Neuburg Siliceous Earth
in combination with ATH flame retardancy
in silicone rubber

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Aluminium hydroxide – short ATH – is commonly used as a filler for flame retardant applications due to the following mechanisms:

- Thermal decomposition of ATH into Al_2O_3 and H_2O
  - → Endothermic reaction detracts thermal energy
  - → H_2O cools down the surface and dilutes flammable gases
  - → Oxide residue
    - → Adsorbs sooty particles
    - → Protective layer, which disabels release of low molecular weight decomposition products and flammable gases
    - → Heat barrier, which protects intact elastomer

Status Quo

Neuburg Siliceous Earth, as well as its calcined version, finds use as a functional filler in elastomers.

For peroxide-cured high consistency silicone rubber Aktisil Q is the best choice, since it offers the following benefits:

- Excellent extrusion properties
  - → Strongly improved dimensional stability
  - → No stickiness of uncured extrudates
- Extremely low compression set
- Very good oil resistance
- Applicable in combination with all commonly used peroxides
  - → Reduction resp. prevention of blooming of bis-(2,4-dichlorobenzoyl)-peroxide

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Status Quo

The Calcined Neuburg Siliceous Earth Silfit Z 91 is an alternative for Aktisil Q in case the following aspects have priority:

- Costs
- Color neutrality

→ By the optimized production process the typical yellowish tint of Neuburg Siliceous Earth is eliminated

- Low filler loading

Neuburg Siliceous Earth and flame retardancy:

- Dilution of flammable gases by reduction of the polymer portion with increasing filler loading

→ No active flame retardancy with Neuburg Siliceous Earth

Objective

Demonstrating possible synergisms which result from the **combination** of

Neuburg Siliceous Earth and **ATH** in silicone rubber

with regard to

- flame retardant properties
- rheological and mechanical properties.

Fillers, Characteristics

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		Benchmark		Neuburg Siliceous Earth	
		ATH	ATH silanized	Silfit Z 91	Aktisil Q
Particle size d_{50}	[µm]	1.7(1)	1.4(1)	2.0	4.9(1)
Particle size d_{97}	[µm]	6.1(1)	3.7(1)	9.7	18(1)
Oil absorption	[g/100g]	29	33	59	40
Specific surface area BET	[m²/g]	3.5(2)	3.5(2)	7.0	6.5
Functionalization		-	vinyl	-	methacryl
Calcination		-	-	yes	-

(1)determined in isopropanol (2)as specified by the manufacturer

Formulation

in phr	ATH	ATH silanized	ATH Aktisil Q	ATH Silfit Z 91
ATH	100	-	70	70
ATH silanized	-	100	-	-
Aktisil Q	-	-	30	-
Silfit Z 91	-	-	-	30
Elastosil AUX Curing Agent E	1.5			
Elastosil R 401/40	100			

Preparation and Curing of the Compound

- **Mixing**

  Open mill Ø 150 x 300 mm

  Batch volume: approx. 1000 g

  Temperature: 20 °C

  Mixing time: approx. 10 min.

- **Curing**

  Press, 5 min. / 115 °C

- **Post-cure**

  4 h / 200 °C

Flame Retardant Properties

- LOI (Limiting Oxygen Index)
- Cone Calorimeter
  - → Heat Release Rate – HRR
  - → Peak Heat Release Rate – PHRR
  - → Time to Peak Heat Release Rate – tt PHRR
  - → Average Rate of Heat Emitted – ARHE
  - → Maximum Average of Heat Emitted – MARHE
- Smoke density

LOI
Limiting Oxygen Index

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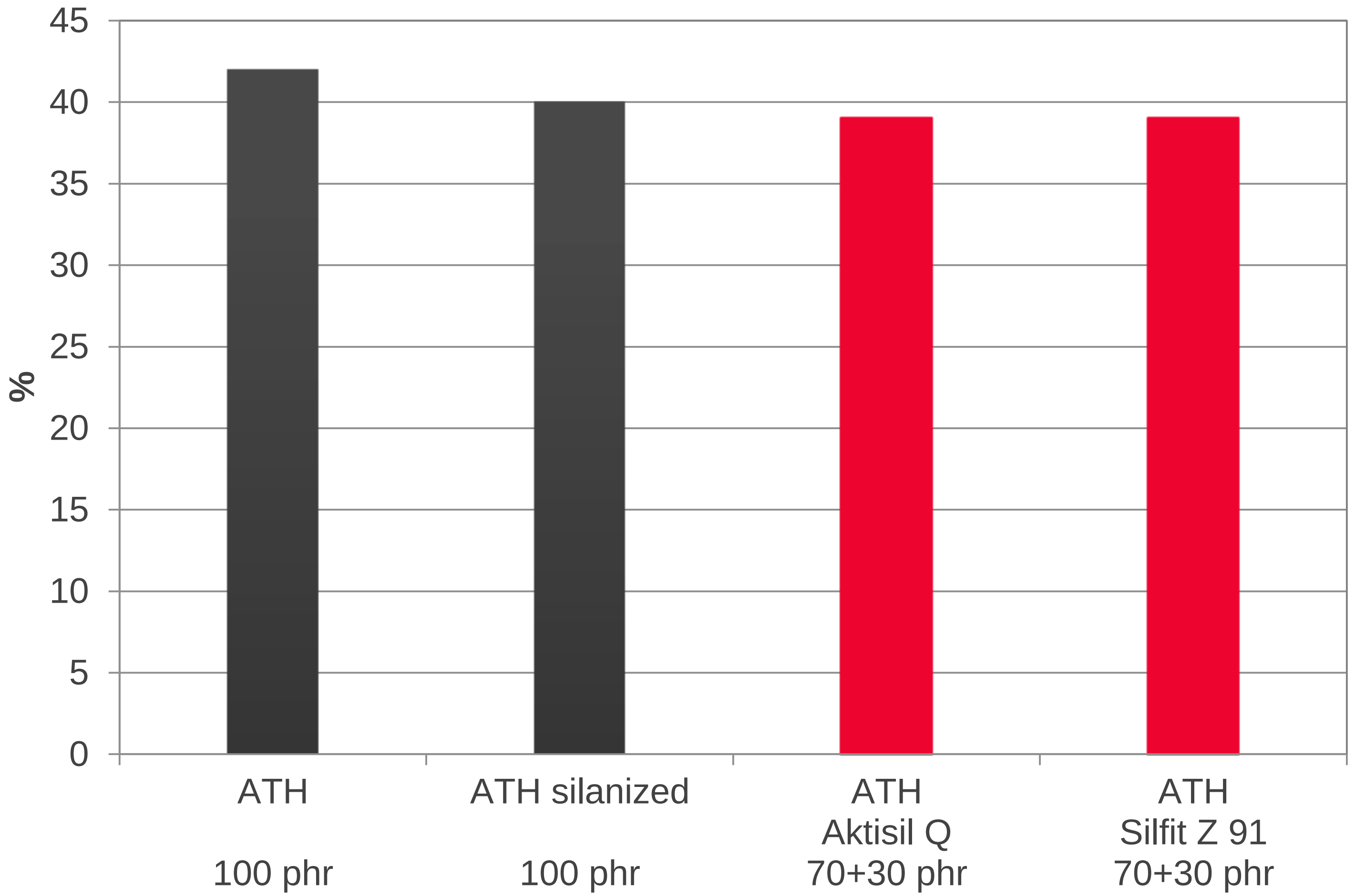
ISO 4589, press-cured



Heat Release Rate

ISO 5660, 50 kW/m²

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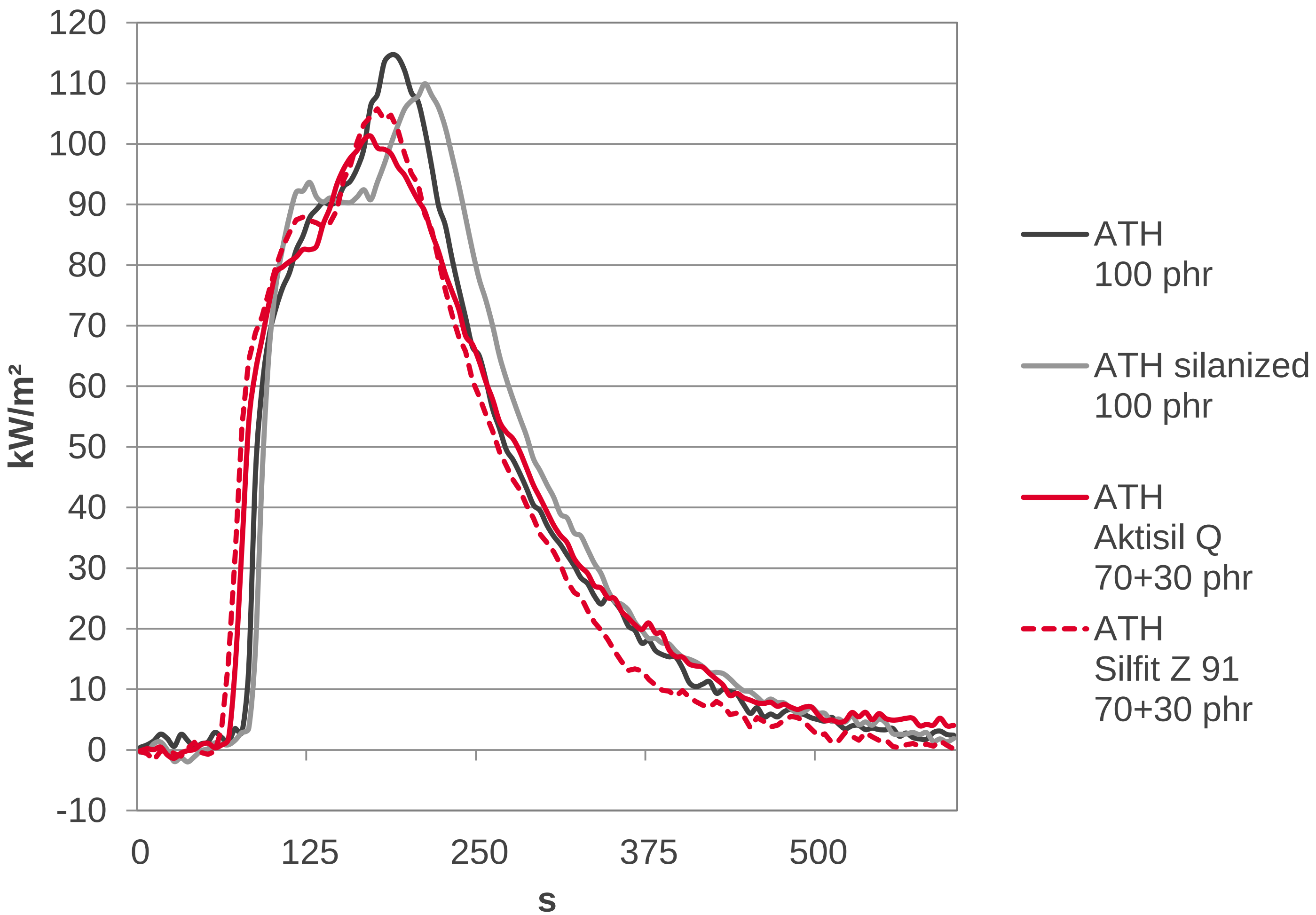

Maximum
Heat Release Rate

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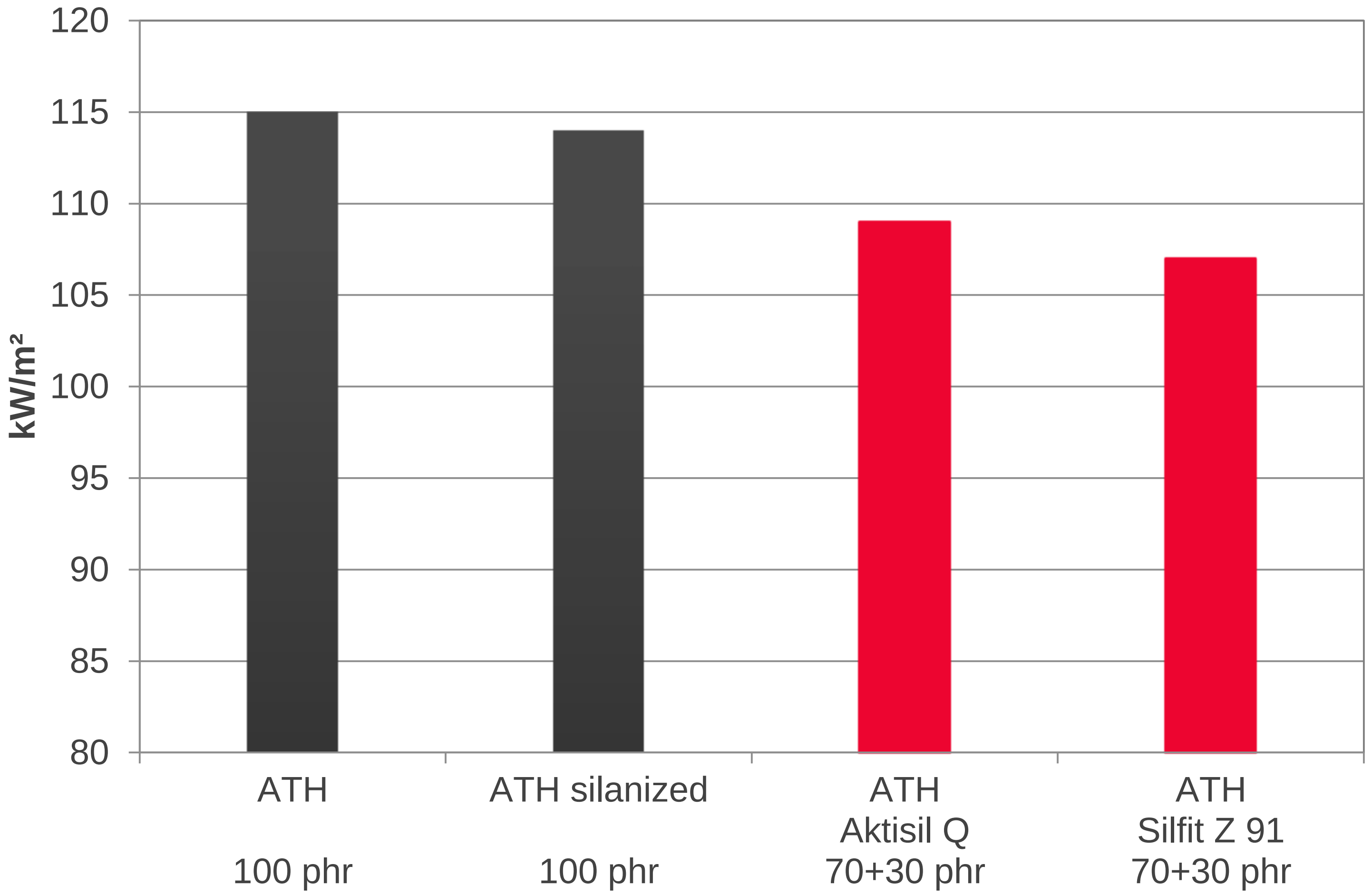
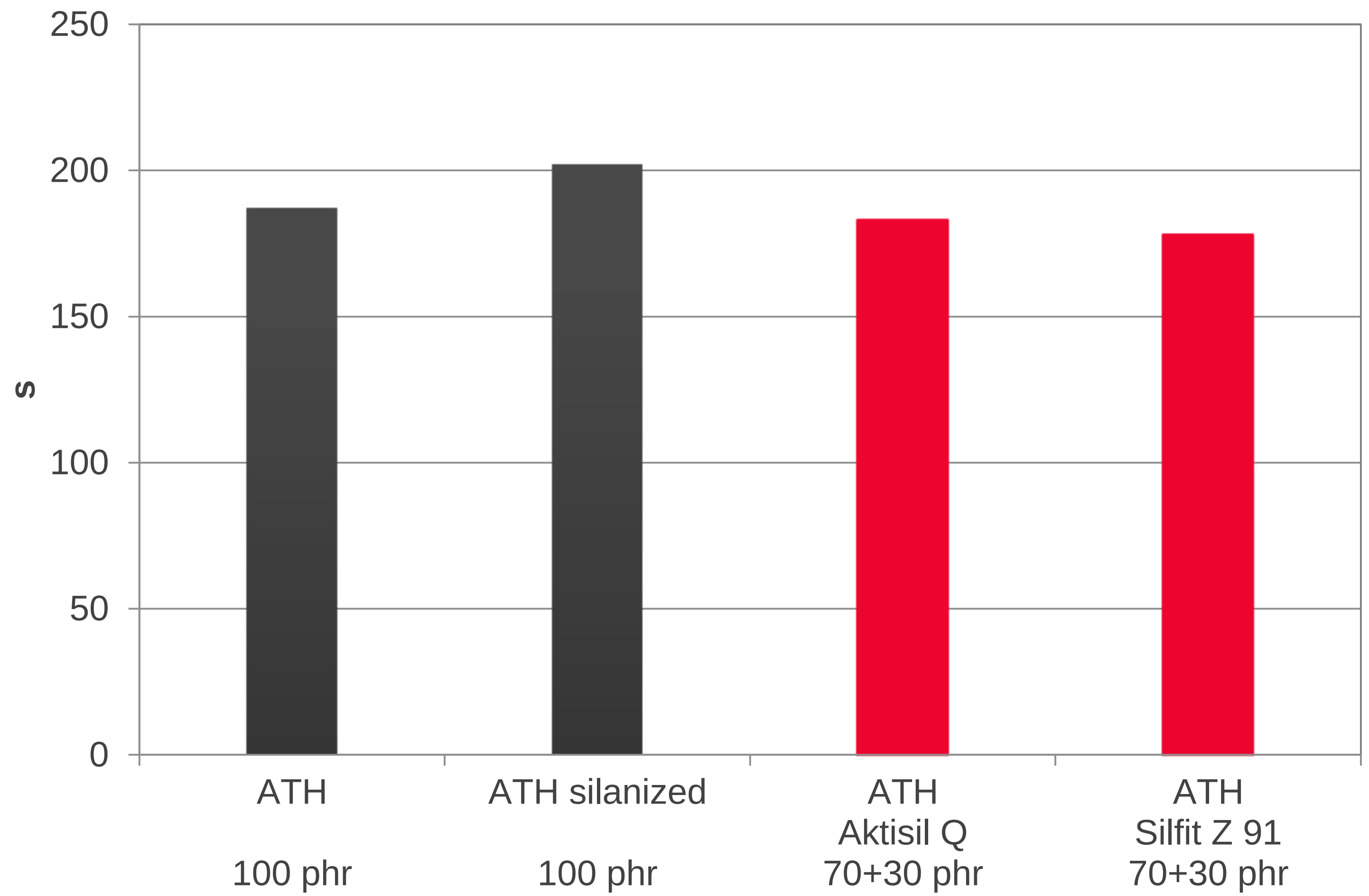
ISO 5660, 50 kW/m²



Time to Maximum Heat Release Rate

ISO 5660, 50 kW/m²



Average

Rate of Heat Emitted

ISO 5660, 50 kW/m²

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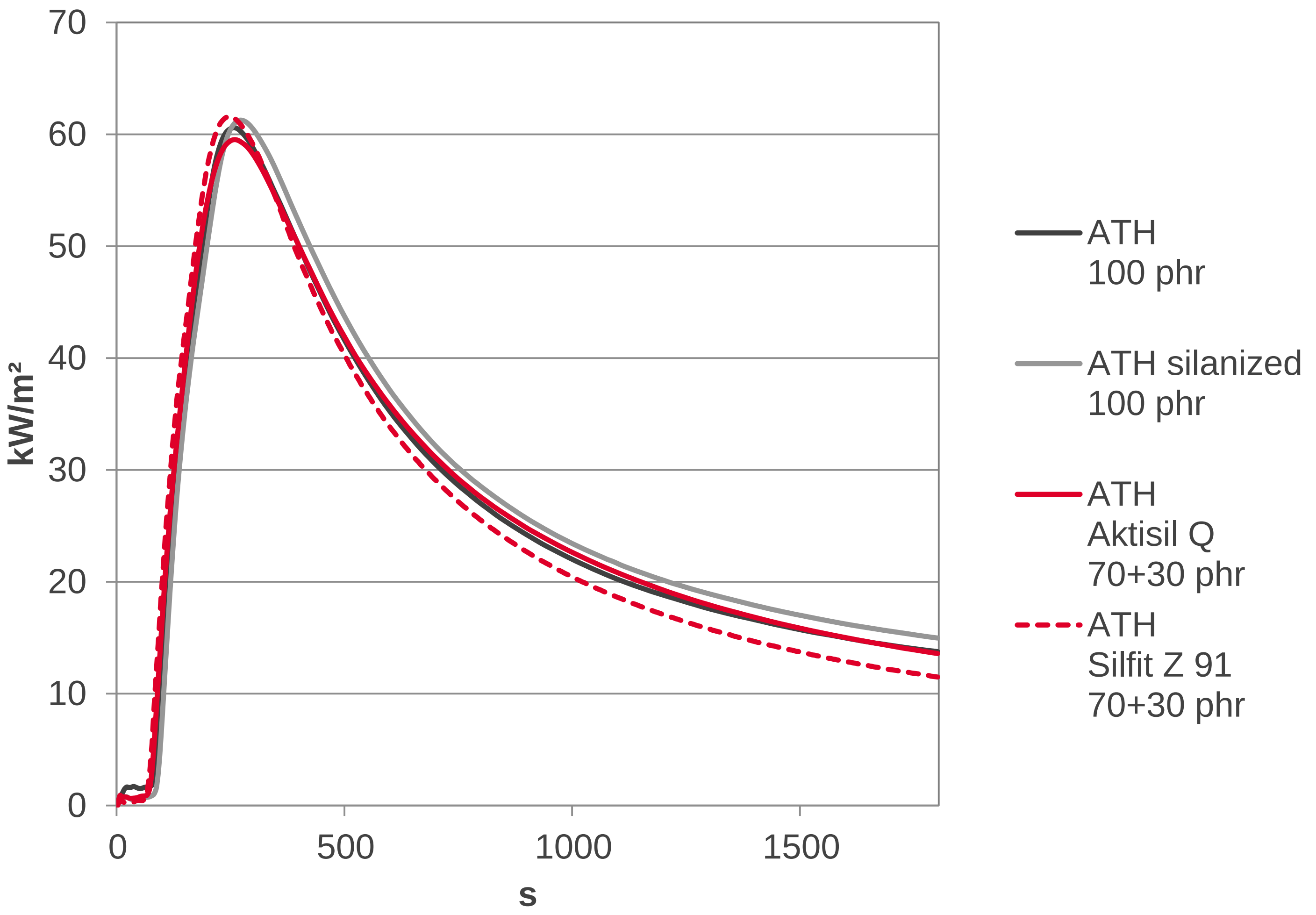

Maximum of Average Rate of Heat Emitted

ISO 5660, 50 kW/m²

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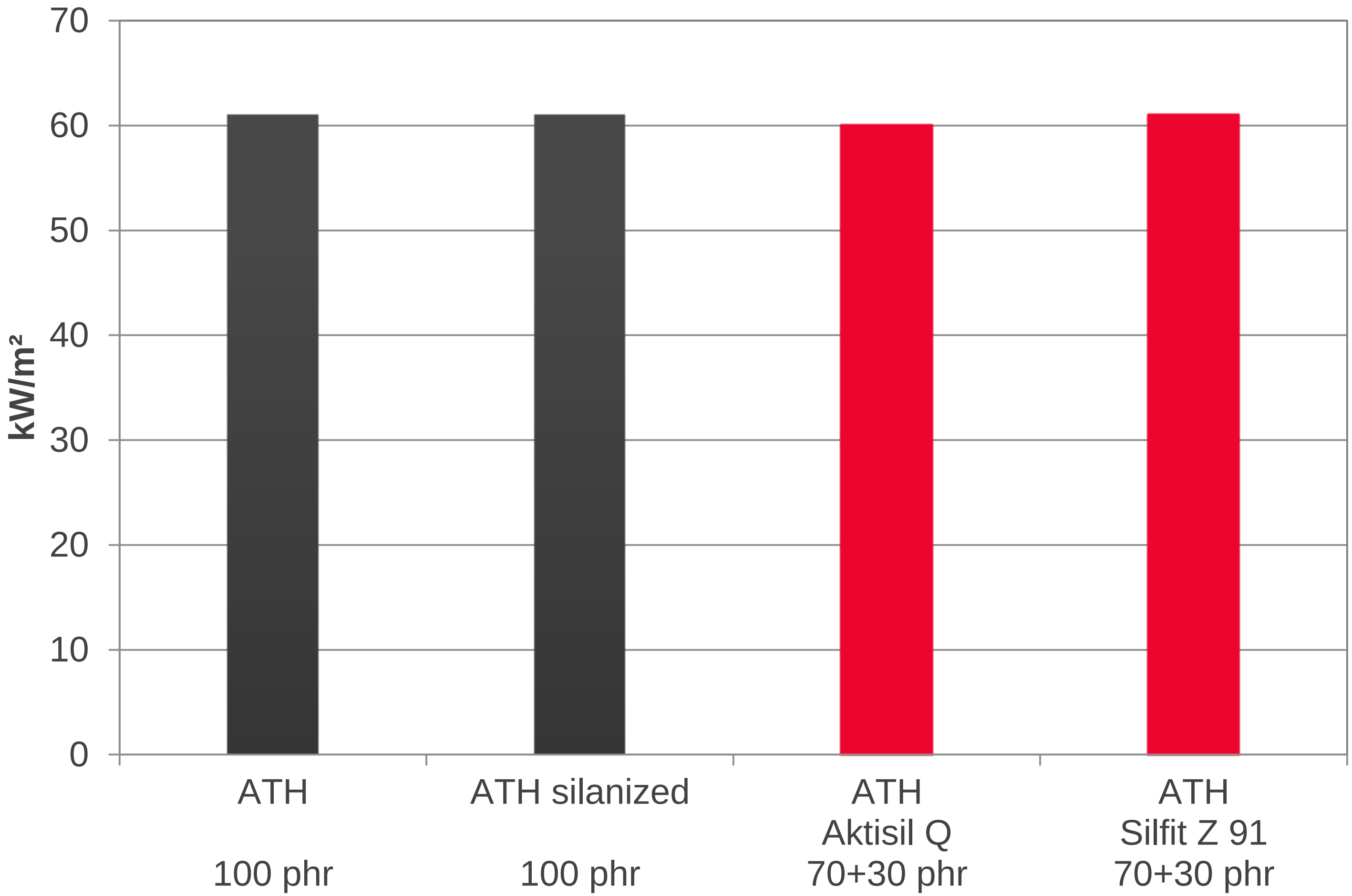
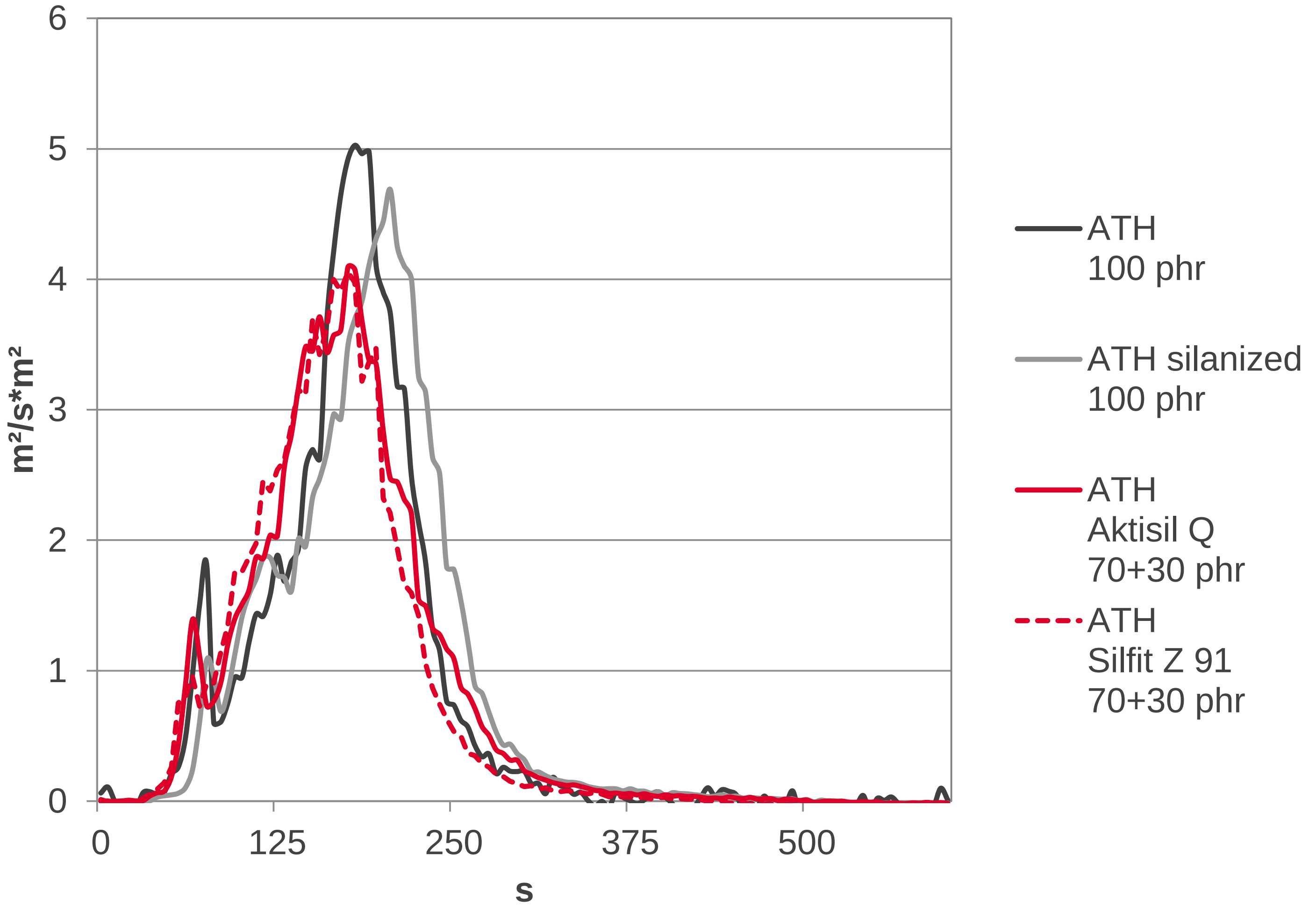


Smoke Density

ISO 5660, 50 kW/m²

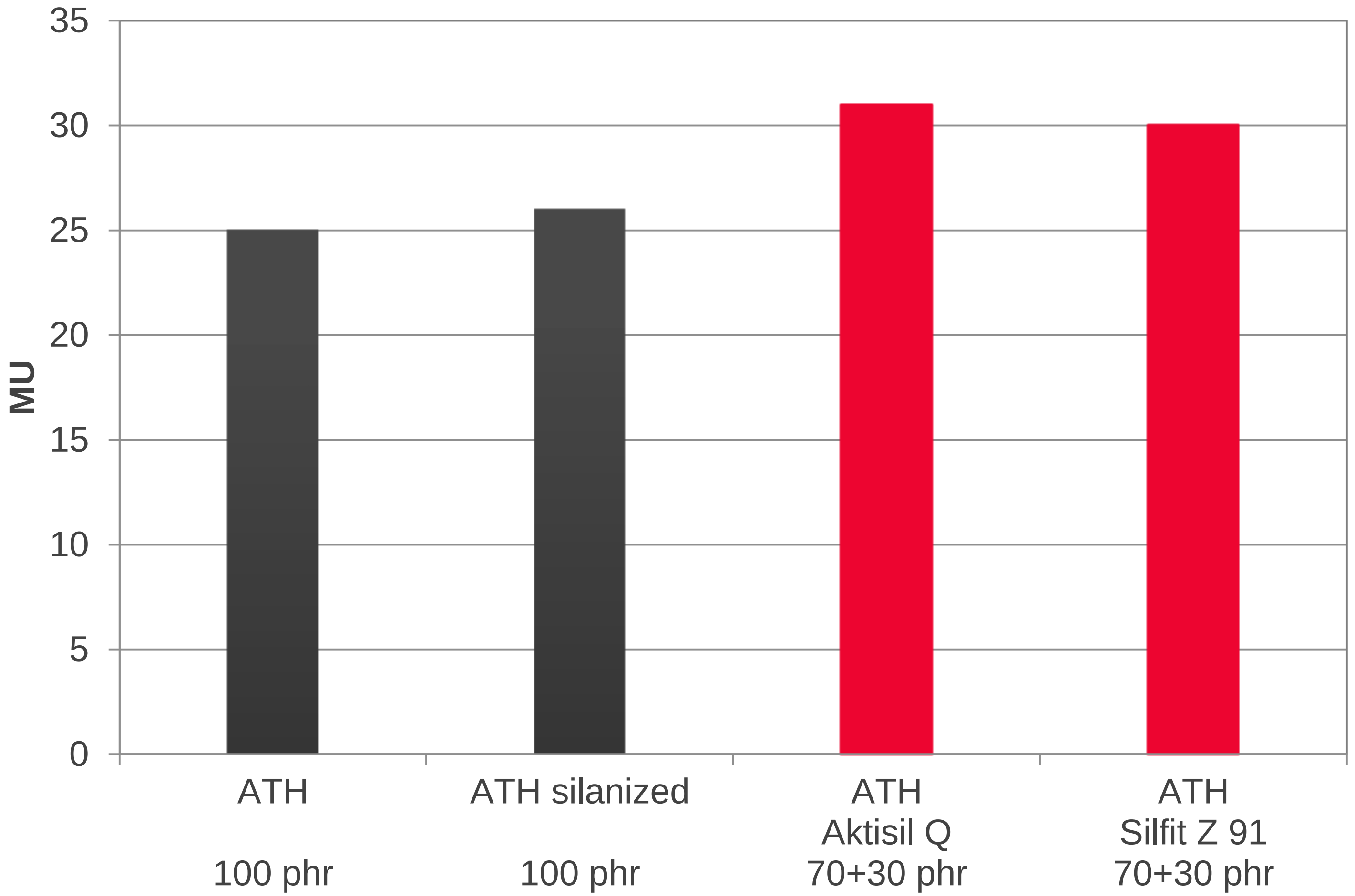


Rheology and Mechanical Properties

- Rheology
  - Mooney viscosity
  - Curing data
- Mechanical properties – standard data (hardness, tensile strength, elongation at break, compression set)
  - After press-cure and post-cured
  - After hot air aging (press-cured and post-cured)

Mooney Viscosity

DIN 53 523 Part 3, ML 1+4, 70 °C



Mooney Scorch Time

DIN 53 523 Part 4, ML +5, 70 °C

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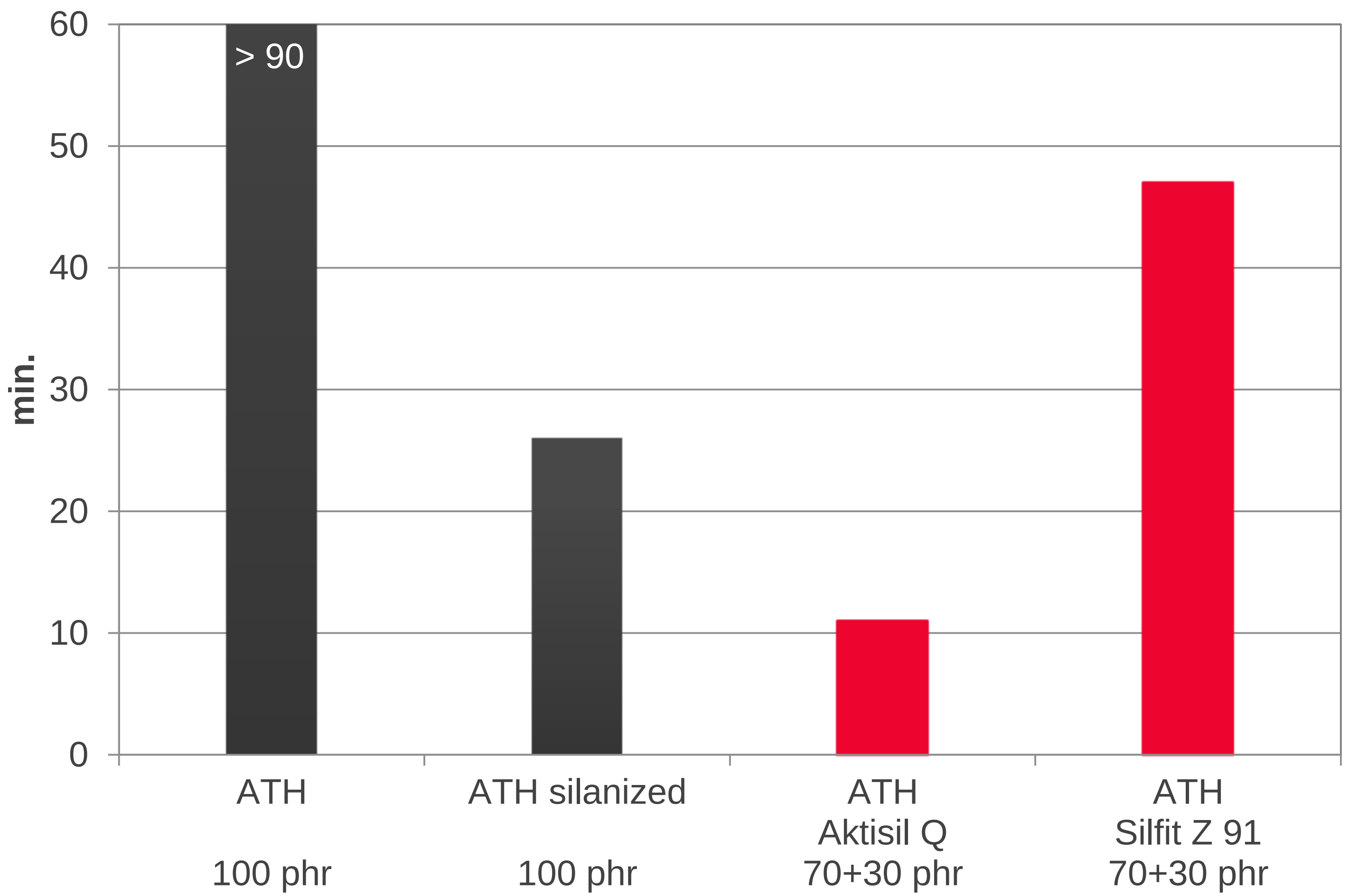
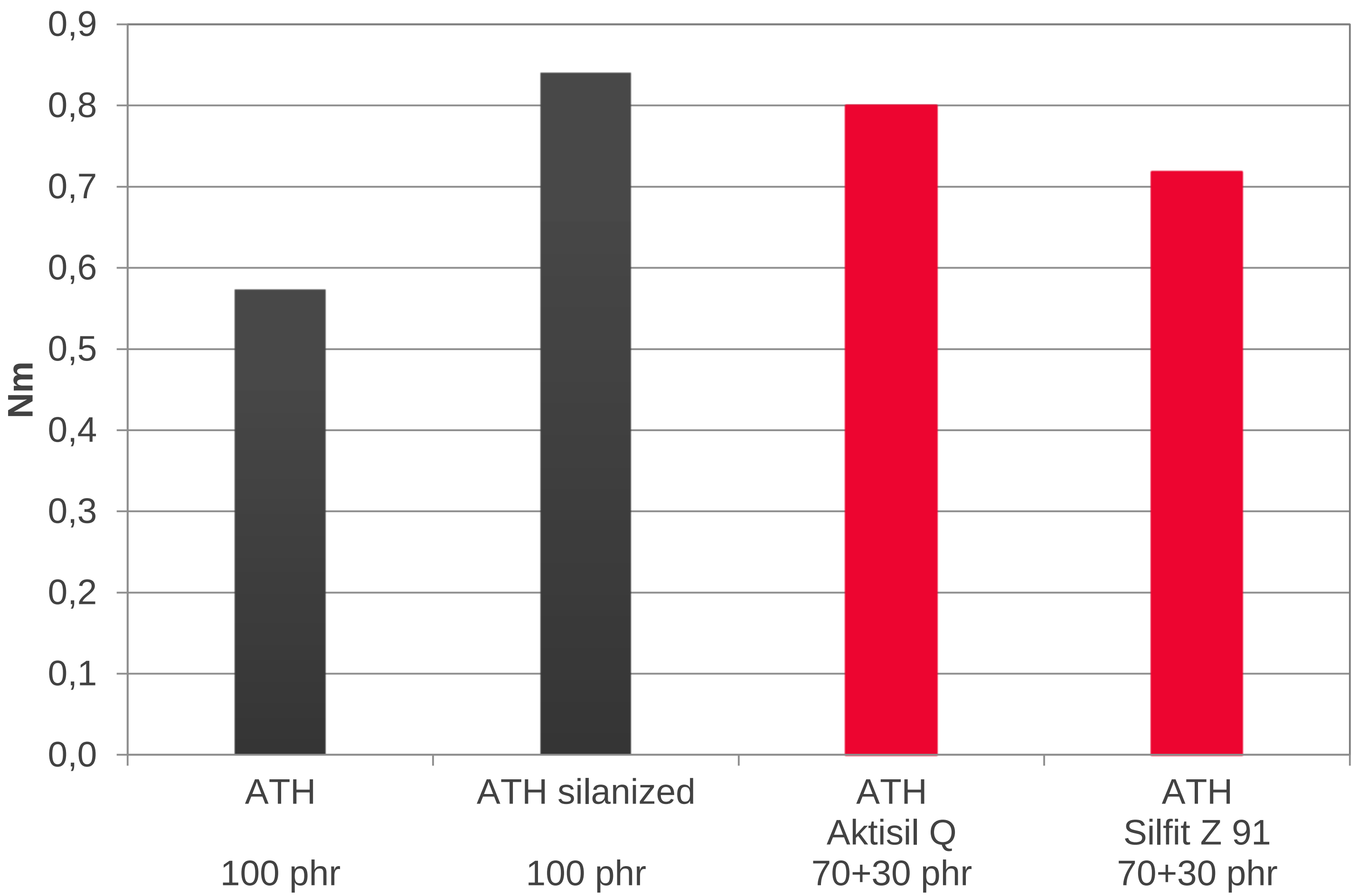


Torque Maximum

DIN 53 529-A3, 115 °C, 0.2° deflection – Göttfert Elastograph



Cure Rate Maximum

DIN 53 529-A3, 115 °C, 0.2° deflection – Göttfert Elastograph

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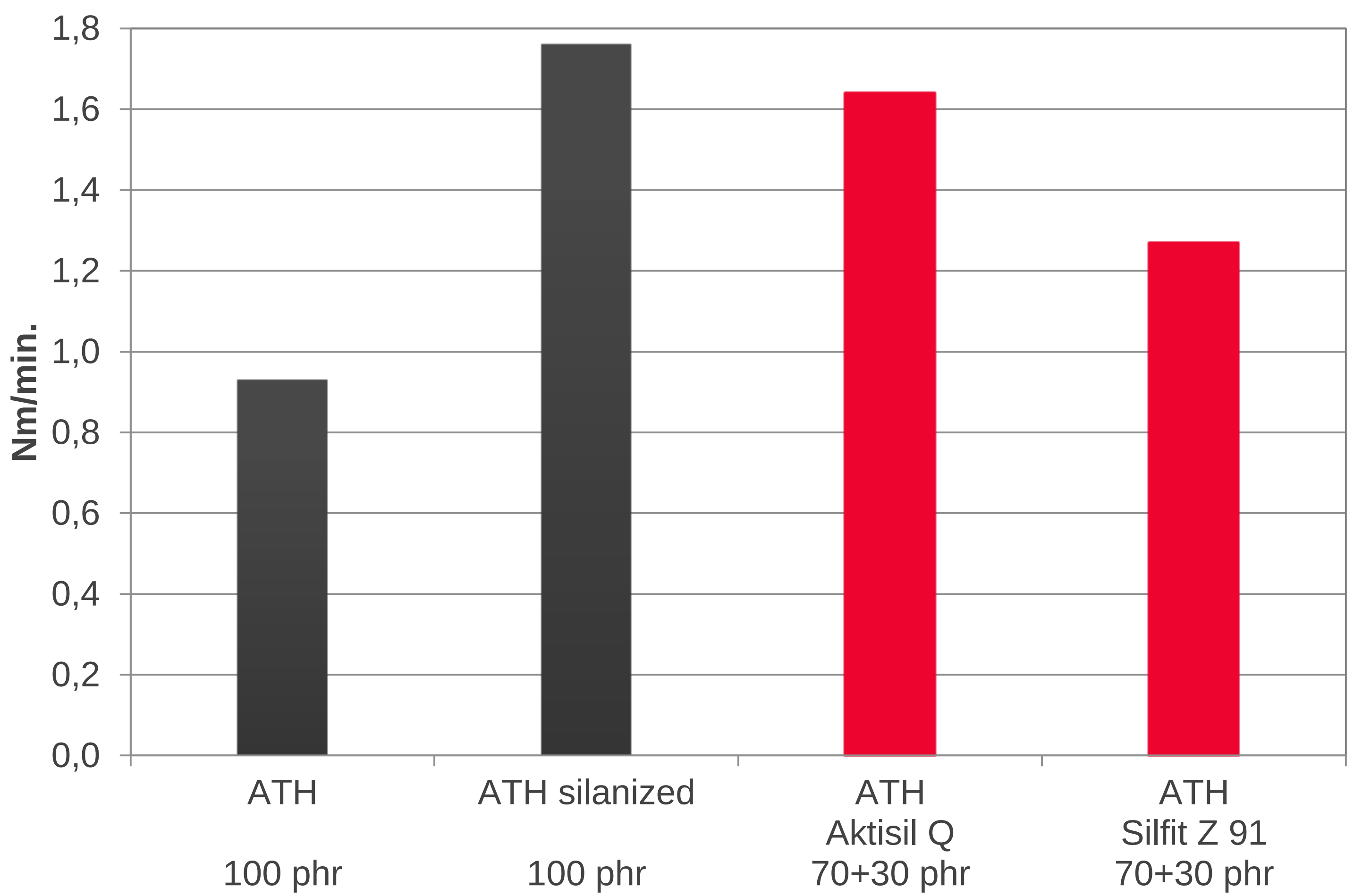

Conversion Time t_{90}

DIN 53 529-A3, 115 °C, 0.2° deflection– Göttfert Elastograph

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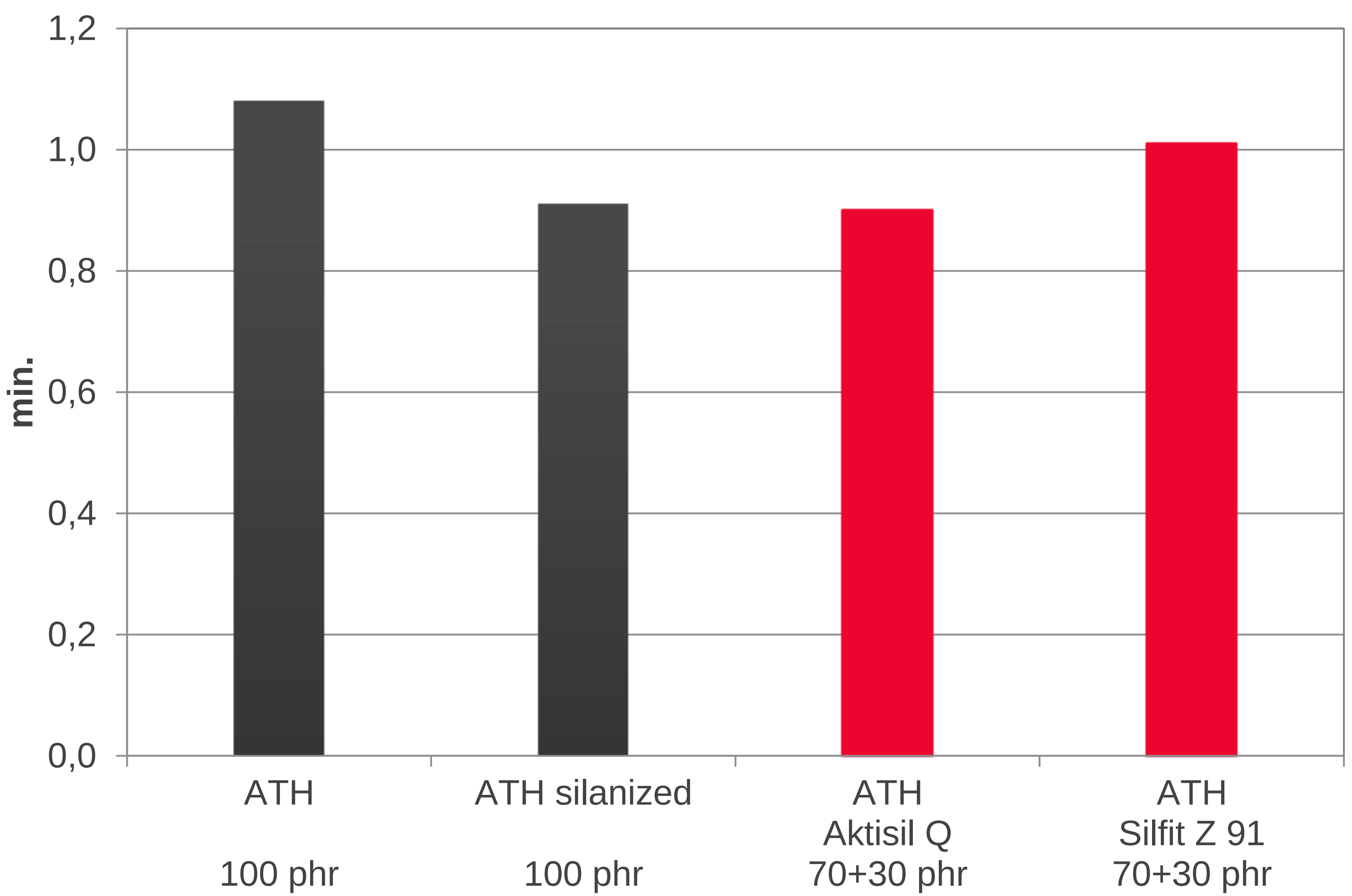

Loss Angle tan δ after 10 minutes cure

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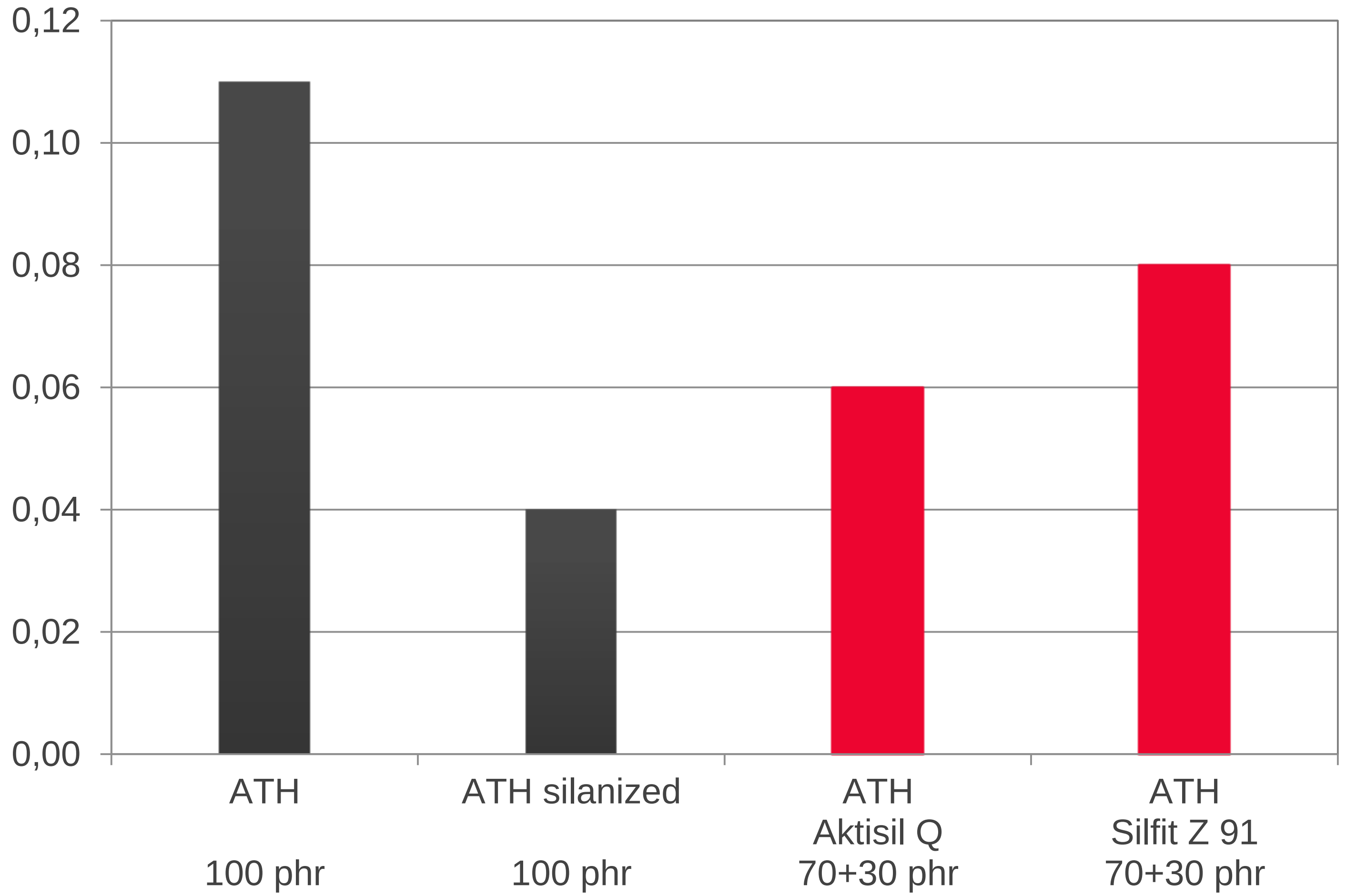
DIN 53 529-A3, 115 °C, 0.2° deflection – Göttfert Elastograph



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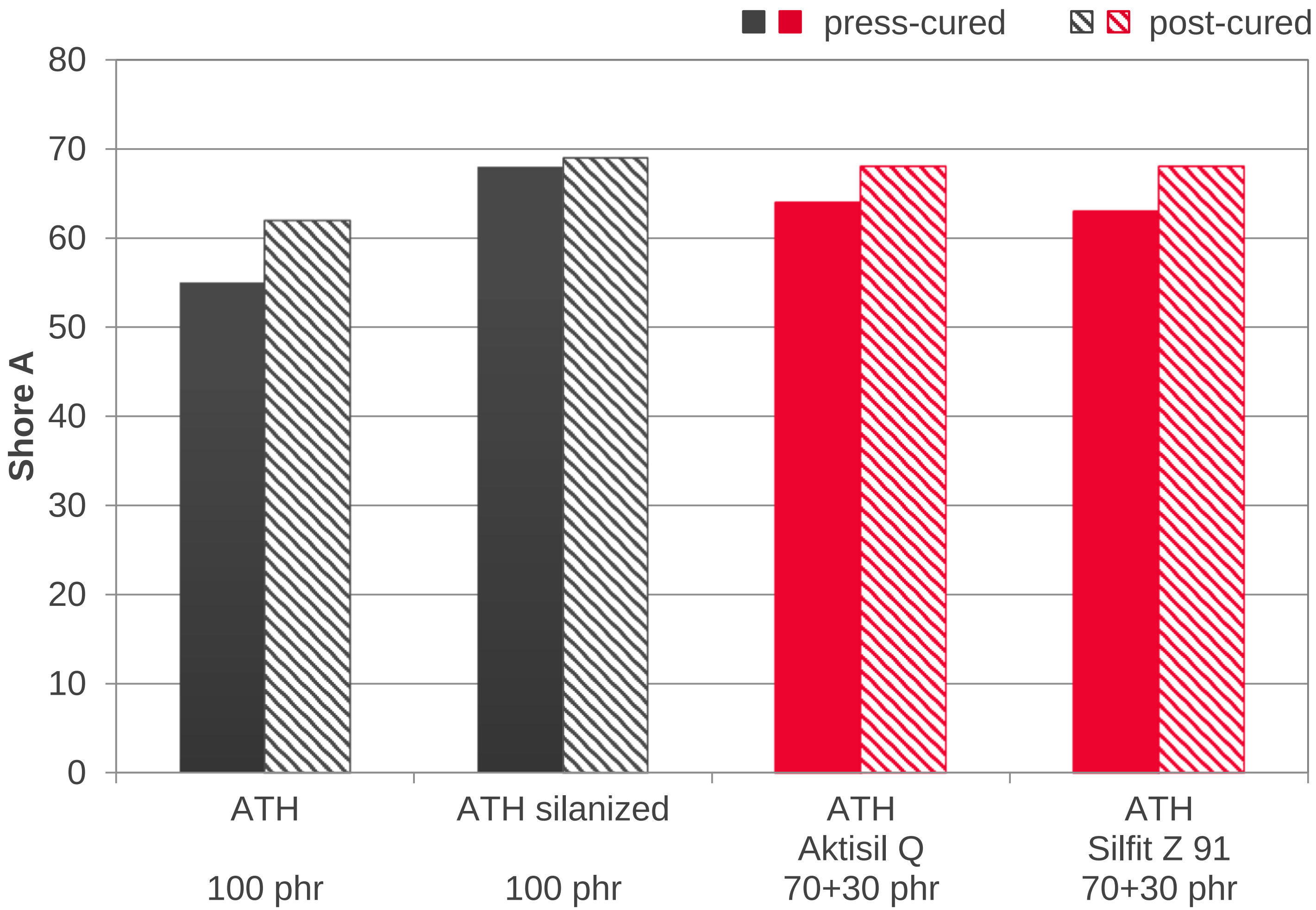
Hardness

DIN 53 505-A, piled-up S2 dumbbells



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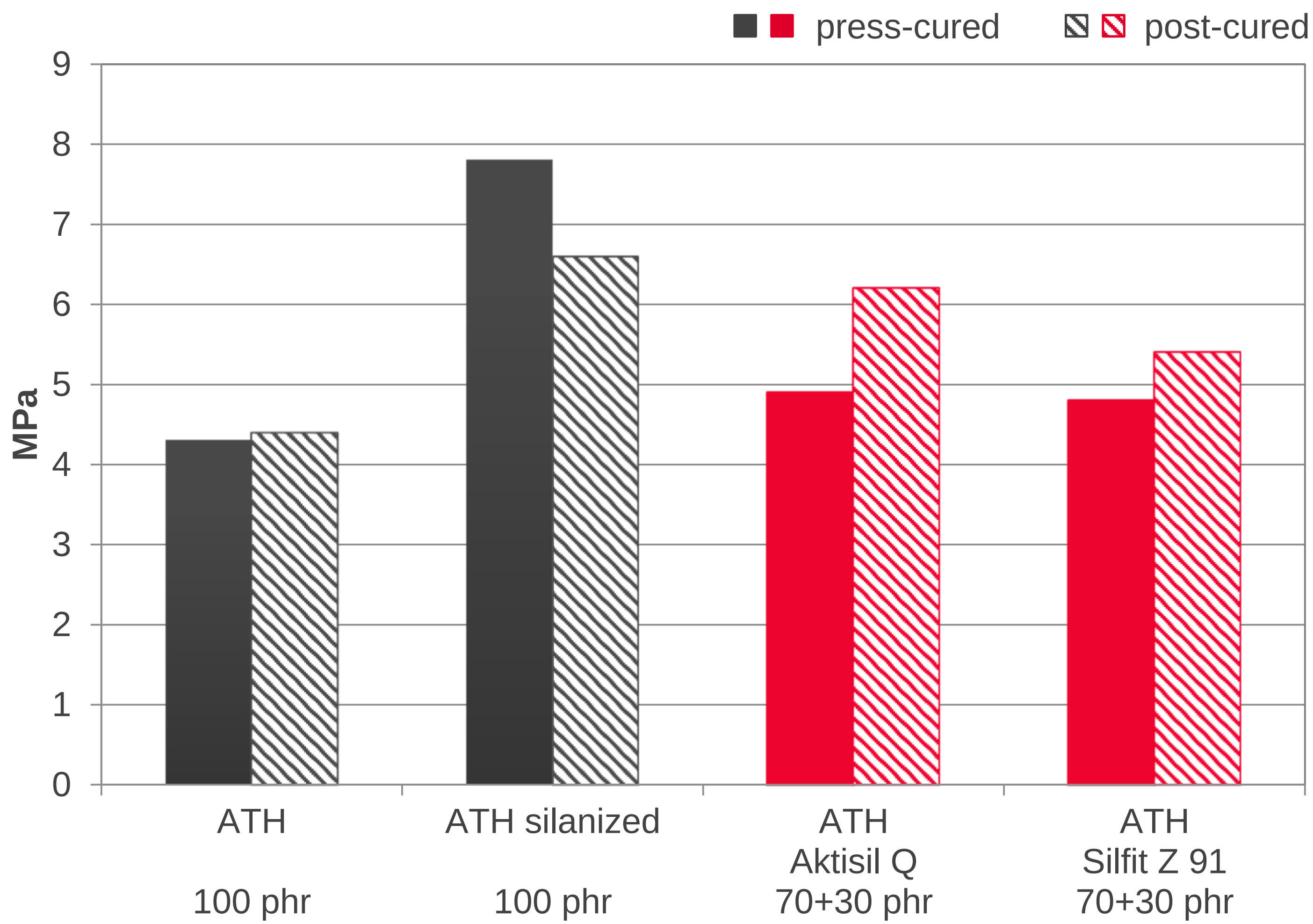
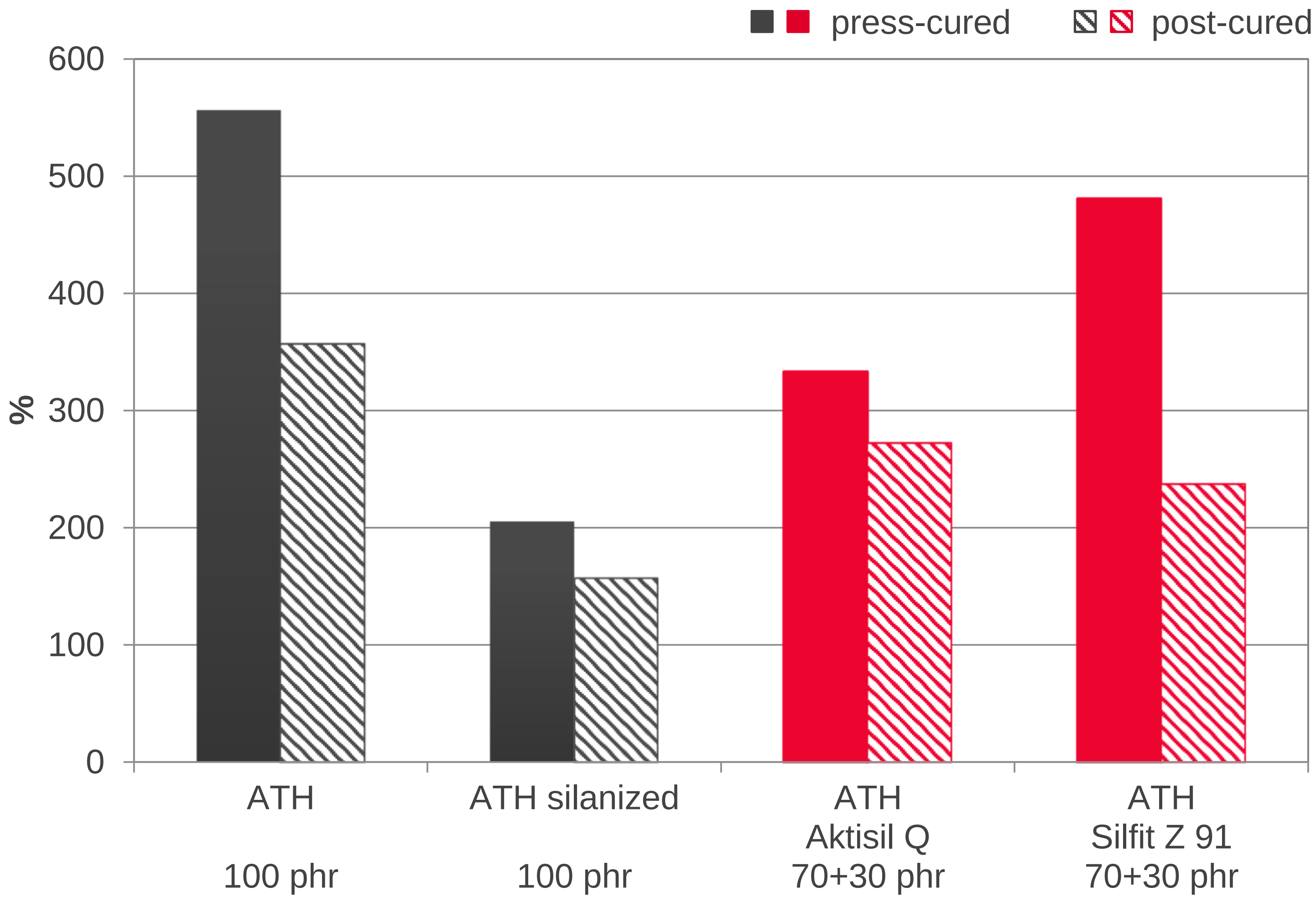
Tensile Strength

DIN 53 504, S2



Elongation at Break

DIN 53 504, S2



Modulus 100 %

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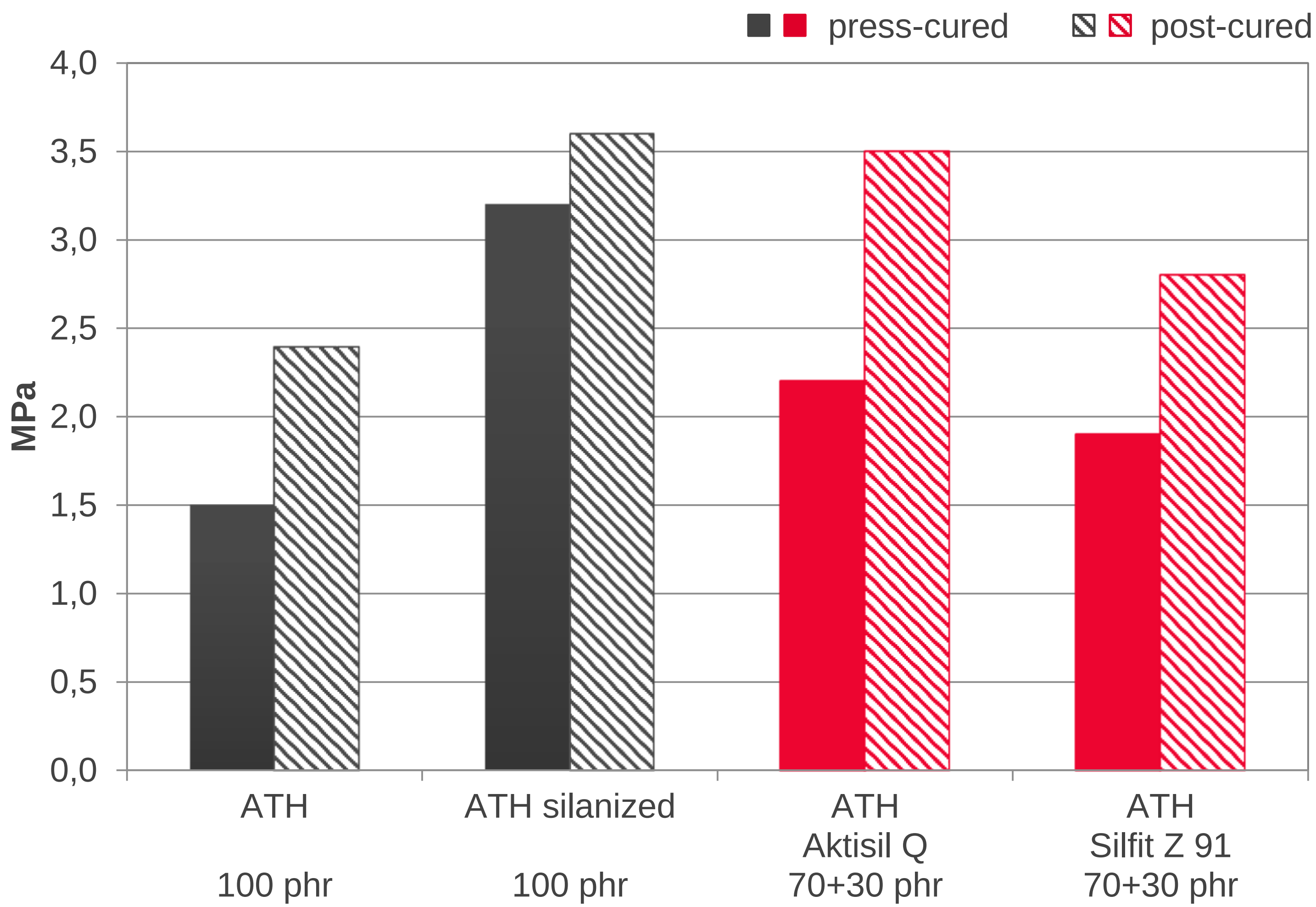
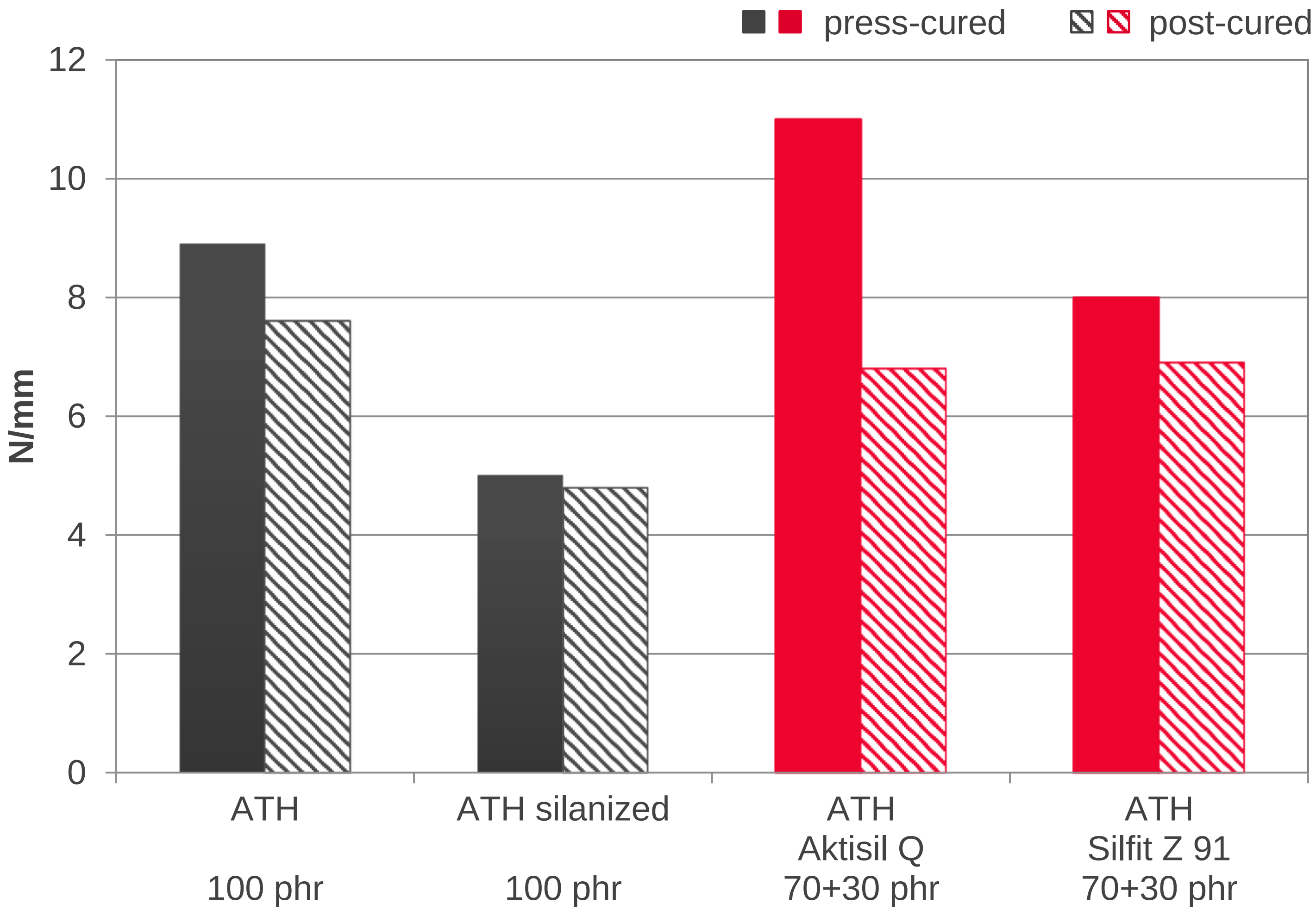
DIN 53 504, S2



Tear Resistance

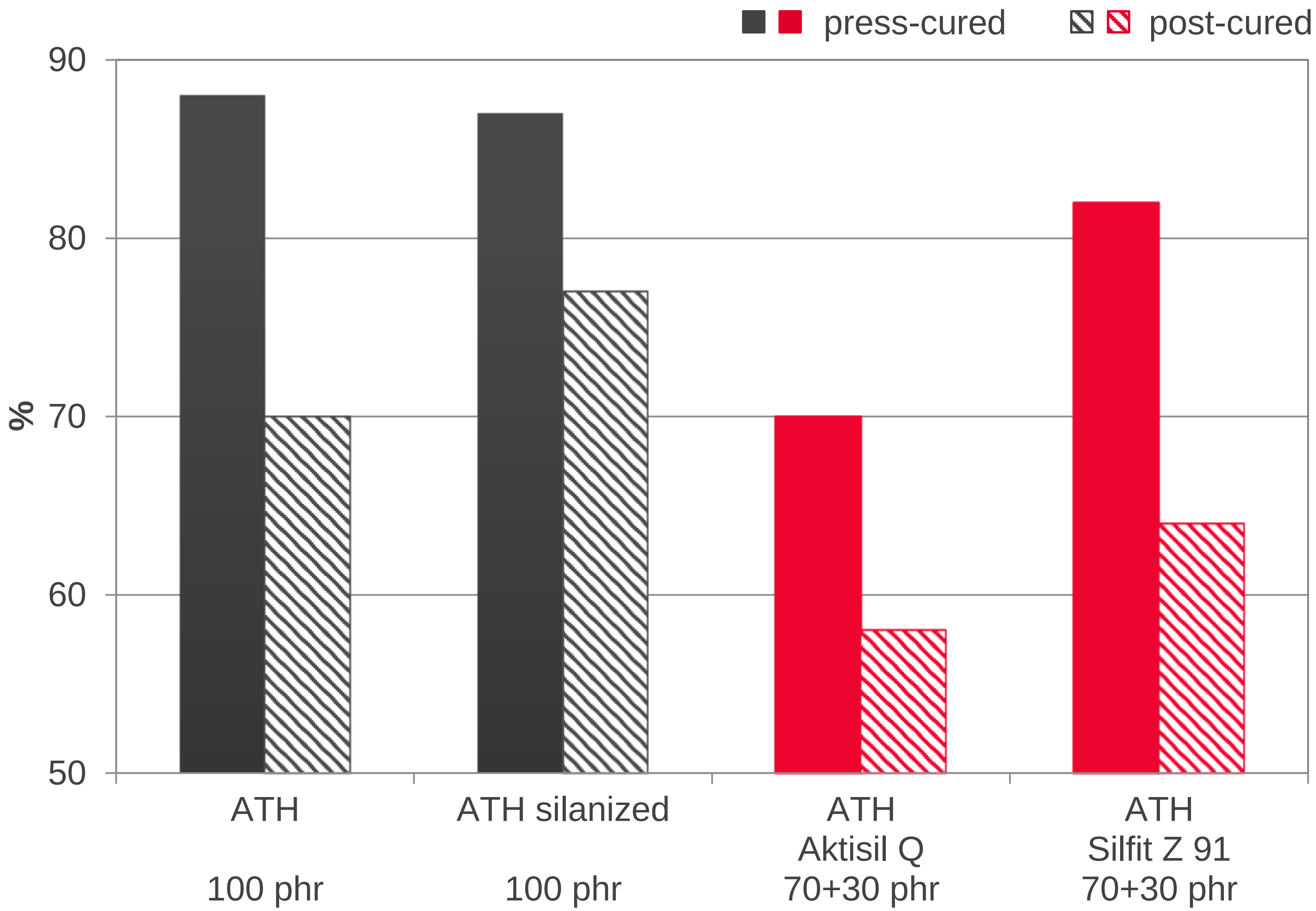
DIN ISO 34-1, Graves



Compression Set

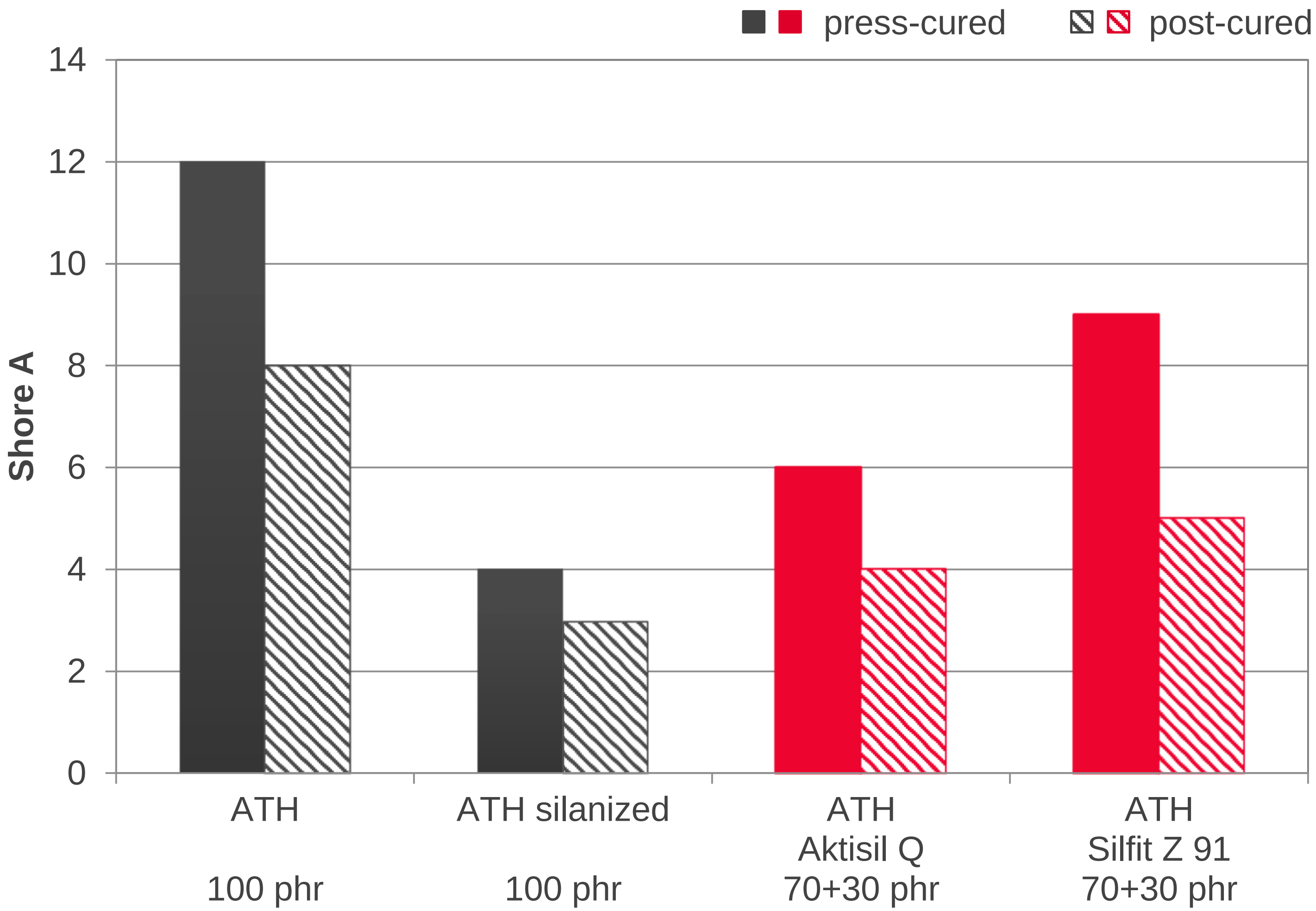
24 h / 175 °C

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



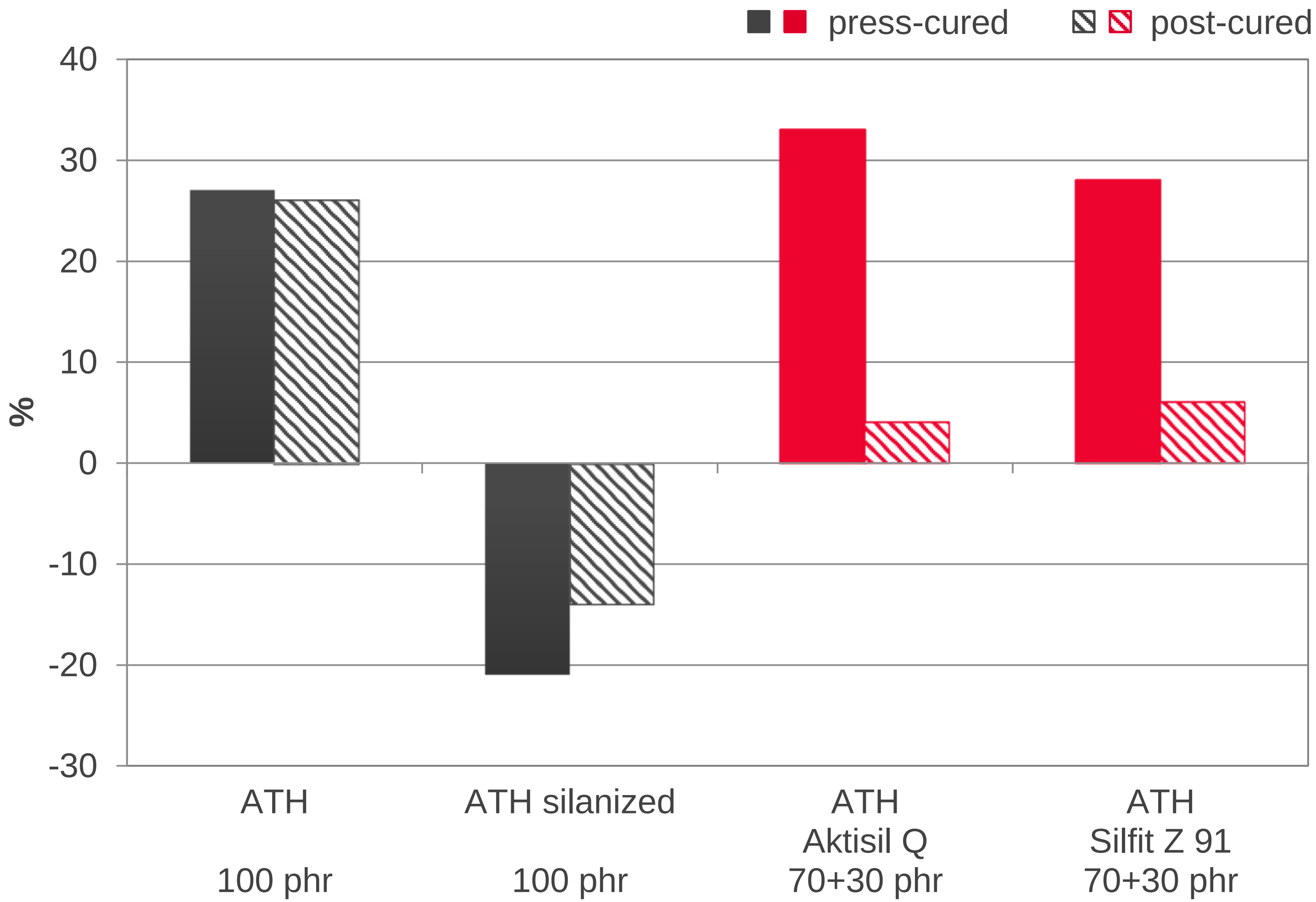
Hot Air Aging
Change of Hardness

DIN 53 508, 168 h / 200 °C



Hot Air Aging
Change of Tensile Strength

DIN 53 508, 168 h / 200 °C



Hot Air Aging
Change of Elongation at Break

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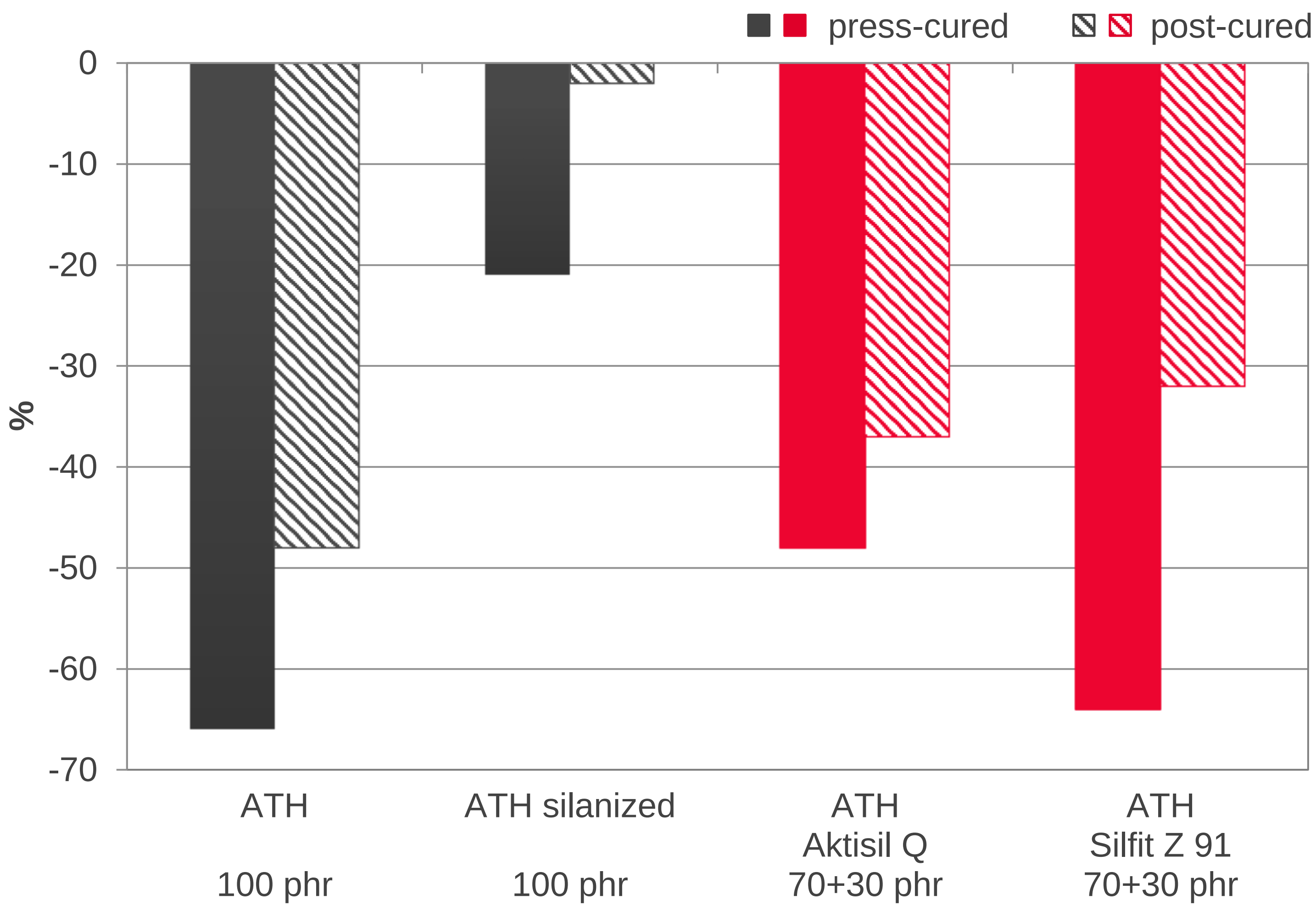
DIN 53 508, 168 h / 200 °C



Hot Air Aging
Change of Modulus 100 %

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DIN 53 508, 168 h / 200 °C

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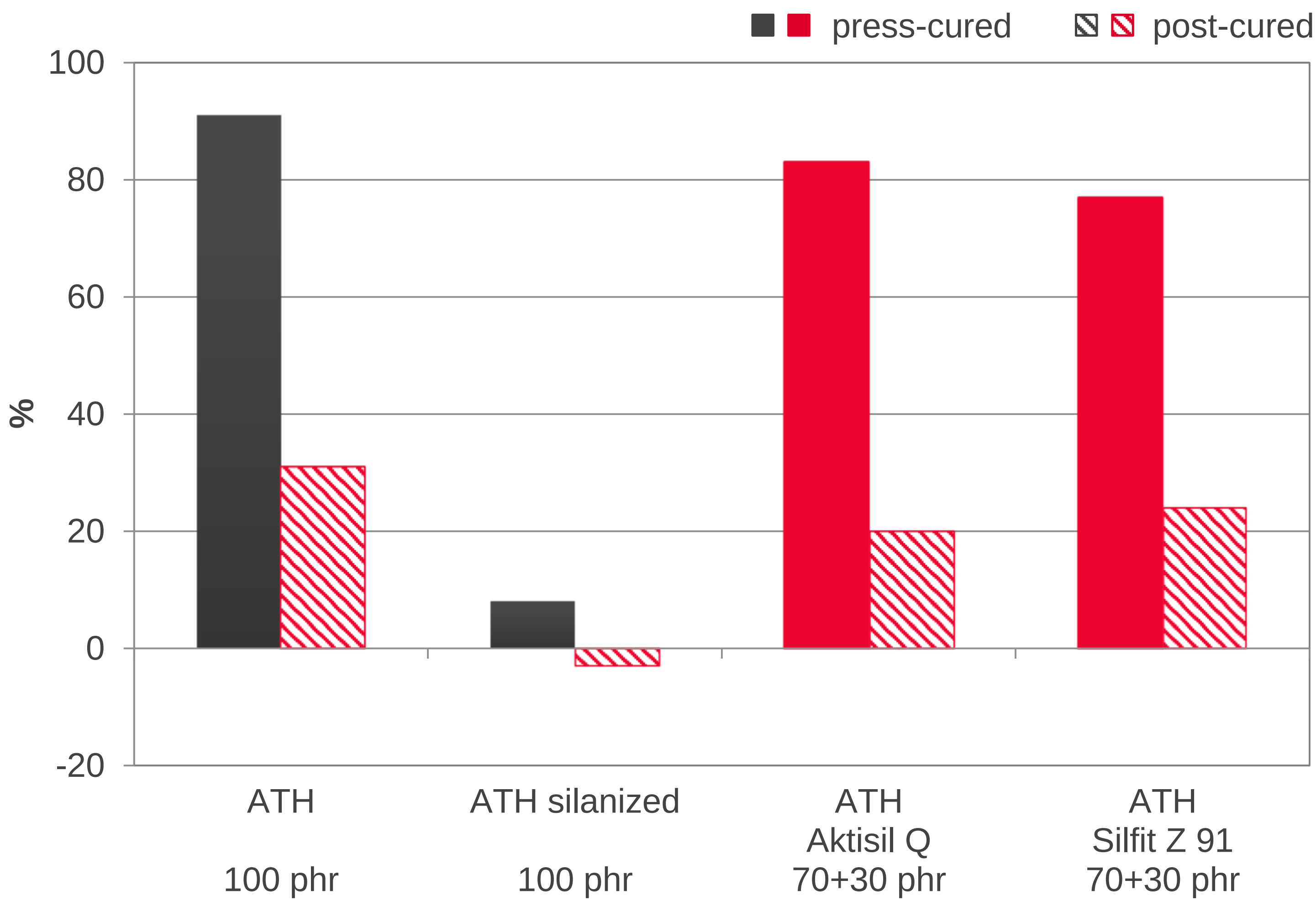

Conclusion

The combination of Neuburg Siliceous Earth grades with non-surface-treated aluminium hydroxide (ATH) vs. ATH (straight) and vinyl-silane-treated ATH (straight):

- ✓ No negative effects on flame retardancy (comparable: LOI, heat release rate, smoke density)
- ✓ reduction of compound costs

Blend ATH with Silfit Z 91:

- ✓ more advantageous rheology for extrusion (esp. vs. the non-surface-treated ATH)
- ✓ mechanical properties between those of the non-surface-treated and silanized ATH

  + additional reduction of compression set
- ✓ improved aging behavior in hot air compared to non-surface-treated ATH

Conclusion

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Blend with Aktisil Q:

- ✓ more advantageous rheology for extrusion (esp. vs. the non-surface-treated ATH)
- ✓ unites the good mechanical properties of the non-surface-treated and the silanized ATH:

  e.g. high tensile strength (cf. ATH silanized) along with good elongation at break (cf. ATH) and high tear resistance (cf. ATH)

  + markedly reduced compression set
- ✓ improved aging behavior in hot air compared to non-surface-treated ATH
- ✓ **Combination of Neuburg Siliceous Earth with ATH is possible without any losses regarding flame retardancy, along with improved rheological and mechanical properties.**

We thank Nabaltec AG, Schwandorf for performing the tests concering flame retardancy as well as for the always friendly and qualified support.

We supply material for good ideas!

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