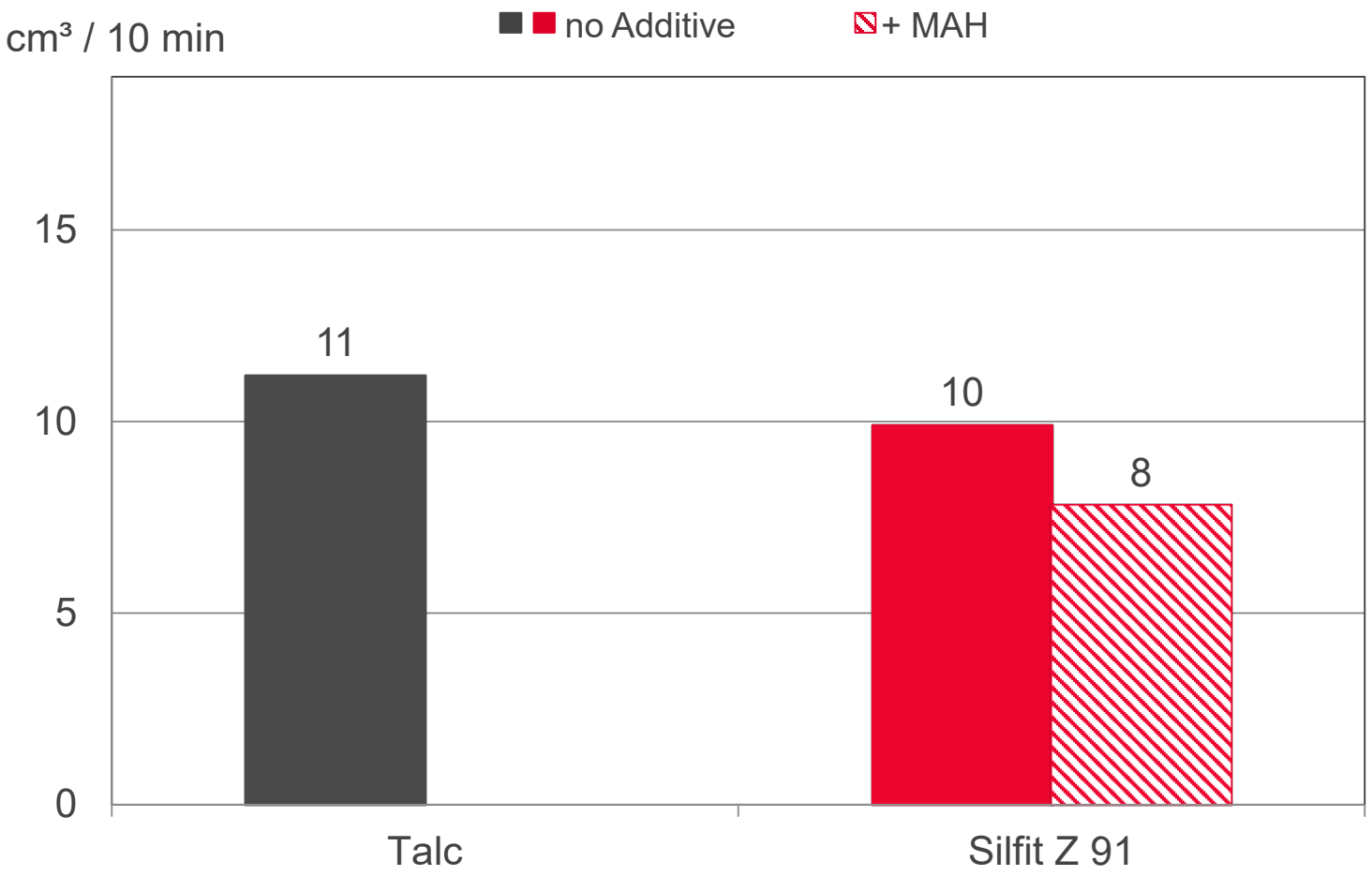


| | Talc | Silfit Z 91 | |
|---|---|---|---|
| | --- | --- | MAH |
| **PP Homopolymer** MFR 17 g/10 min (230 °C, 2.16 kg) | 57.7 | 52.7 | 57.7 |
| **Stabilizer and black color batch** | 2.3 | 2.3 | 2.3 |
| **Talc** ultrafine | 40 | --- | --- |
| **Silfit Z 91** | --- | 40 | 40 |
| **MAH-modified PP** (1 % MAH) | --- | --- | 5 |
| **Total** | **100** | **100** | **100** |

# Melt Volume-flow Rate

DIN ISO 1133; 230 °C, 2.16 kg

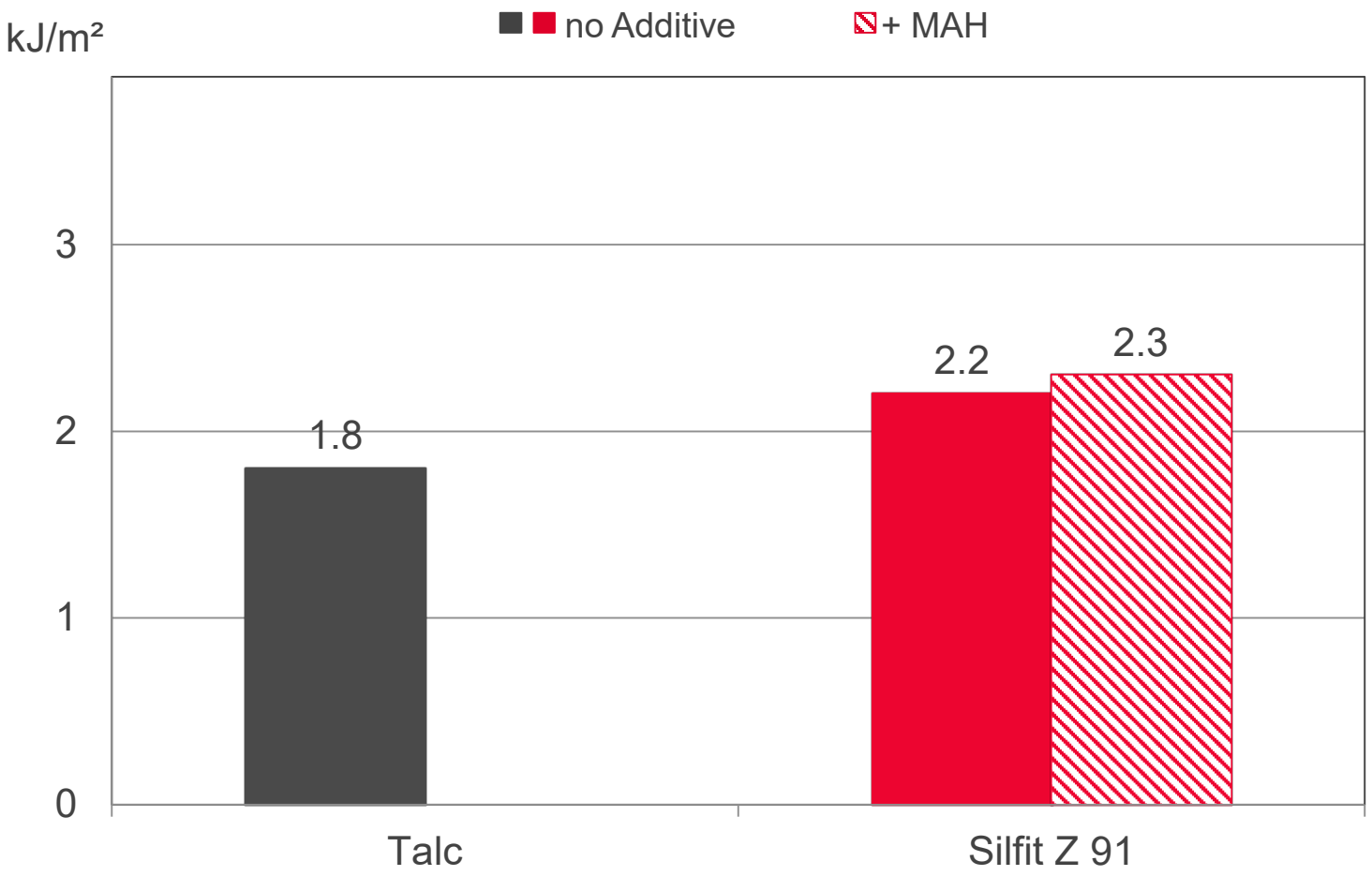




# Charpy
# Notched Impact Strength 23 °C

DIN EN ISO 179-1/1eA

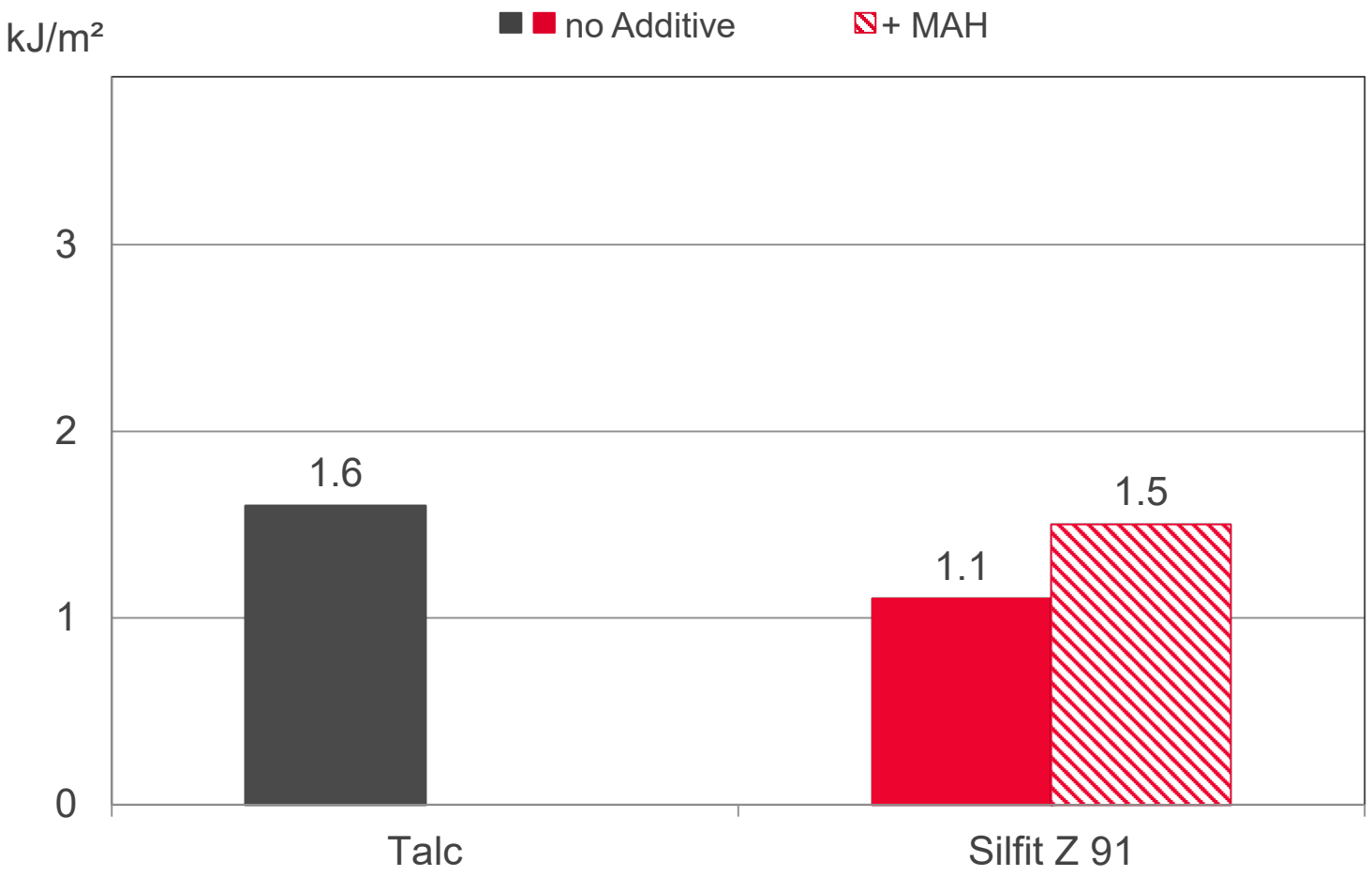




# Charpy
# Notched Impact Strength -30 °C

DIN EN ISO 179-1/1eA

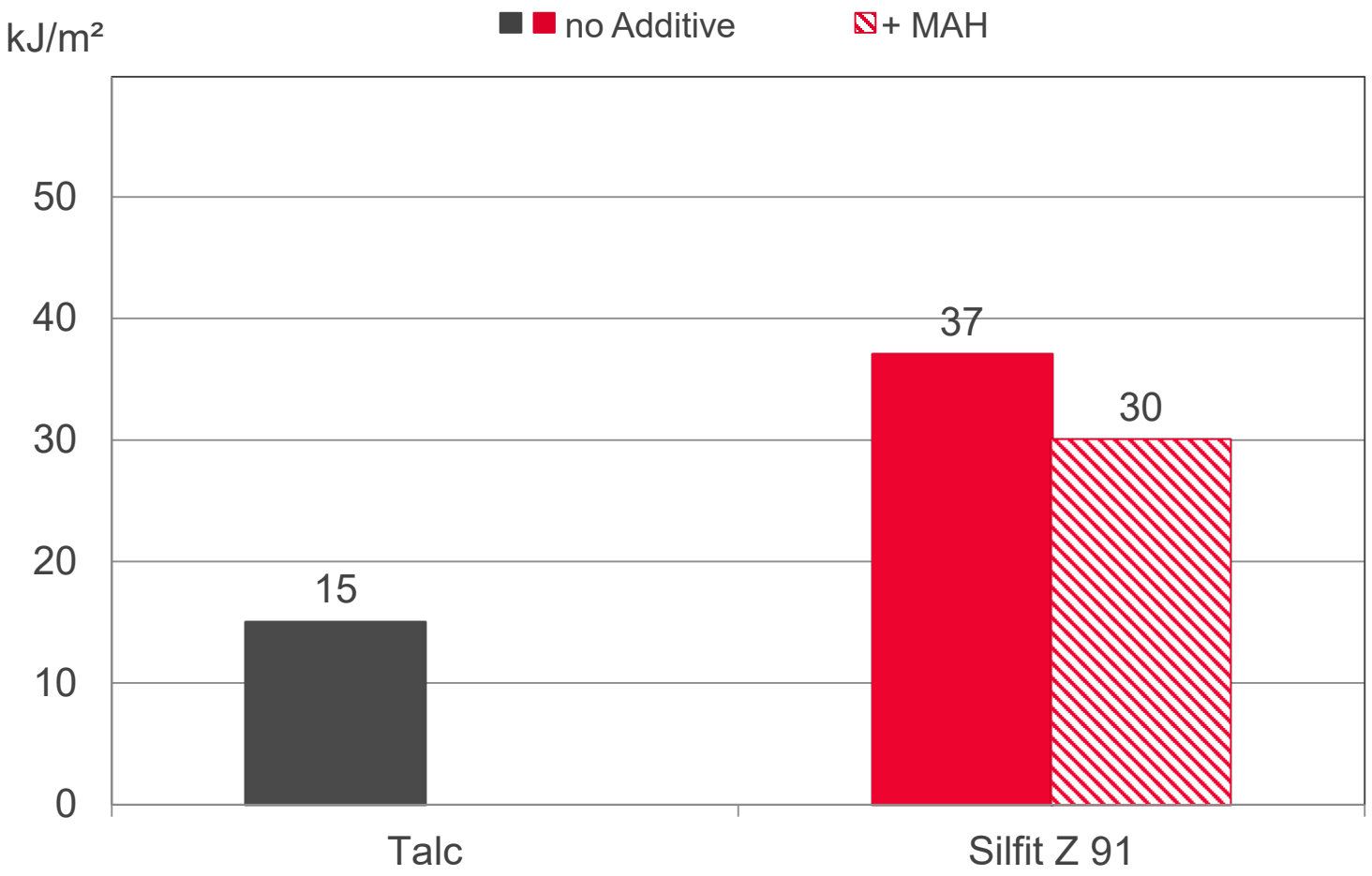




# Charpy
# Impact Strength 23 °C

DIN EN ISO 179-1/1eU

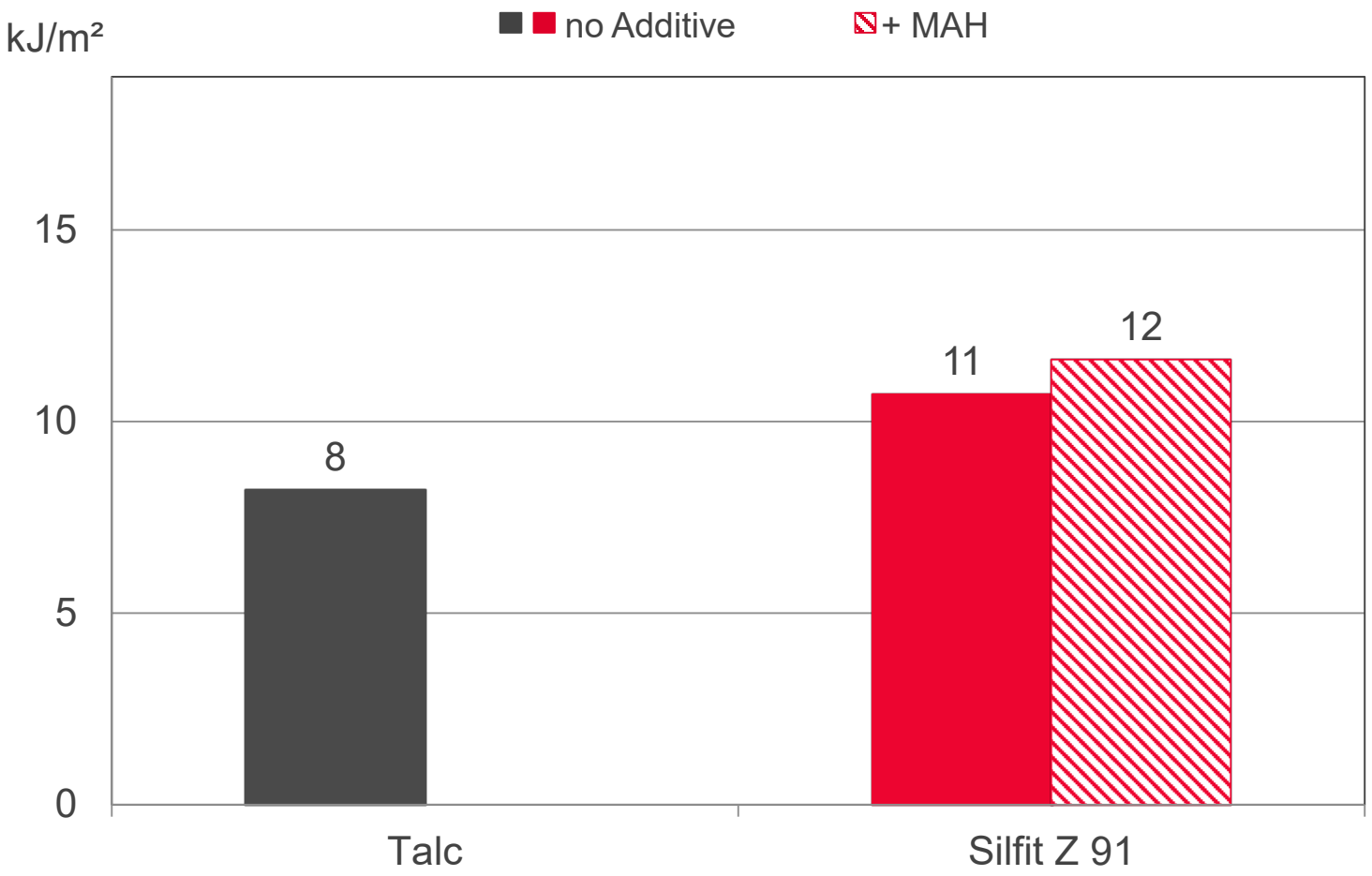




# Charpy
# Impact Strength -30 °C

DIN EN ISO 179-1/1eU

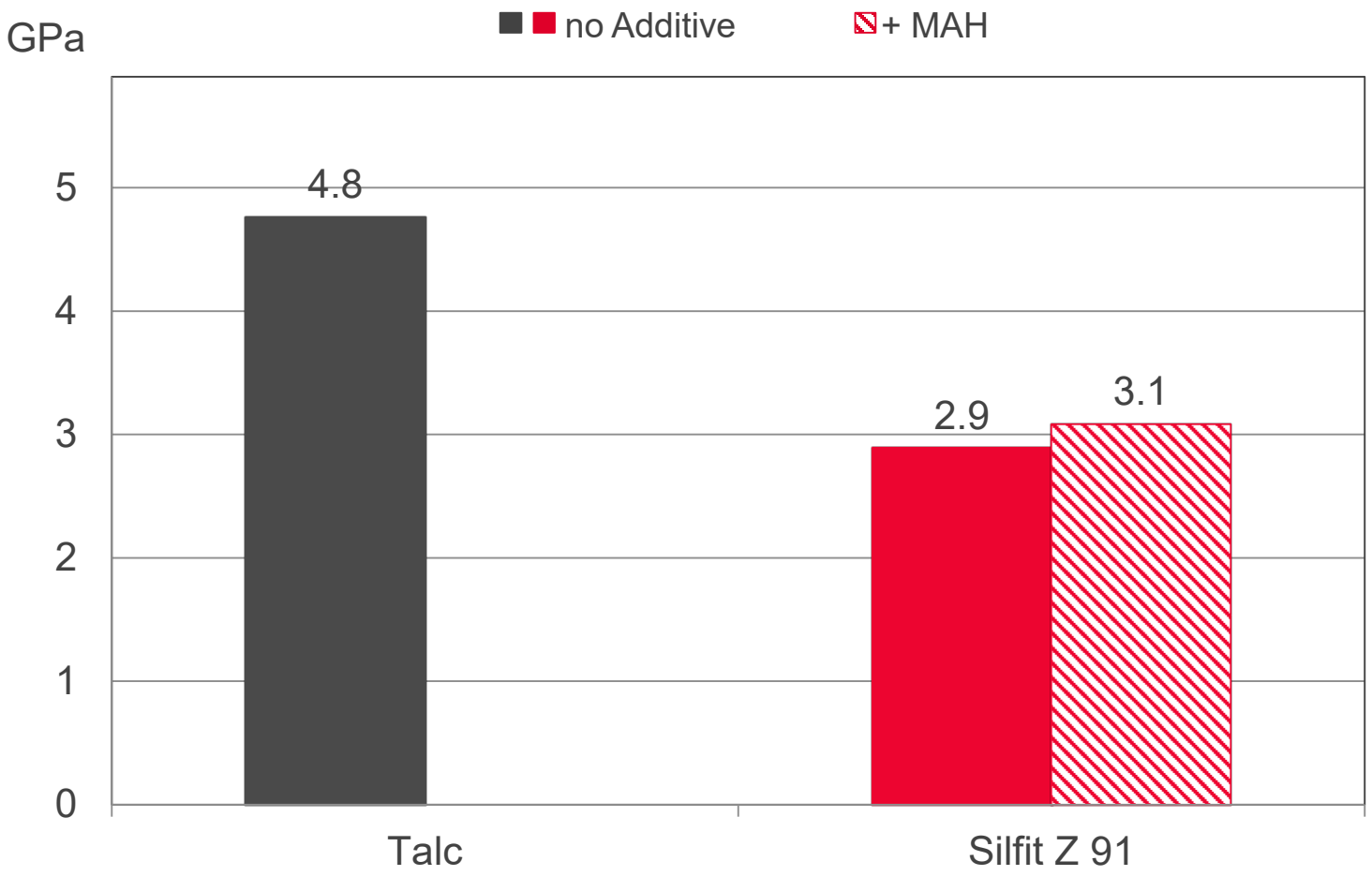




# Tensile Modulus

DIN EN ISO 527-1,-2; 1 mm/min

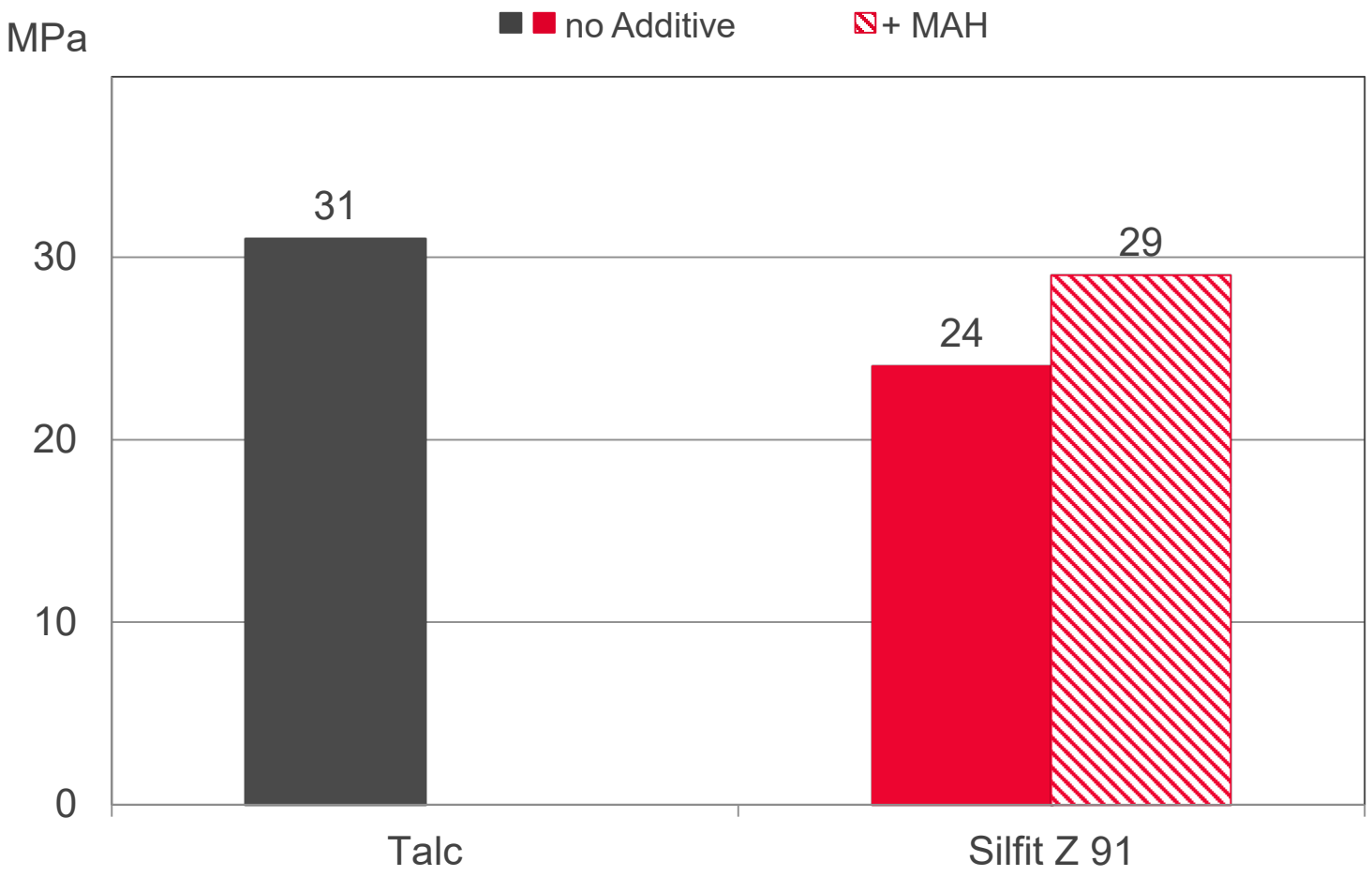




# Tensile Strength

DIN EN ISO 527-1,-2; 5 mm/min

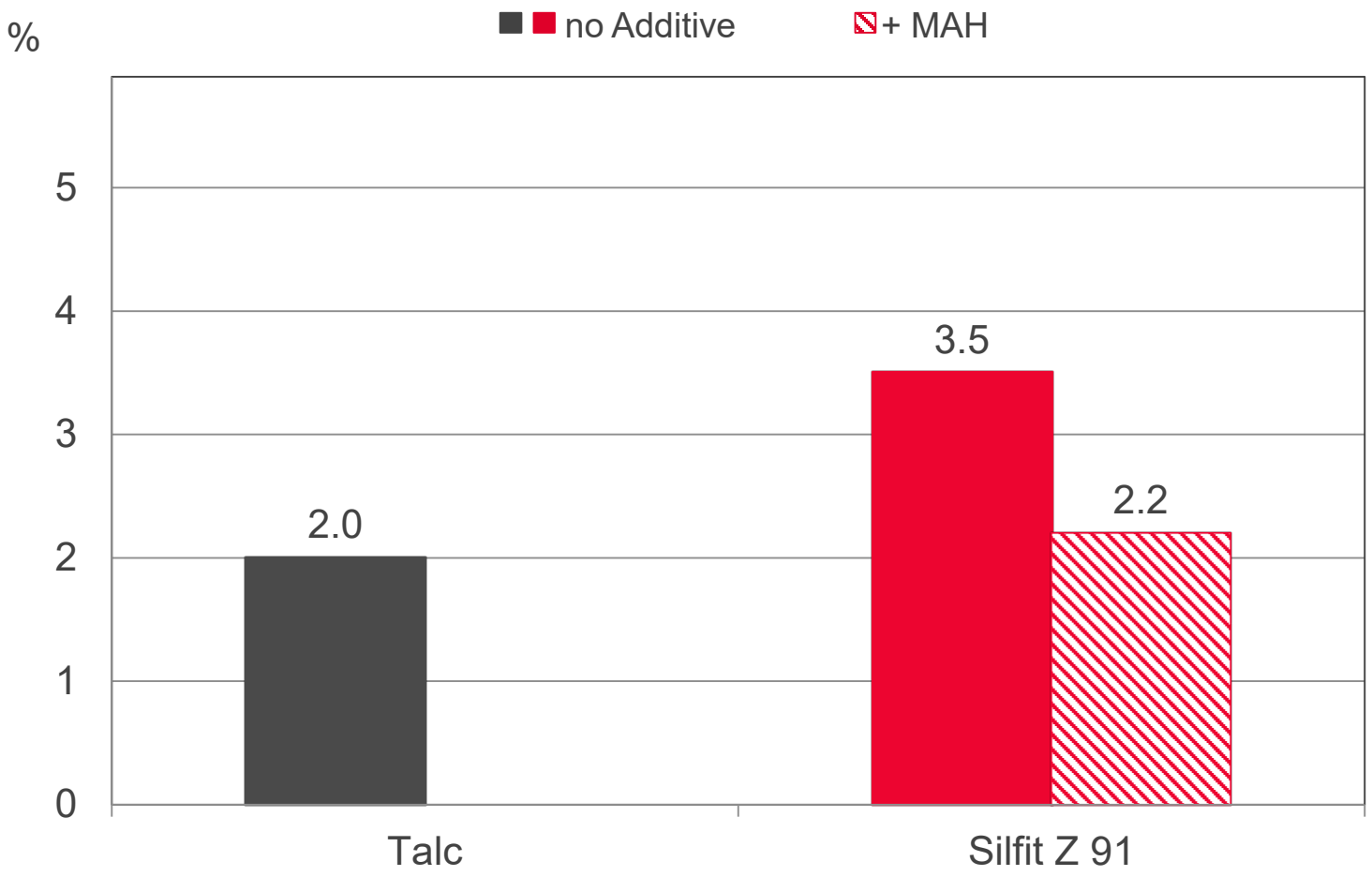




# Tensile Strain at Break

DIN EN ISO 527-1,-2; 5 mm/min

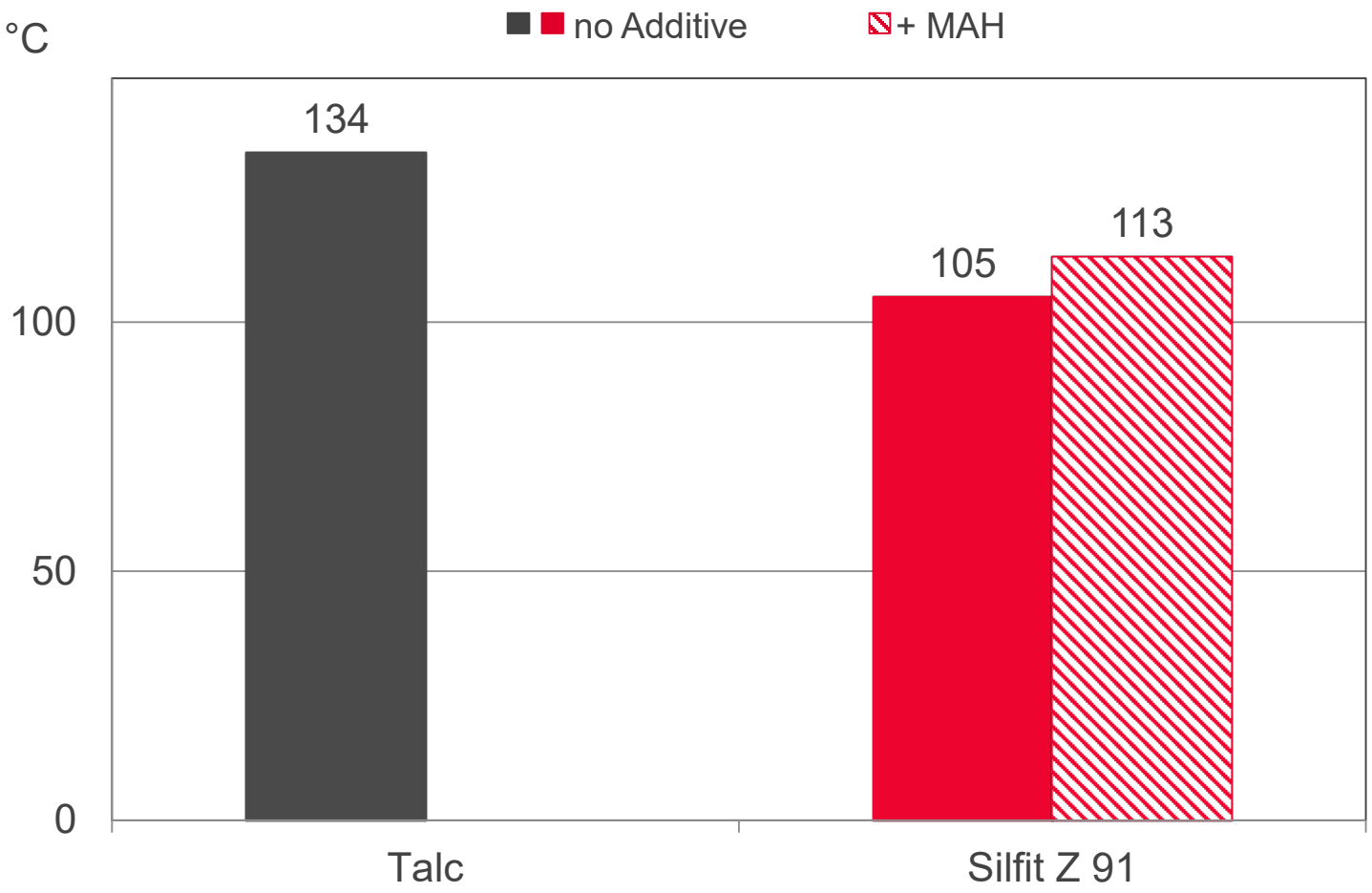




# HDT
# Heat Distortion Temperature

DIN EN ISO 75-1, -2; Method Bf (0.45 MPa)

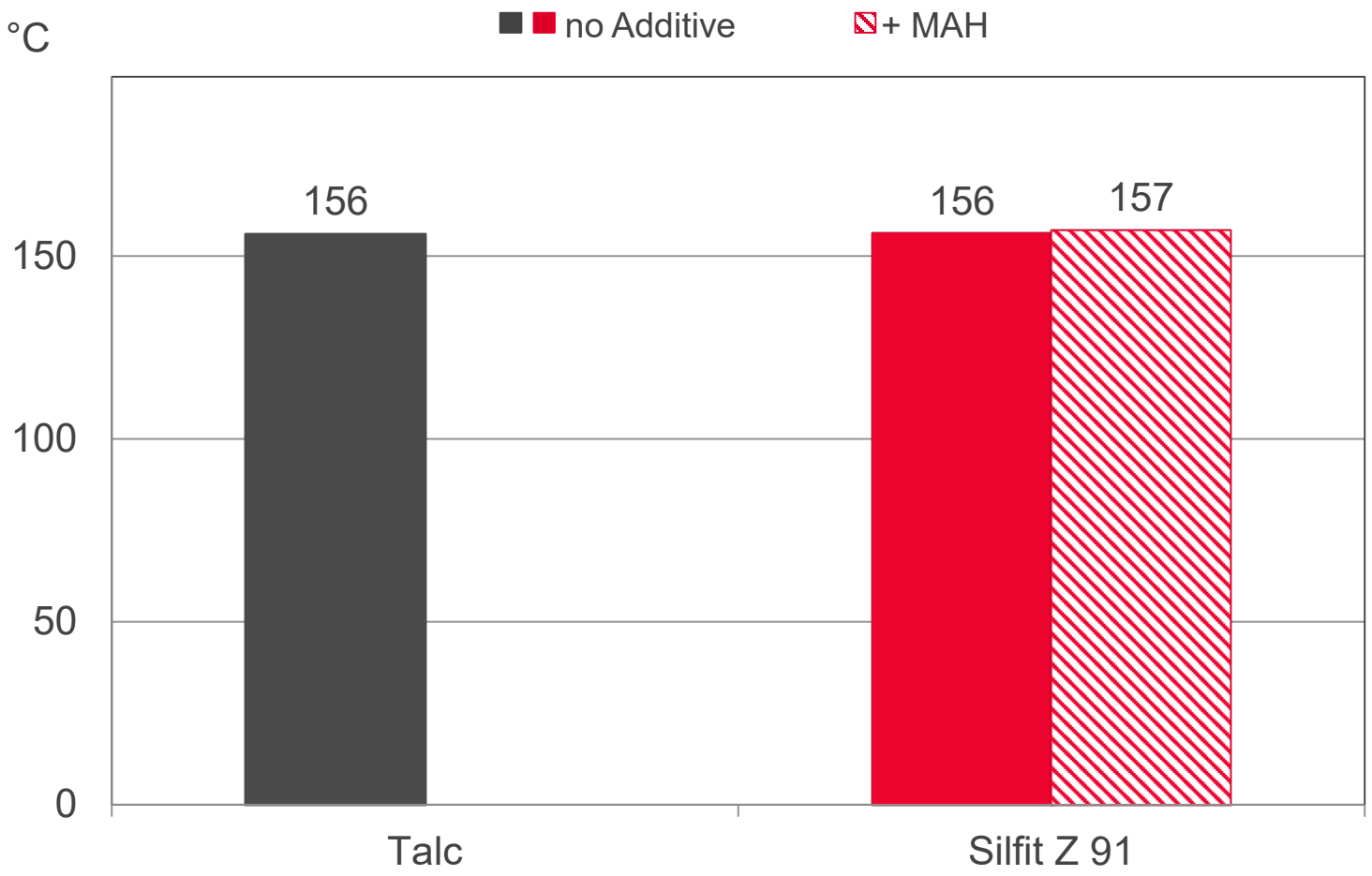




# Vicat Softening Point

DIN EN ISO 306; Method A50 (50 °C/h, 10 N)

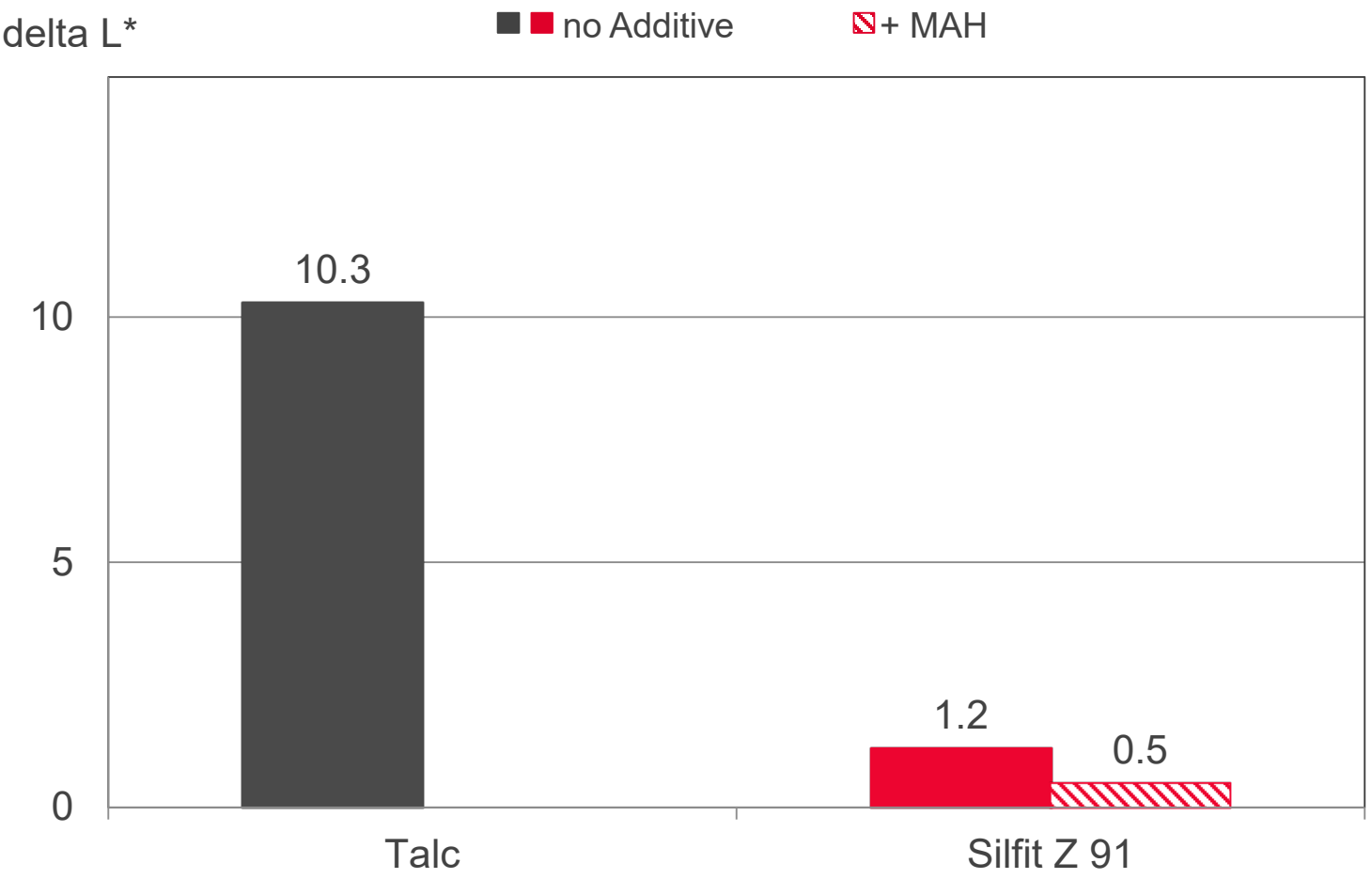




# Scratch Resistance
# K 31 (fine grain)

VW PV 3952, 10 N





# Summary
# General



Compared to talc, Calcined Neuburg Siliceous Earth shows in PP:

- similar melt flow rate
- somewhat lower heat deflection
- same softening temperature
- lower stiffness
- somewhat lower strength

+ higher tensile strain at break

+ higher impact strength, even at low-temperature; further increase by adding MAH to Aktifit AM

+ improved indentation by writing

+ improved scratch resistance, especially with the combination Aktifit AM + MAH

# Summary in Detail



- Silfit Z 91 and especially Aktifit AM improve impact strength and scratch resistance.
- Addition of MAH-modified PP results in a further improvement of the scratch resistance, especially with Aktifit AM; further effects: improvement of tensile strength/yield stress and strain properties as well as of HDT and impact strength.
- In PP homopolymer Silfit Z 91 already performs well.
- Comparing 40 % CSNE to 20 % talc, Silfit Z 91 and Aktifit AM outperform talc regarding scratch resistance and strain properties (Aktifit AM + MAH) despite the higher filler content.
- This is also valid for PP homopolymer.
- As a result of the high impact resistance with Silfit Z 91 and Aktifit AM, in compounds designed for very high impact strength, special impact modifiers can be reduced, which also should help to alleviate differences in stiffness.

# We supply material for good ideas!

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# PP Copo 20 % Filler
# Summary table (1)



| | | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|---|
| | **Additive** | **---** | **MAH** | **Anti-scratch** | **---** | **MAH** | **---** | **MAH** |
| **Density** | g/cm³ | 1.02 | 1.05 | 1.05 | 1.04 | 1.05 | 1.05 | 1.04 |
| **Filler content** | % | 20.5 | 22.4 | 20.8 | 21.1 | 20.8 | 20.9 | 20.7 |
| **Melt volume-flow rate** | cm³/10min | 17.2 | 13.9 | 18.0 | 20.2 | 15.9 | 17.8 | 15.5 |
| **Heat deflection** | | | | | | | | |
| HDT (Bf; 0.45 MPa) | °C | 113 | 112 | 97 | 88 | 90 | 96 | 99 |
| Vicat A (50 °C/h; 10 N) | °C | 146 | 147 | 147 | 147 | 147 | 147 | 150 |
| **Indentation by writing delta gloss 60°** | | | | | | | | |
| K 09 (coarse grain) | DGG | 1.0 | 1.2 | 0.9 | 0.6 | 0.8 | 0.6 | 0.7 |
| K 31 (fine grain) | DGG | 1.1 | 0.7 | 0.6 | 0.8 | 0.5 | 0.5 | 0.5 |
| **Scratch resistance delta brightness L*** | | | | | | | | |
| K 09 (coarse grain) | DL | 4.6 | 3.7 | 2.7 | 4.0 | 3.1 | 3.6 | -0.1 |
| K 31 (fine grain) | DL | 5.1 | 2.5 | 2.2 | 3.4 | 2.5 | 2.7 | -0.6 |

# PP Copo 20 % Filler
# Summary table (2)



| | | | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|---|---|
| | | Additive | --- | MAH | Anti-scratch | --- | MAH | --- | MAH |
| **Tensile modulus 1 mm/min** | | GPa | 2.7 | 2.7 | 2.6 | 1.9 | 2.0 | 1.9 | 1.9 |
| **Tensile test 5 mm/min** | | | | | | | | | |
| Tensile strength | | MPa | 24.0 | 25.8 | 22.3 | 21.0 | 22.5 | 19.7 | 23.1 |
| Tensile strain at break | | % | 13 | 10 | 18 | 70 | 32 | 18 | 27 |
| **Tensile test 50 mm/min** | | | | | | | | | |
| Yield stress | | MPa | 25.7 | 27.8 | 24.0 | 22.6 | 26.1 | 23.9 | 28.0 |
| Yield strain | | % | 2.9 | 3.0 | 2.9 | 3.3 | 3.3 | 3.5 | 3.9 |
| Nominal strain at break | | % | 13 | 8 | 15 | 35 | 20 | 27 | 25 |
| **Notched impact strength** | | | | | | | | | |
| Charpy 1eA | 23 °C | kJ/m² | 5.6 | 3.6 | 4.8 | 6.1 | 6.0 | 6.1 | 5.7 |
| Charpy 1eA | -30 °C | kJ/m² | 1.8 | 1.3 | 1.7 | 1.1 | 1.5 | 1.6 | 2.1 |
| **Impact strength** | | | | | | | | | |
| Charpy 1eU (4 J) | 23 °C | kJ/m² | 54 | 32 | 45 | NB | 70 | NB | NB |
| Charpy 1eU (7.5 J) | 23 °C | kJ/m² | --- | --- | --- | 145 | --- | 115 | 126 |
| Charpy 1eU | -30 °C | kJ/m² | 19 | 15 | 16 | 32 | 28 | 34 | 48 |

# PP Copo 40 % Filler
# Summary table (1)



| | | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|---|
| | Additive | --- | MAH | Anti-scratch | --- | MAH | --- | MAH |
| **Density** | g/cm³ | 1.24 | 1.24 | 1.25 | 1.21 | 1.19 | 1.24 | 1.23 |
| **Filler content** | % | 40.2 | 40.1 | 39.8 | 39.3 | 37.9 | 40.4 | 40.2 |
| **Melt volume-flow rate** | cm³/10min | 12.6 | 11.5 | 10.7 | 12.3 | 11.2 | 11.5 | 8.4 |
| **Heat deflection** | | | | | | | | |
| HDT (Bf; 0.45 MPa) | °C | 123 | 126 | 114 | 104 | 103 | 98 | 111 |
| Vicat A (50 °C/h; 10 N) | °C | 150 | 154 | 149 | 150 | 153 | 151 | 156 |
| **Indentation by writing delta gloss 60°** | | | | | | | | |
| K 09 (coarse grain) | DGG | 1.8 | 1.1 | 1.3 | 1.2 | 0.8 | 1.0 | 0.9 |
| K 31 (fine grain) | DGG | 1.2 | 1.7 | 1.2 | 0.7 | 1.2 | 0.9 | 0.7 |
| **Scratch resistance delta brightness L*** | | | | | | | | |
| K 09 (coarse grain) | DL | 11.1 | 6.0 | 9.4 | 7.4 | 5.9 | 7.1 | 0.9 |
| K 31 (fine grain) | DL | 10.7 | 6.3 | 5.1 | 7.0 | 3.4 | 4.6 | 0.1 |

# PP Copo 40 % Filler
# Summary table (2)



| | | | Talc | | | Silfit Z 91 | | Aktifit AM | |
|---|---|---|---|---|---|---|---|---|---|
| | | Additive | --- | MAH | Anti-scratch | --- | MAH | --- | MAH |
| **Tensile modulus 1 mm/min** | | GPa | 4.5 | 4.7 | 4.2 | 2.7 | 2.7 | 2.4 | 2.5 |
| **Tensile test 5 mm/min** | | | | | | | | | |
| Tensile strength | | MPa | 26.0 | 32.1 | 22.8 | 20.9 | 26.0 | 21.0 | 27.0 |
| Tensile strain at break | | % | 2.0 | 2.1 | 2.8 | 2.2 | 5.0 | 3.0 | 20.0 |
| **Tensile test 50 mm/min** | | | | | | | | | |
| Yield stress | | MPa | 27.6 | 34.5 | 24.7 | 21.8 | 27.4 | 22.0 | 29.2 |
| Yiled strain | | % | 1.7 | 2.4 | 1.6 | 1.6 | 2.4 | 1.8 | 2.8 |
| Nominal strain at break | | % | 2.7 | 2.9 | 4.0 | 1.6 | 4.8 | 4.0 | 17.5 |
| **Notched impact strength** | | | | | | | | | |
| Charpy 1eA | 23 °C | kJ/m² | 2.1 | 3.0 | 2.2 | 3.8 | 2.5 | 4.5 | 5.7 |
| Charpy 1eA | -30 °C | kJ/m² | 1.0 | 1.2 | 0.8 | 0.9 | 1.6 | 1.6 | 2.2 |
| **Impact strength** | | | | | | | | | |
| Charpy 1eU | 23 °C | kJ/m² | 15 | 20 | 15 | 78 | 49 | 69 | 93 |
| Charpy 1eU | -30 °C | kJ/m² | 9 | 12 | 8 | 17 | 21 | 18 | 38 |

# PP Homo 40 % Filler
# Summary table



| | | Talc | Silfit Z 91 | |
|---|---|---|---|---|
| | **Additive** | **---** | **---** | **MAH** |
| **Density** | g/cm³ | 1.22 | 1.23 | 1.23 |
| **Filler content** | % | 38.5 | 39.6 | 39.7 |
| **Melt volume-flow rate** | cm³/10min | 11.2 | 9.9 | 7.8 |
| **Heat deflection** | | | | |
| HDT (Bf; 0.45 MPa) | °C | 134 | 105 | 113 |
| Vicat A (50 °C/h; 10 N) | °C | 156 | 156 | 157 |
| **Tensile modulus 1 mm/min** | GPa | 4.8 | 2.9 | 3.1 |
| **Tensile test 5 mm/min** | | | | |
| Tensile strength | MPa | 31 | 24 | 29 |
| Tensile strain at break | % | 2.0 | 3.5 | 2.2 |
| **Notched impact strength** | | | | |
| Charpy 1eA 23 °C | kJ/m² | 1.8 | 2.2 | 2.3 |
| Charpy 1eA -30 °C | kJ/m² | 1.6 | 1.1 | 1.5 |
| **Impact strength** | | | | |
| Charpy 1eU 23 °C | kJ/m² | 15 | 37 | 30 |
| Charpy 1eU -30 °C | kJ/m² | 8.2 | 10.7 | 11.6 |
| **Scratch resistance delta brightness L*** | | | | |
| K 31 (fine grain) | DL | 10.3 | 1.2 | 0.5 |