Collapse resistant extrusions and further benefits with Neuburg Siliceous Earth in peroxide cured high consistency silicone rubber

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- **Introduction**
- **Experimental**
  - Formulation
  - Fillers and their characteristic properties
  - Testing
- **Results**
  - Preliminary Tests at fixed filler loading
  - Comparison at different filler loading
- **Summary**

Where and Why is Silicone Rubber used?

Heat cured (HTV) silicone rubbers find application in

- Mechanical engineering
- Automotive sector
- Wire and cable or electrical industries
- Household appliances

as

- Moldings: gaskets, seals and O-rings
- Extrusions: profiles, hoses and cable insulations

due to

- Easy processing
- Outstanding mechanical properties including excellent compression set
- Very good chemical and thermal resistance

Why using Fillers?

- For cost reasons inactive fillers such as quartz flour or diatomaceous earth are added to the compounds
- Optimizing processing properties
- Mechanical properties in most cases remain unchanged, in some cases they are even improved

Objective

Evaluation of the property profile of

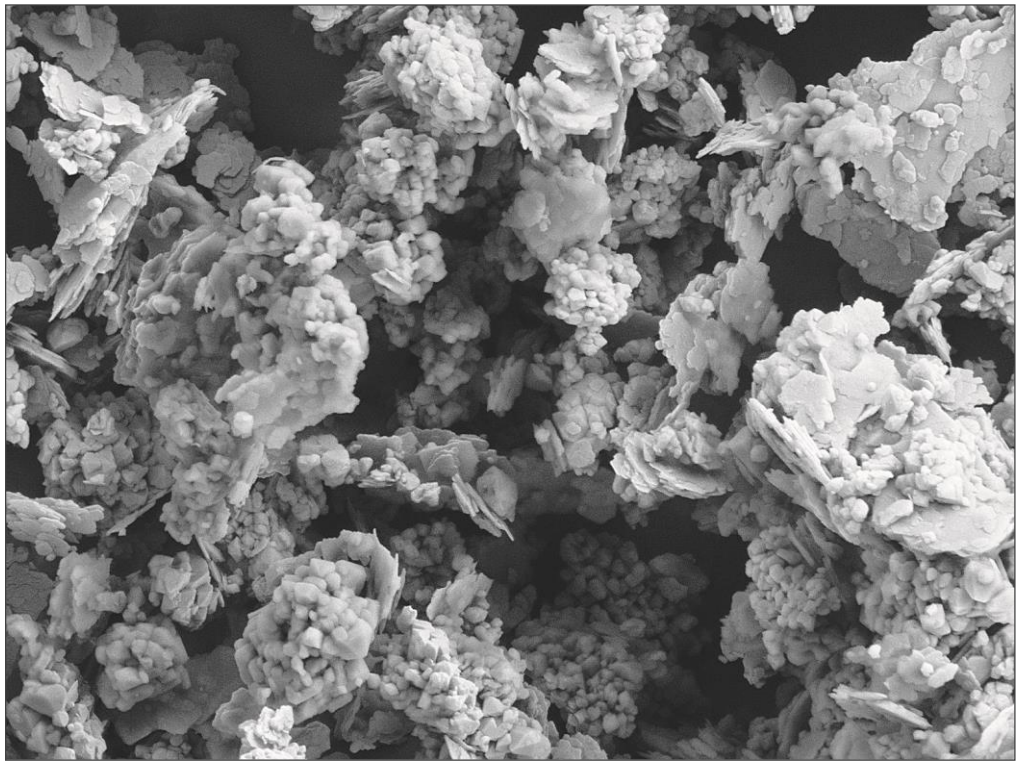
- Neuburg Siliceous Earth, in this case Aktisil Q,

  in comparison with the typical fillers

- Diatomaceous earth

- Quartz flour

What is Neuburg Siliceous Earth?



A natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods.

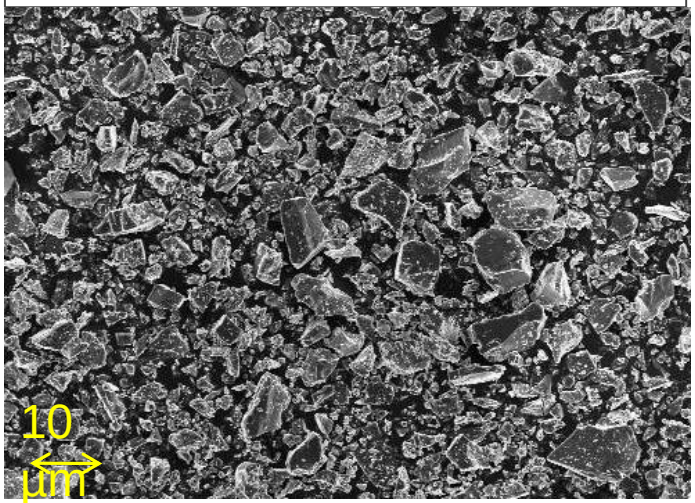
The silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.

Compound Formulations

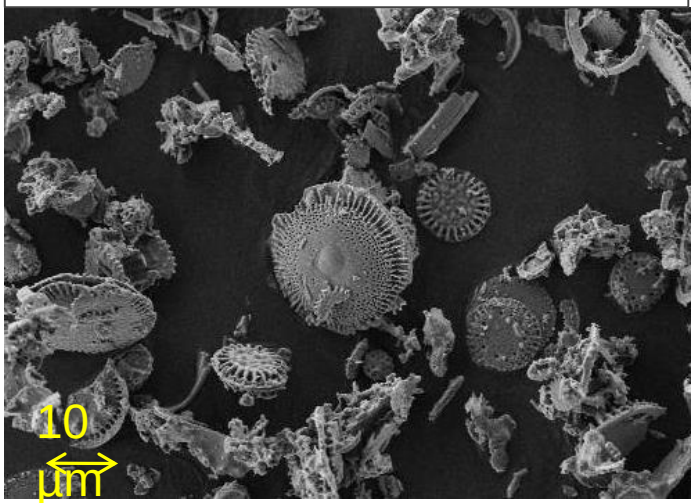
		phr		
Polymer	Base Compound Elastosil R401/40	100		
Filler	as indicated	0 – 100		
Curing Agent	Bis-(2,4-dichlorobenzoyl)-peroxide (50 %) Elastosil AUX Curing Agent E	1,5	-	-
	2,5-Bis-(tert. Butylperoxy)-2,5-dimethylhexane (45 %) Elastosil AUX Curing Agent C6	-	1,2	-
	Dicumyl Peroxide (DCP) (40 %) Perkadox BC-40X-ps	-	-	0,99

Filler SEM Images

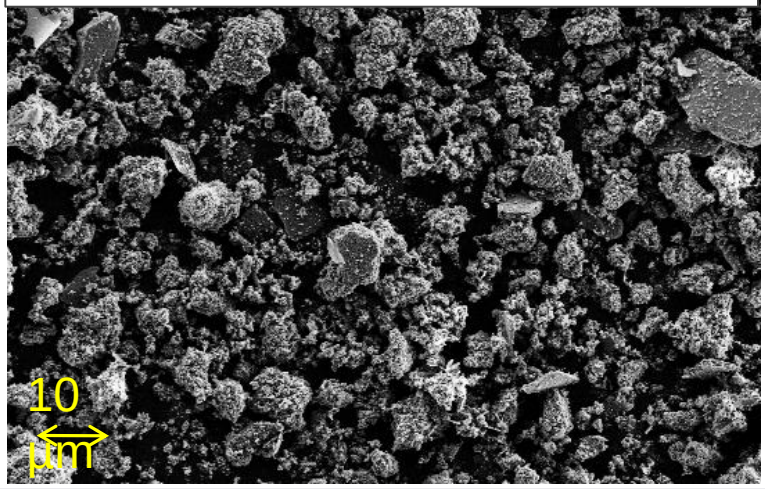
Quartz flour



Diatomaceous Earth



Neuburg Siliceous Earth, Aktisil Q



Filler Characteristics

	Functiona-lization	Particle size [µm]		Oil absorption	Spec. surface area BET	Abrasivity index (*)
		d_{50}	d_{97}	[g/100g]	[m²/g]	[-]
Quartz flour vst	Vinyl silane	3.4	13	32	3.2	100
Diatomaeous Earth, flux calcined	none	11.9	33	116	1.5	100
Aktisil Q	Methacryl	4.2	17	42	6.5	50

Standards

Mooney viscosity	DIN 53 523, T4
Mooney scorch	DIN 53 523, T4
Curing profiles	DIN 53 529, T3
Tensile test	DIN 53 504, S2
Hardness	DIN 53 505
Compression set	DIN ISO 815, B

Testing Conditions

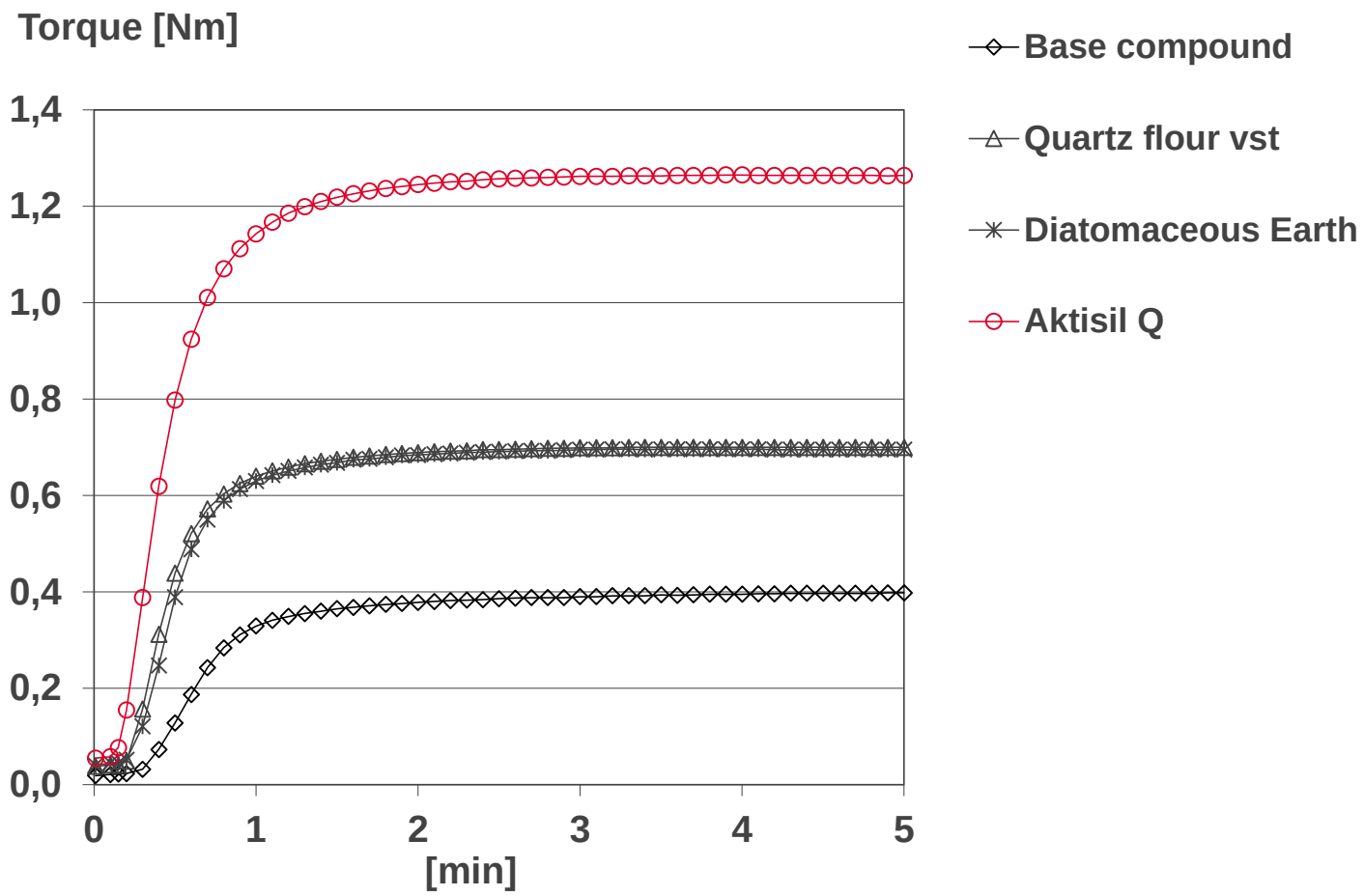
Cure time:	5 min
Cure temperature:	
– Curing Agent E:	115°C
– Curing Agent C 6:	165°C
– Dicumyl Peroxide:	180°C
Post cure:	4 h / 200°C
Heat ageing:	168 h / 200°C
Aging in reference oil IRM 903:	72 h / 150°C

Preliminary Tests

	Loading
Reference fillers	
• Quartz flour, vinyl silane treated	100 phr
• Diatomaceous Earth, flux calcined	50 phr
Neuburg Siliceous Earth	
• Aktisil Q	100 phr

Curing Profiles

Curing Agent E (115°C)



Hardness

INTRODUCTION

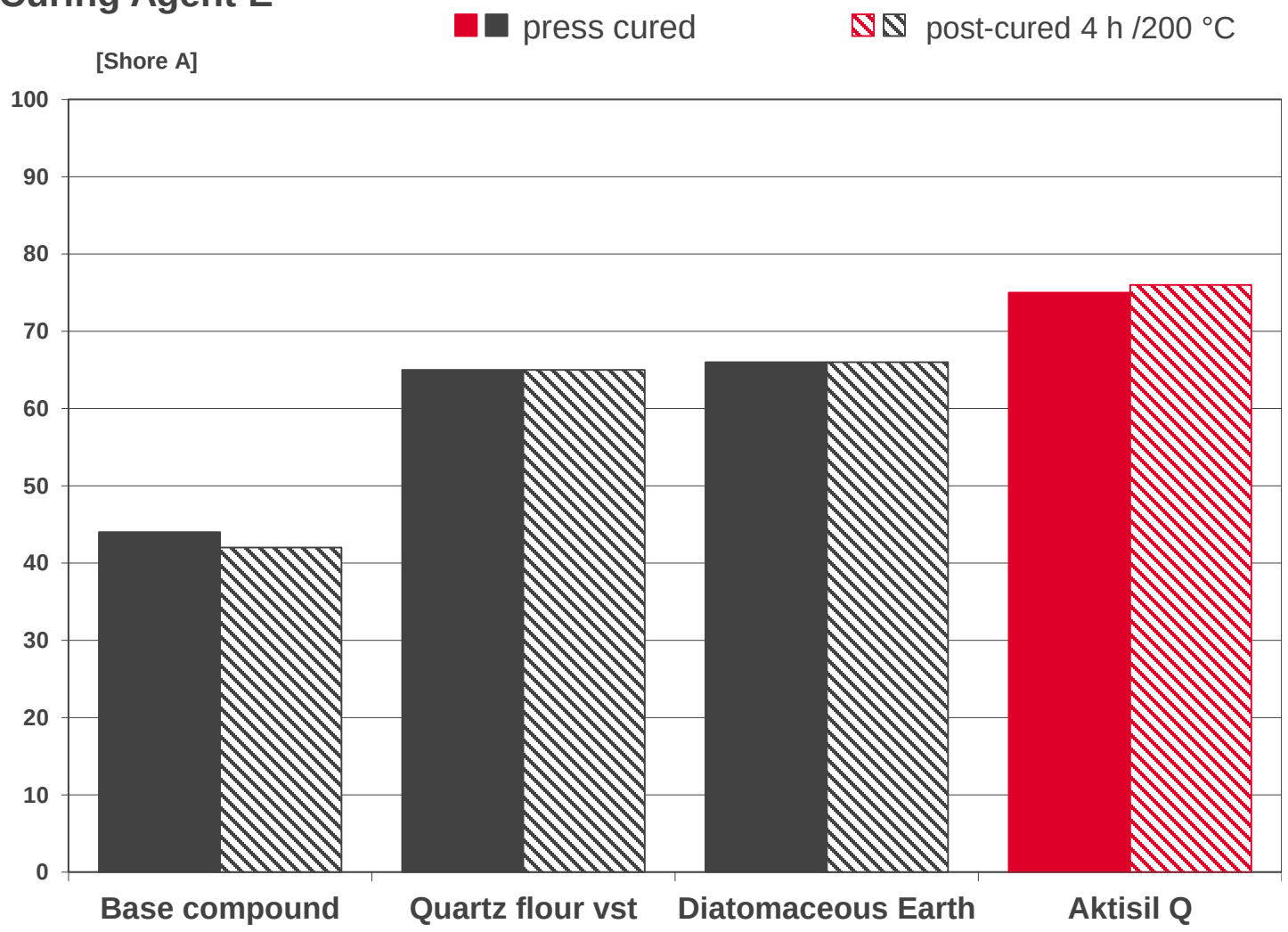
EXPERIMENTAL

PRELIMINARY TESTS

MAIN TESTS

SUMMARY

Curing Agent E



Compression Set
24 h / 175 °C

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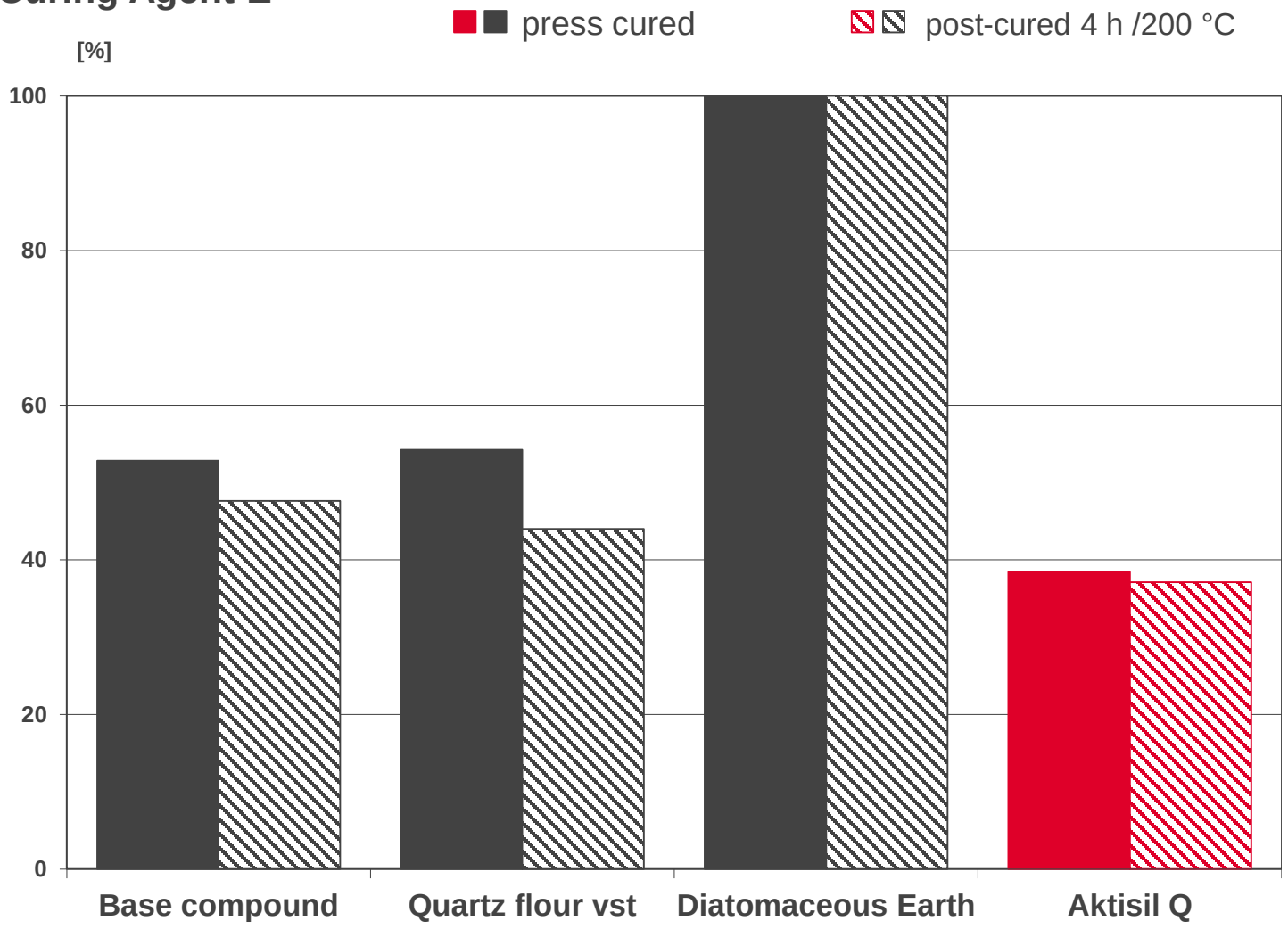
Curing Agent E



Extrusion

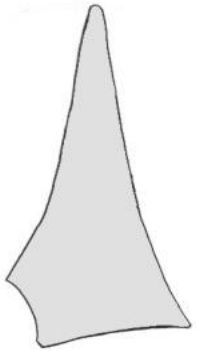
Technical characteristics of the extruder:

- Schwabenthan-Extruder Polytest 30 R,
- Screw diameter 30 mm,
- Screw length 15D

Extrusion conditions:

- 25°C in all zones

- Garvey profile



- Hose die, Diameter: 20 / 16.5 mm

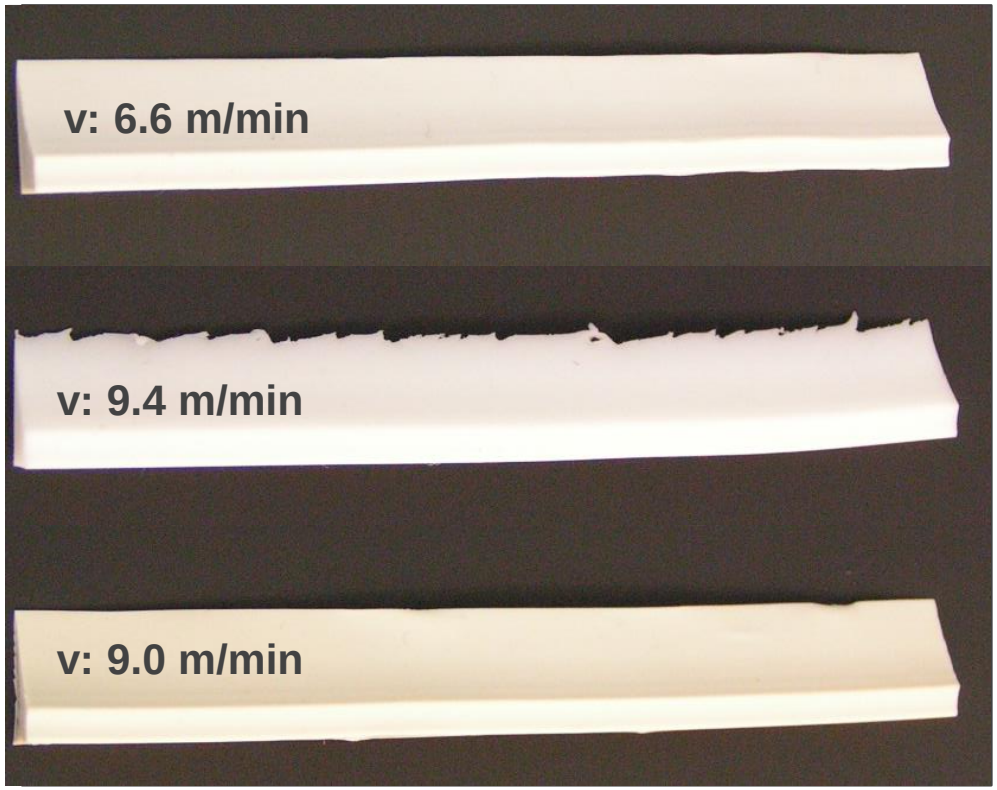
Extrusion
Garvey Profile

100 rpm

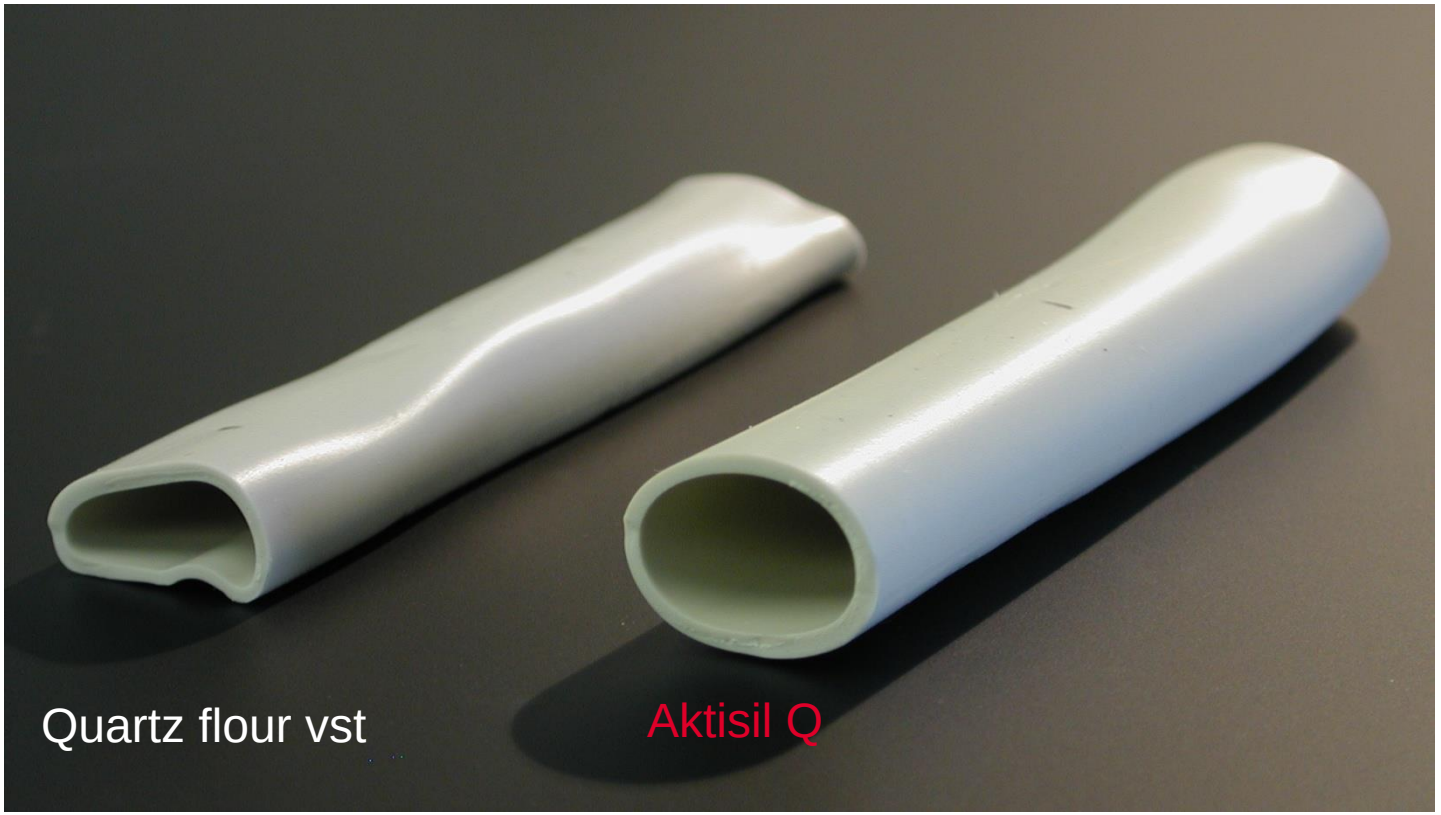
Quartz flour vst

Diatomaceous Earth

Aktisil Q



Extrusion
Collapse Resistance



Extrusion
Collapse Resistance

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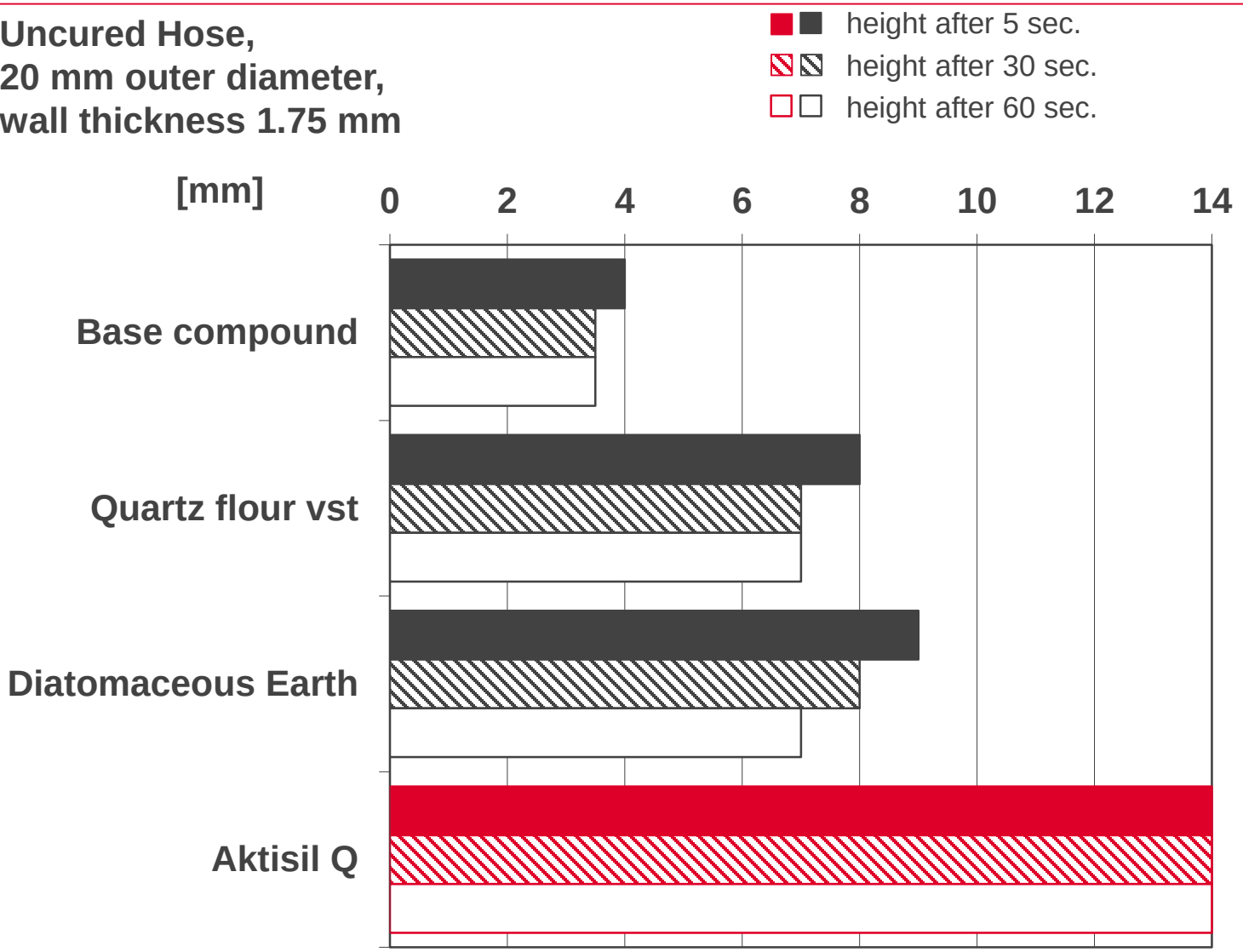
Uncured Hose,
20 mm outer diameter,
wall thickness 1.75 mm



Extrusion
Stickiness

Uncured Extruded Compound	Stickiness
Quartz flour vst	Yes
Diatomaceous Earth fc	None
Aktisil Q	None

Preliminary Tests Conclusion

Aktisil Q provides:

→ high state of cure

→ higher hardness versus quartz flour vst

→ low compression set

→ excellent extrusion properties

→ outstanding collapse resistance

→ elimination of stickiness

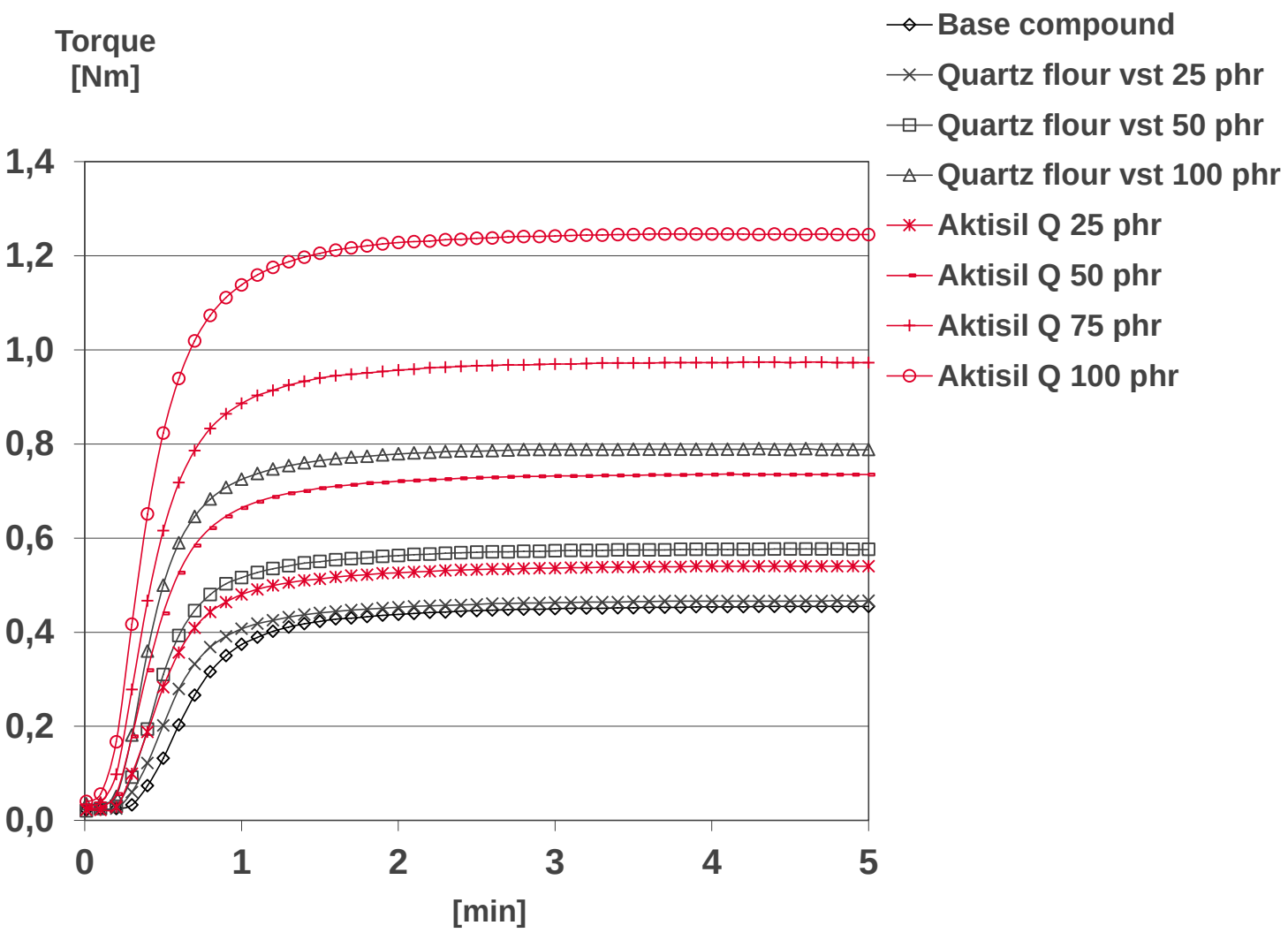
Comparison of different loadings of Aktisil Q and silane treated Quartz flour is shown in the following.

Curing Agent E

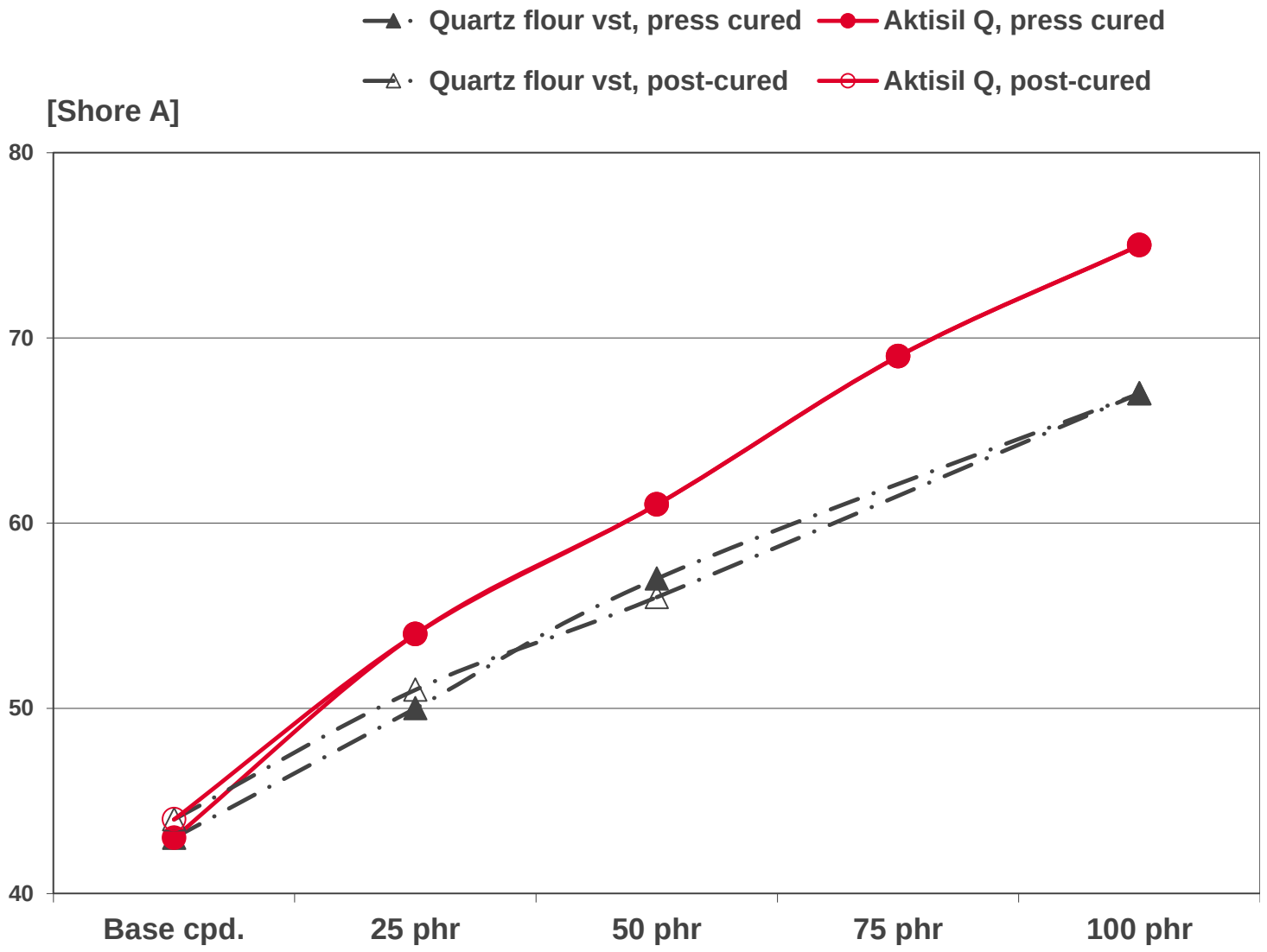
Formulation, loading in phr

		Quartz flour vst			Aktisil Q			
Elastosil R401/40	100	100	100	100	100	100	100	100
Elastosil AUX Curing Agent E	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Quartz flour vst		25	50	100				
Aktisil Q					25	50	75	100

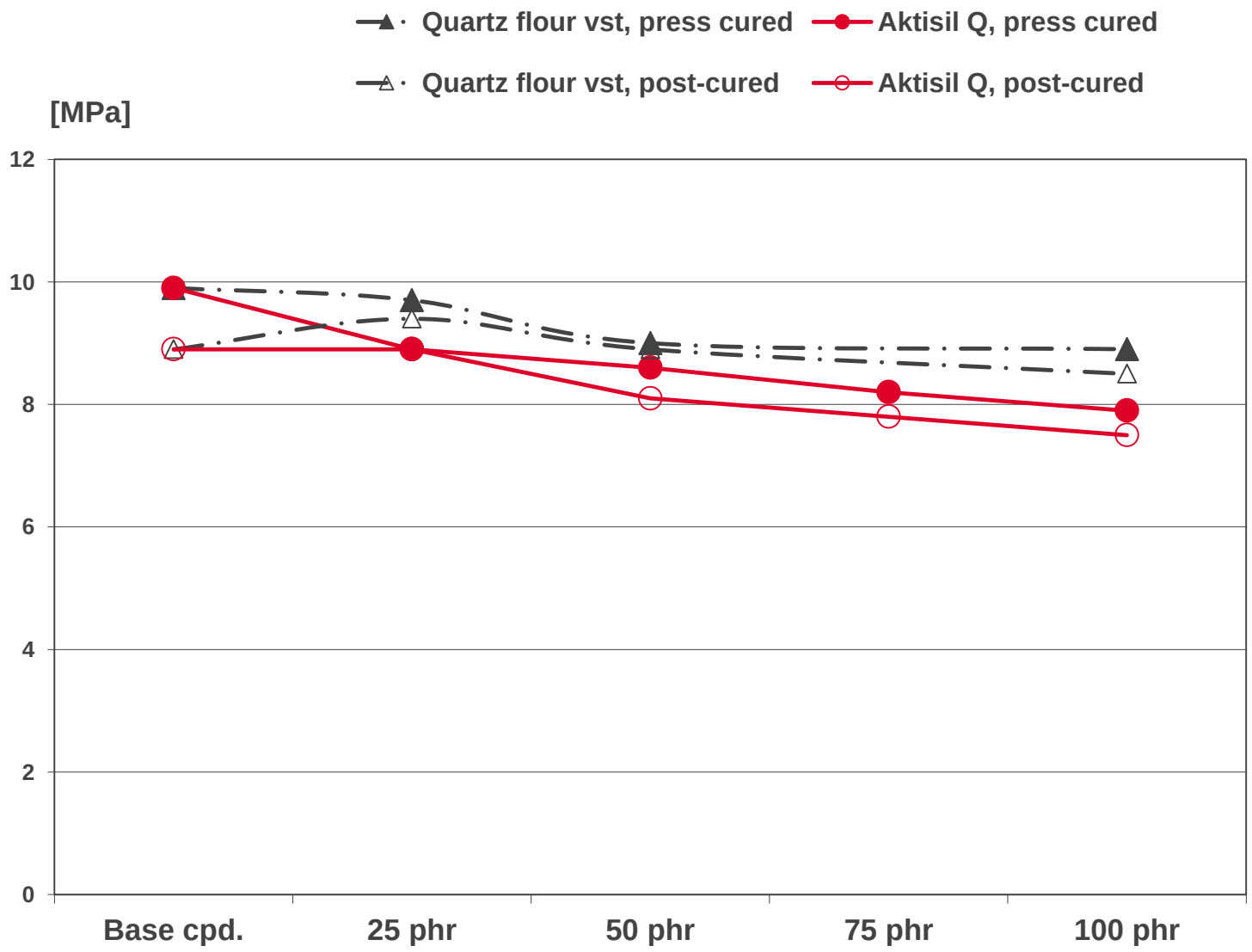
Cure Profiles (115 °C)



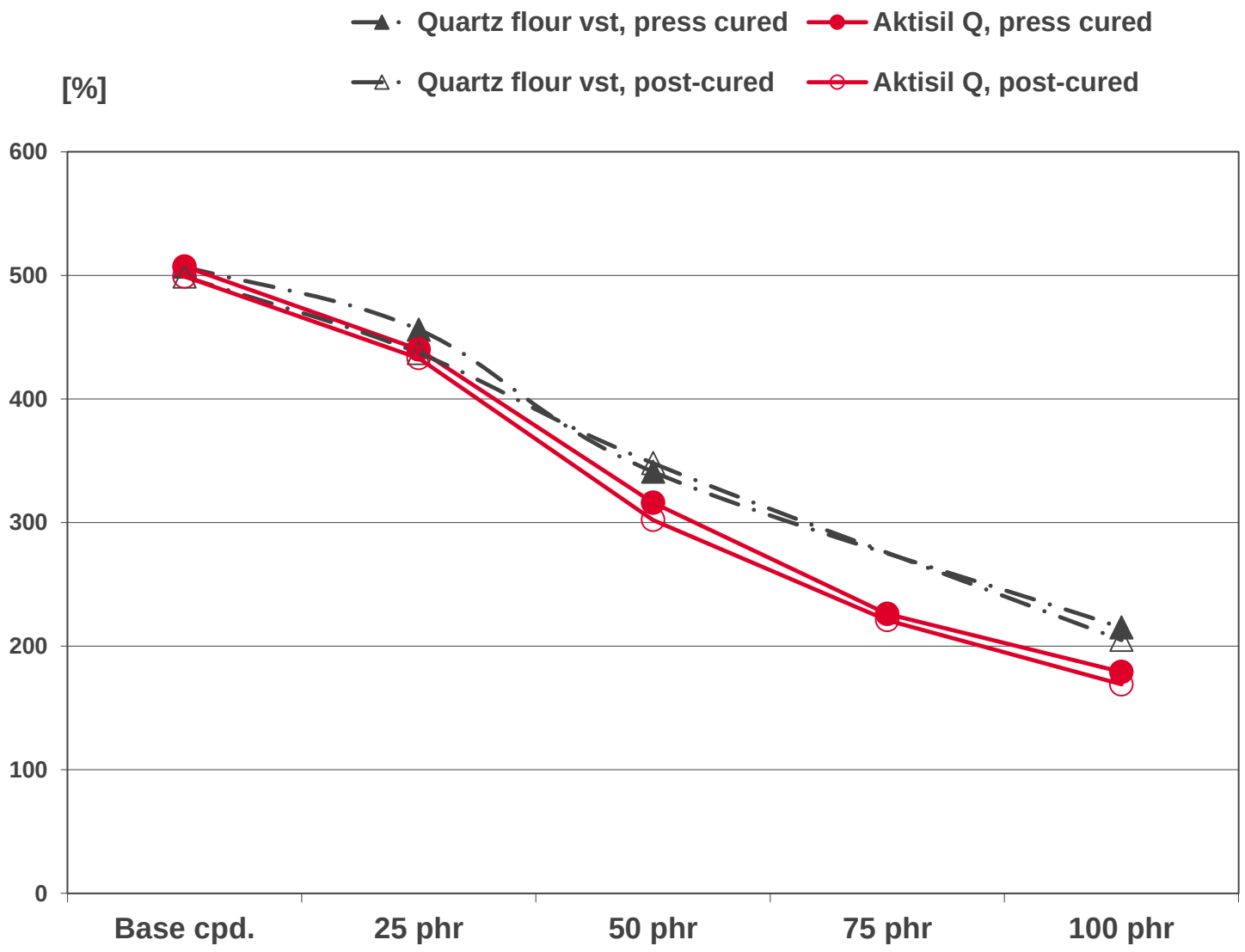
Hardness



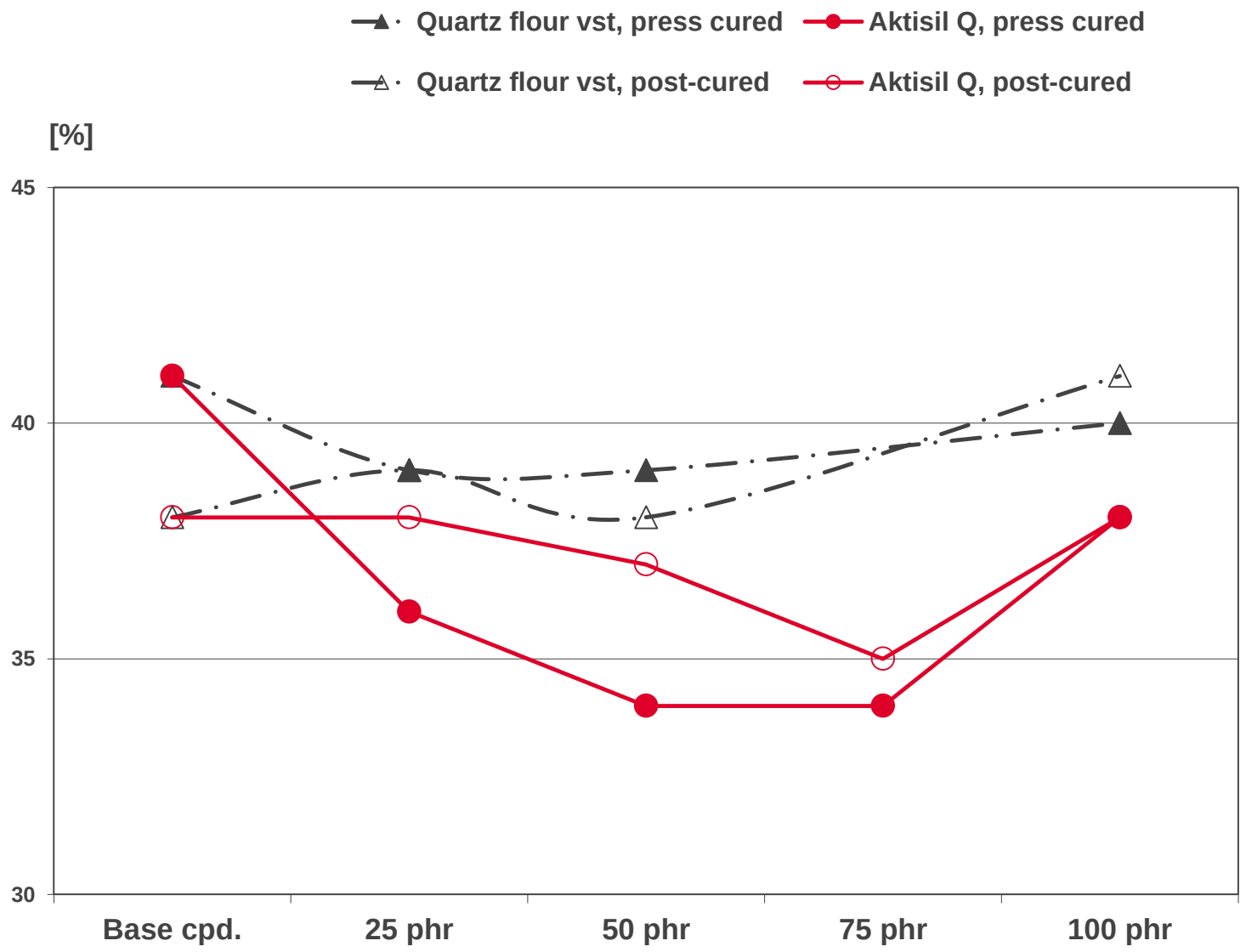
Tensile Strength



Elongation at Break



Compression Set
24 h / 175°C



Oil IRM 903, 72 h / 150°C
Increase of Volume

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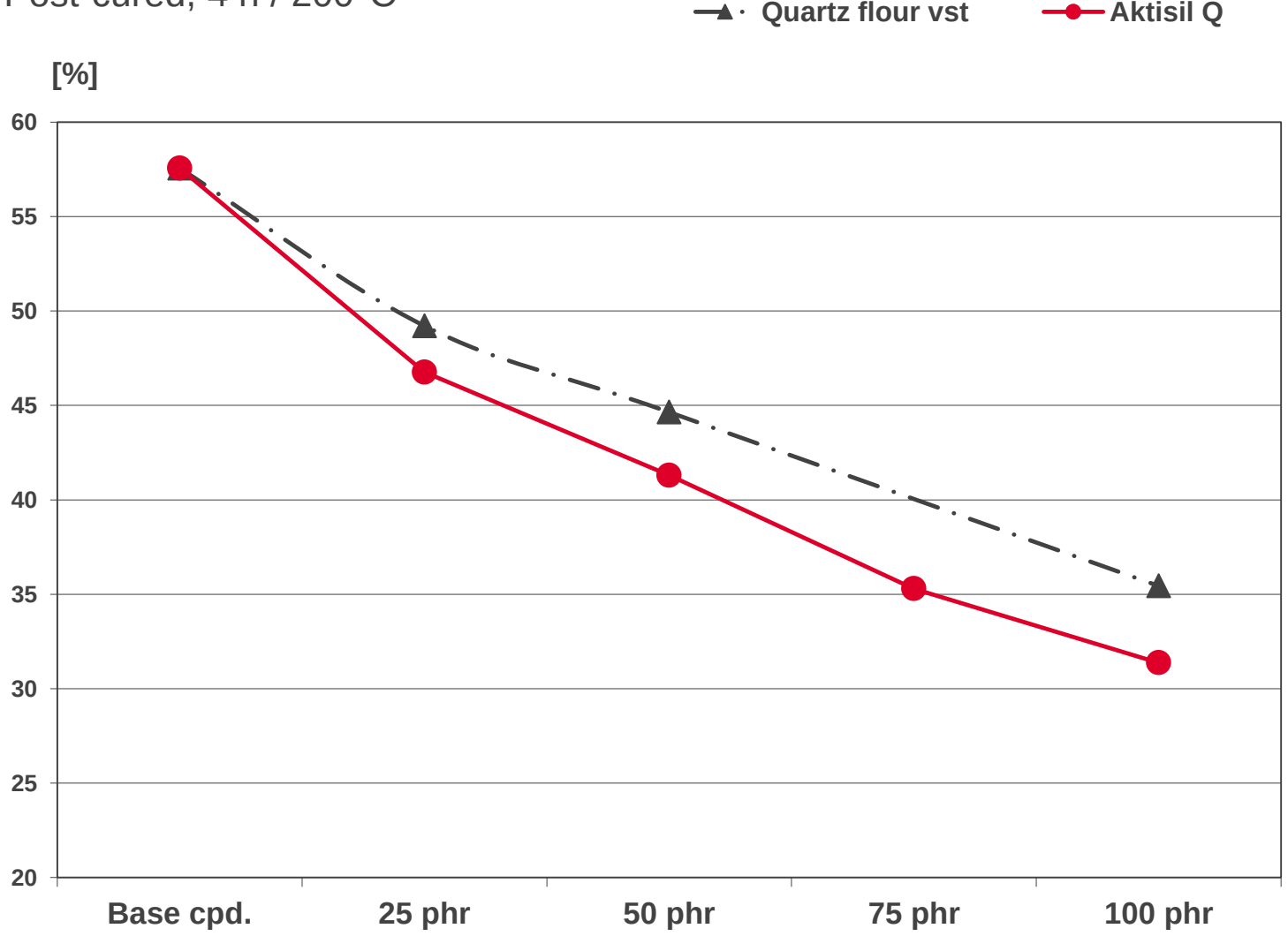
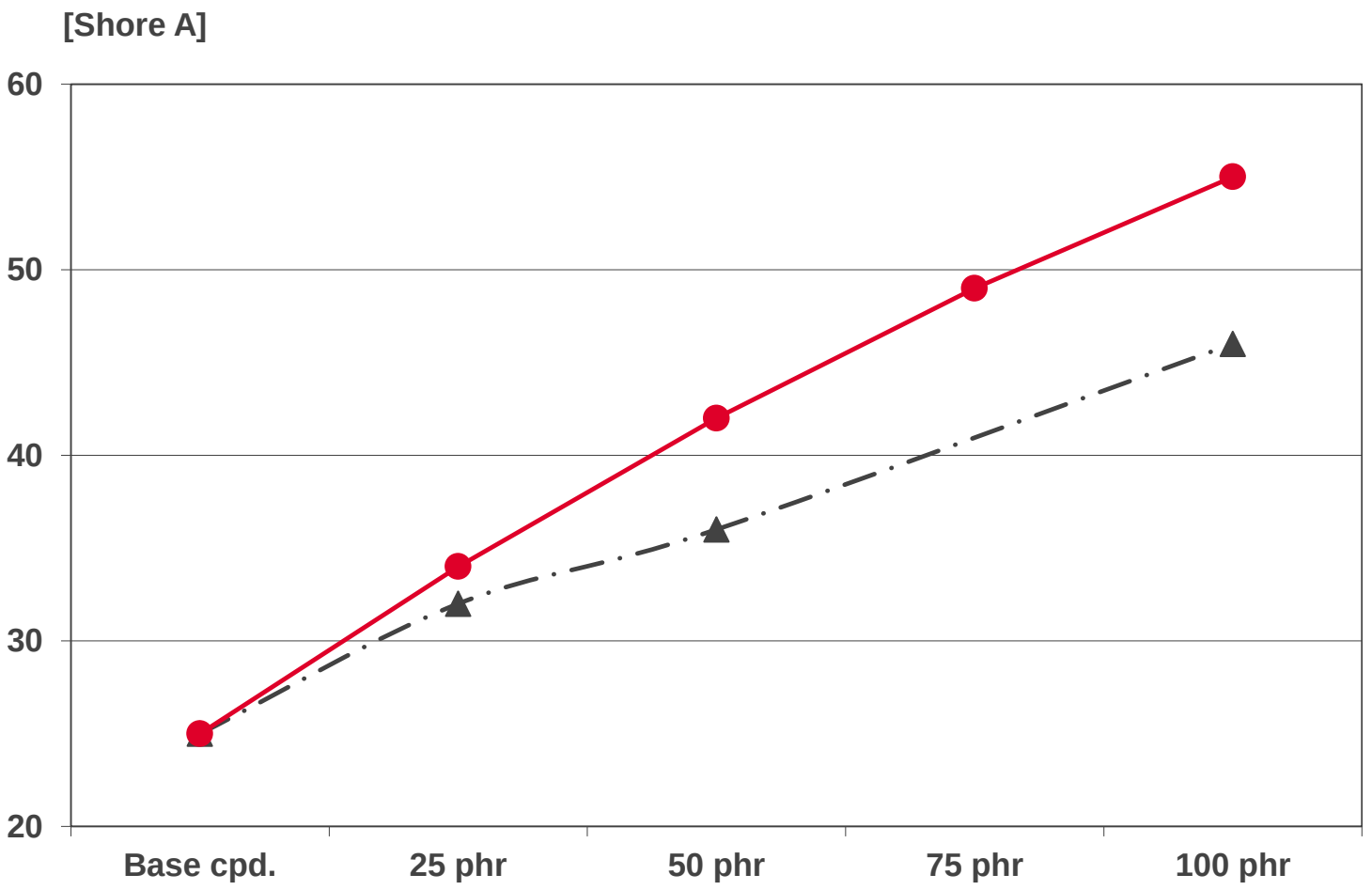
Post-cured, 4 h / 200°C



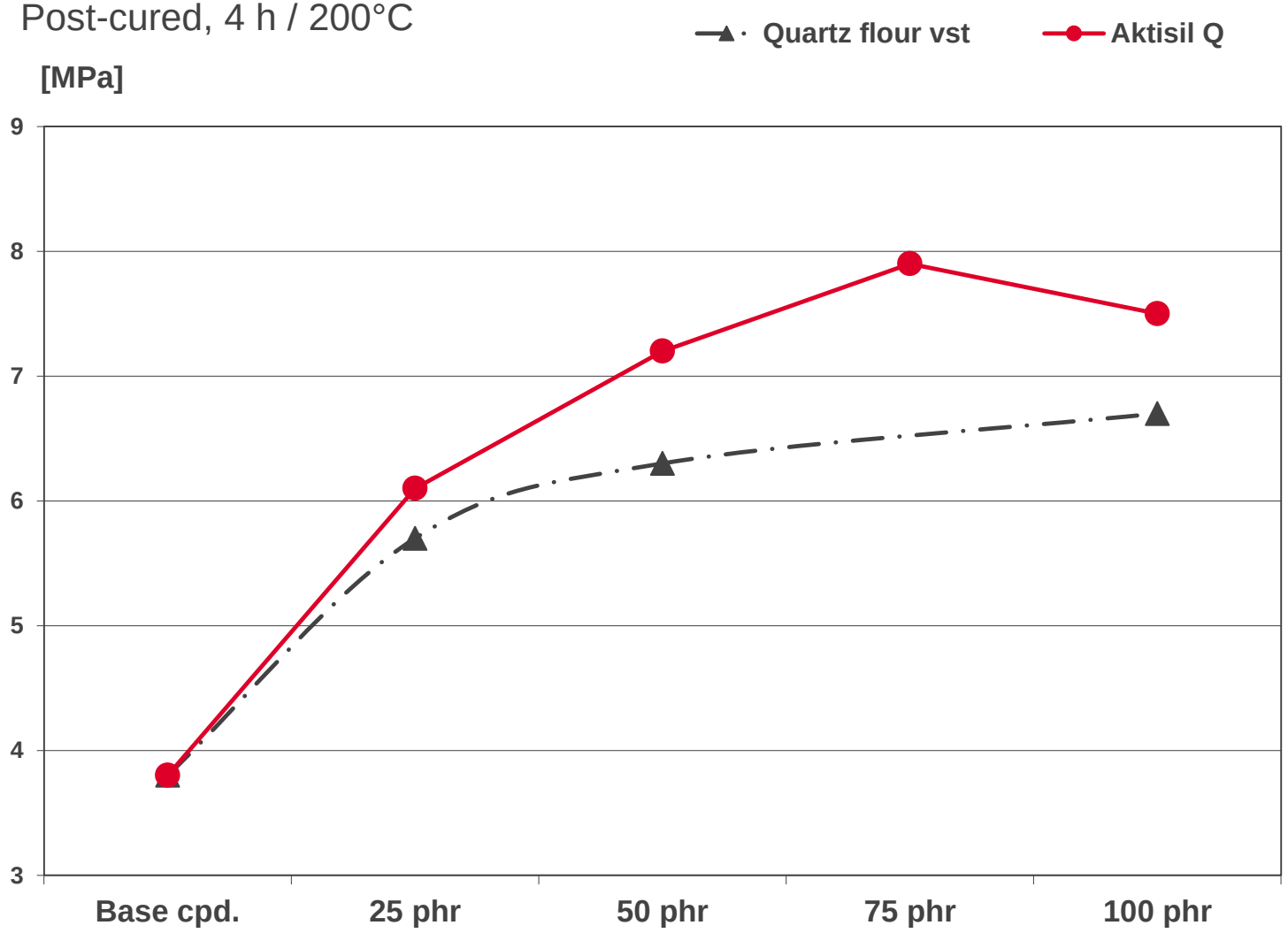
Oil IRM 903, 72 h / 150°C
Remaining Hardness

Post-cured, 4 h / 200°C



Oil IRM 903, 72 h / 150°C Remain. Tensile Strength

Post-cured, 4 h / 200°C



Oil IRM 903, 72 h / 150°C
Change of Tensile Strength

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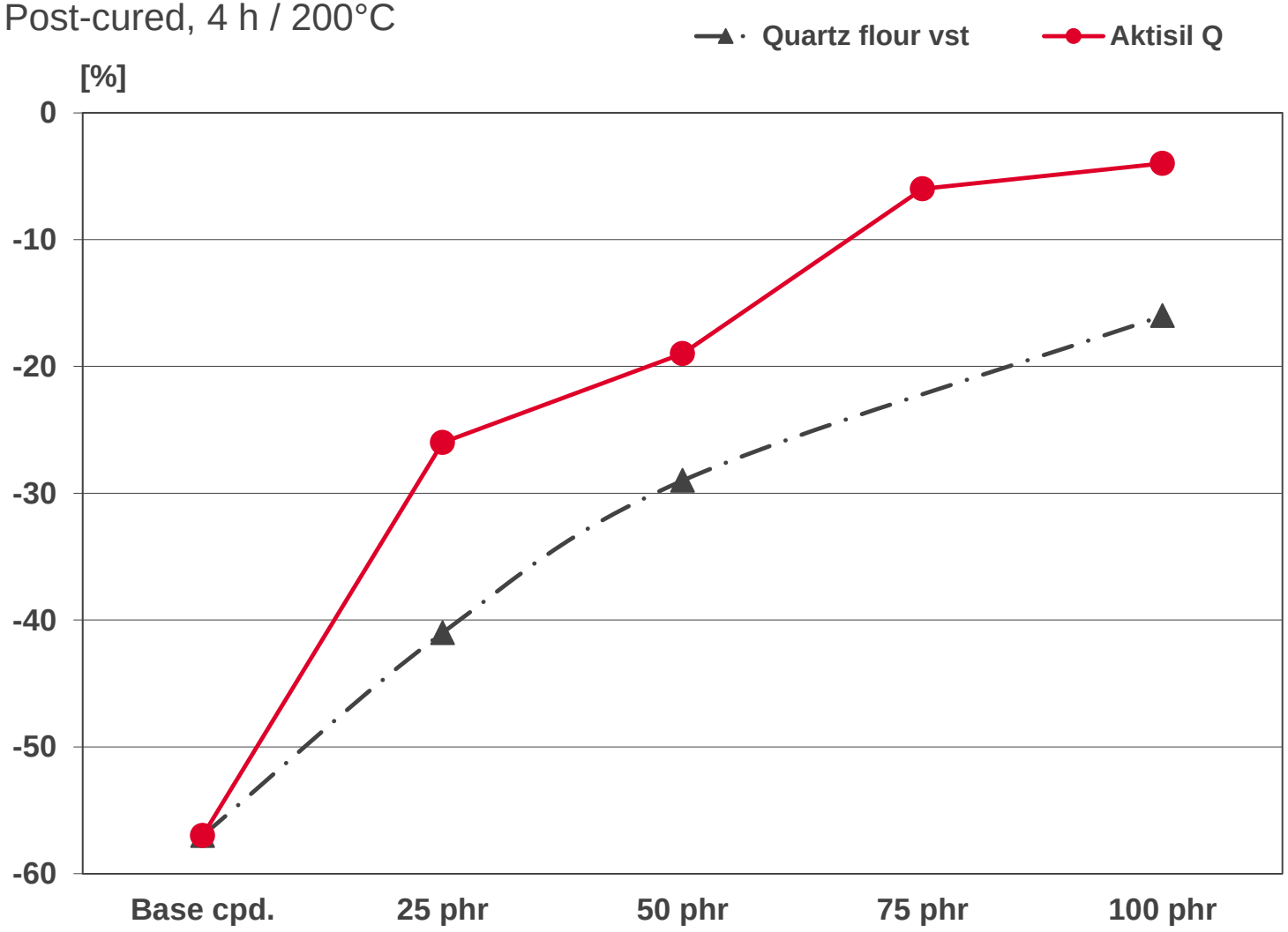
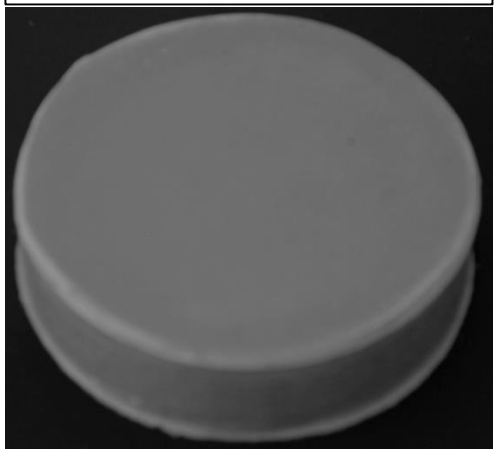
Post-cured, 4 h / 200°C



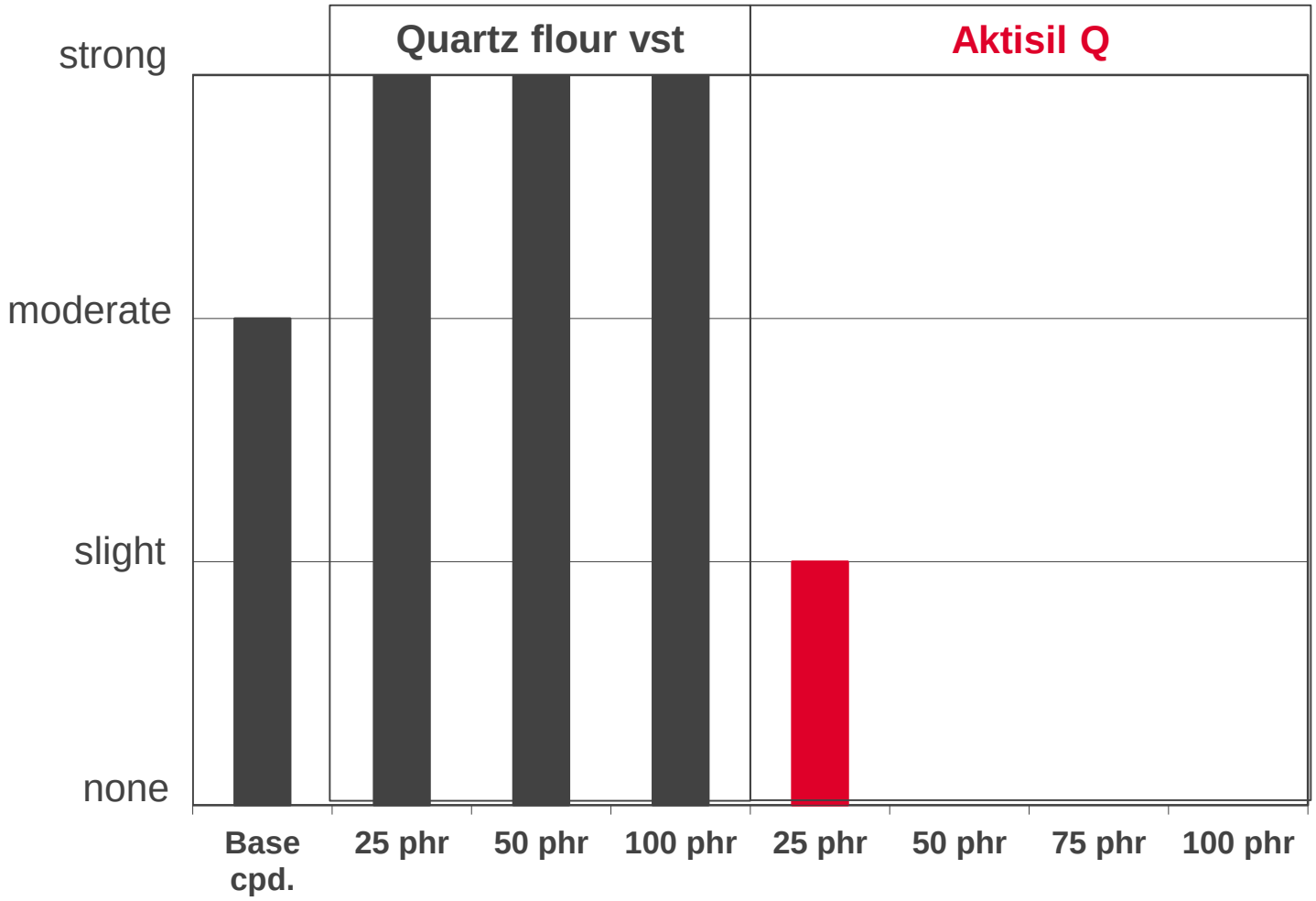
Blooming
using Curing Agent E

Compound colored dark (+ 4 phr black masterbatch)

After 4 Weeks

Post-cured, 4 h / 200°C		Press cured
Base compound	**Quartz flour vst**	**Aktisil Q**
		

Blooming
using Curing Agent E



Summary

Aktisil Q Benefits

- → Lower abrasivity and wear (quartz flour vst, diatomaceous earth calc.)
- → Post-cure can be left out depending on cure properties
- → Similar tensile properties (quartz flour vst)
- → Increase of the hardness level

  Equal hardness for 75 phr Aktisil Q versus 100 phr quartz flour vst
- → Post-cure not necessary using Bis-(2.4-dichlorobenzoyl)-peroxide or DCP (post-cure has a slight negative or no influence)
- → Improvement of compression set up to a loading of 75 phr without post cure (base compound, quartz flour vst, diatomaceous earth calcined)
- → Similar heat aging properties (base compound, quartz flour vst)
- → Improvement of oil resistance (base compound, quartz flour vst, diatomaceous earth calcined)
- → Elimination of blooming using Bis-(2.4-dichlorobenzoyl)-peroxide, even without post-cure (base compound, quartz flour vst)
- → Elimination of stickiness of uncured extrusions (base compound, quartz flour vst)
- → Excellent collapse resistance of extruded profiles and hoses (base compound, quartz flour vst, diatomaceous earth calcined)

Summary
Aktisil Q Benefits

The same effects will largely be found also with the other peroxides

- 2,5-bis-(tert-butylperoxy)-2,5-di-methylhexane
  (Elastosil AUX Curing Agent C 6)
- Dicumyl Peroxide
  (DCP, Perkadox BC-40S-ps)

We supply material for good ideas!

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