Neuburg Siliceous Earth in toughened 2K-epoxy-structural-adhesive

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Contents

Status Quo

- Adhesive bonding is already replacing conventional mechanical joining techniques in many technical applications.
- Structural adhesives are becoming increasingly important, particularly in vehicle and construction engineering.
- 1- or 2-component epoxy-resin based adhesives offer high strength and good chemical and temperature resistance.
- Toughness modifiers are often added to improve flexibility and thus increase shear and peel strength.
- Suitable fillers can also help to optimize the formulation.

System 1

Component A with epoxy-silicone block-copolymer

System 2

Component B with reactive liquid rubber (ATBN)

Formulation

Component A		Parts by weight [pbw]		
Epikote Resin 828LVEL	Epoxy-resin based on bisphenol A	80	80	80
Albiflex 297	Epoxy-silicone block-copolymer	20	20	20
Dynasylan 9116	Alkyl silane, adhesion promoter	3	3	3
Fumed silica	Filler	---	5	---
Neuburg Siliceous Earth	Filler	---	---	50
Total		**103**	**108**	**153**
Component B				
Ancamine 2719	Aliphatic amine (mannich base)	34.37	34.37	34.37
Total A + B		**137.37**	**142.37**	**187.37**

Preparation i

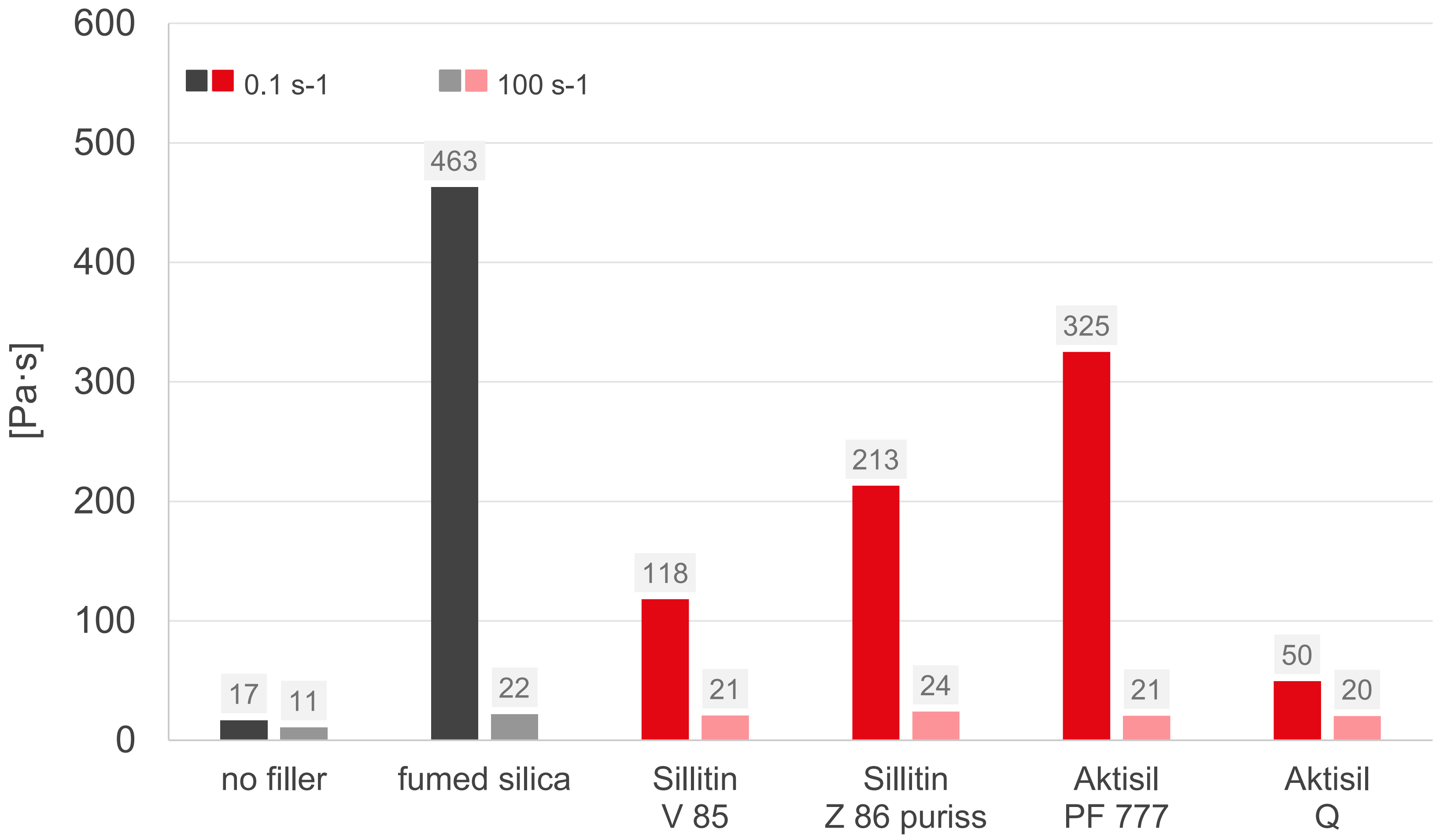
Filler characteristics

	Particle size		Color CIELab			Oil absorption	Density	Specific surface area BET	Surface treatment
	d_{50} [µm]	d_{97} [µm]	L* [-]	a* [-]	b* [-]	[g/100g]	[g/cm³]	[m²/g]	
Fumed silica	-	-	-	-	-	-	2.0	80-120	ja
Sillitin V 85	4.5	18	93.3	1.0	9.2	45	2.6	10	-
Sillitin Z 86 puriss	1.9	9	93.9	1.0	9.7	55	2.6	12	-
Aktisil PF 777	2.2	10	93.6	1.2	10.0	35	2.6	9	alkyl functionalized
Aktisil Q	4.0	18	94.7	0.3	4.1	43	2.6	6	methacryl functionalized

Structure of Neuburg Siliceous Earth i

Viscosity Component A

MCR 300, platte/platte PP25, measuring 7d after preparation



The addition of the filler increases the viscosity noticeably, especially in the low shear range.

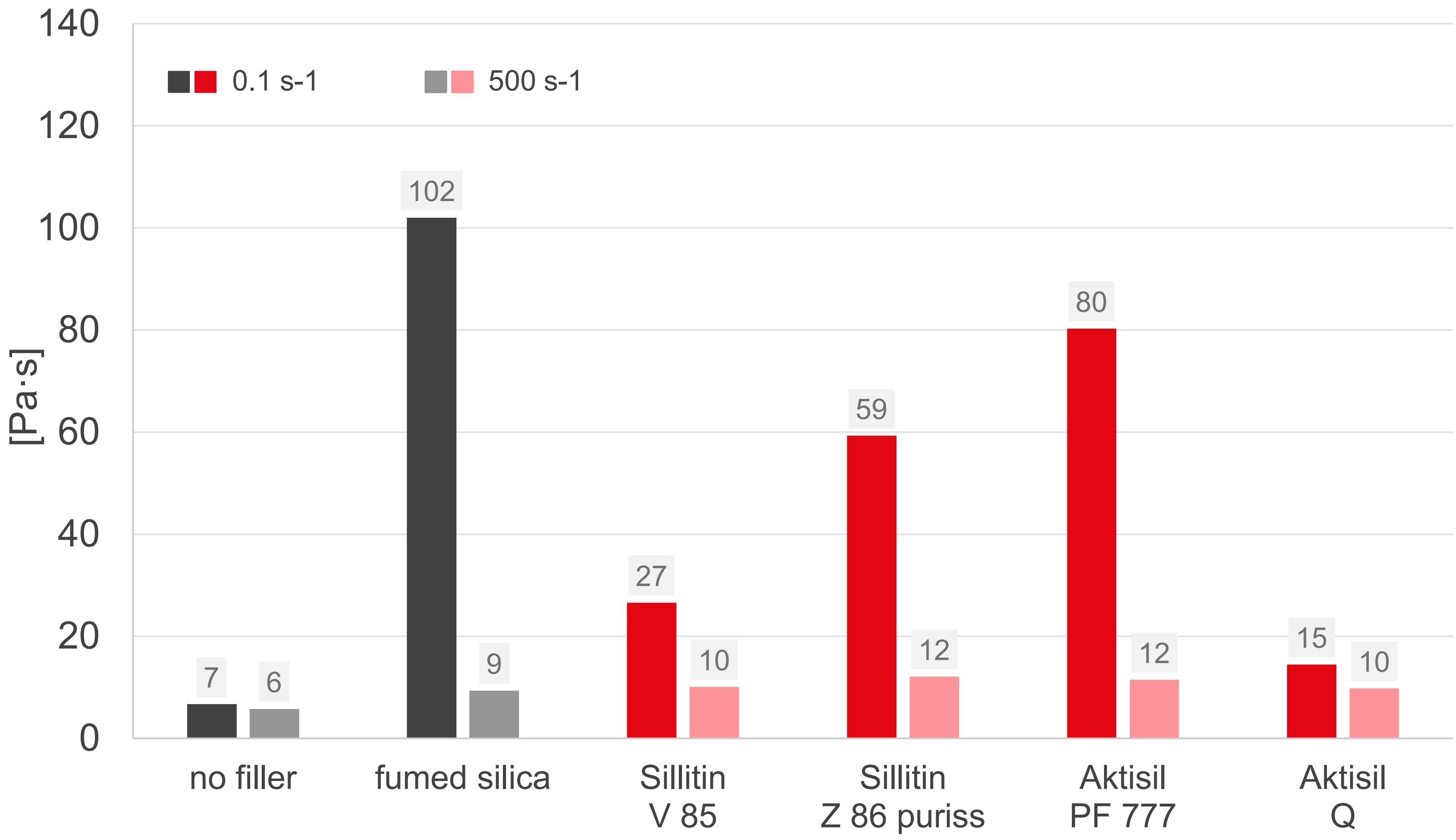
Despite the dosage of 50 pbw, Aktisil Q remains at a very low level, whereas Aktisil PF 777 gives the highest viscosity.

Storage stability

Sedimentation	storage at room temperature for up to 8 weeks: no sediment	✓
Rheology	storage at room temperature for up to 12 weeks: no change in rheology	✓

Viscosity Component A+B

MCR 300, plate/plate PP25



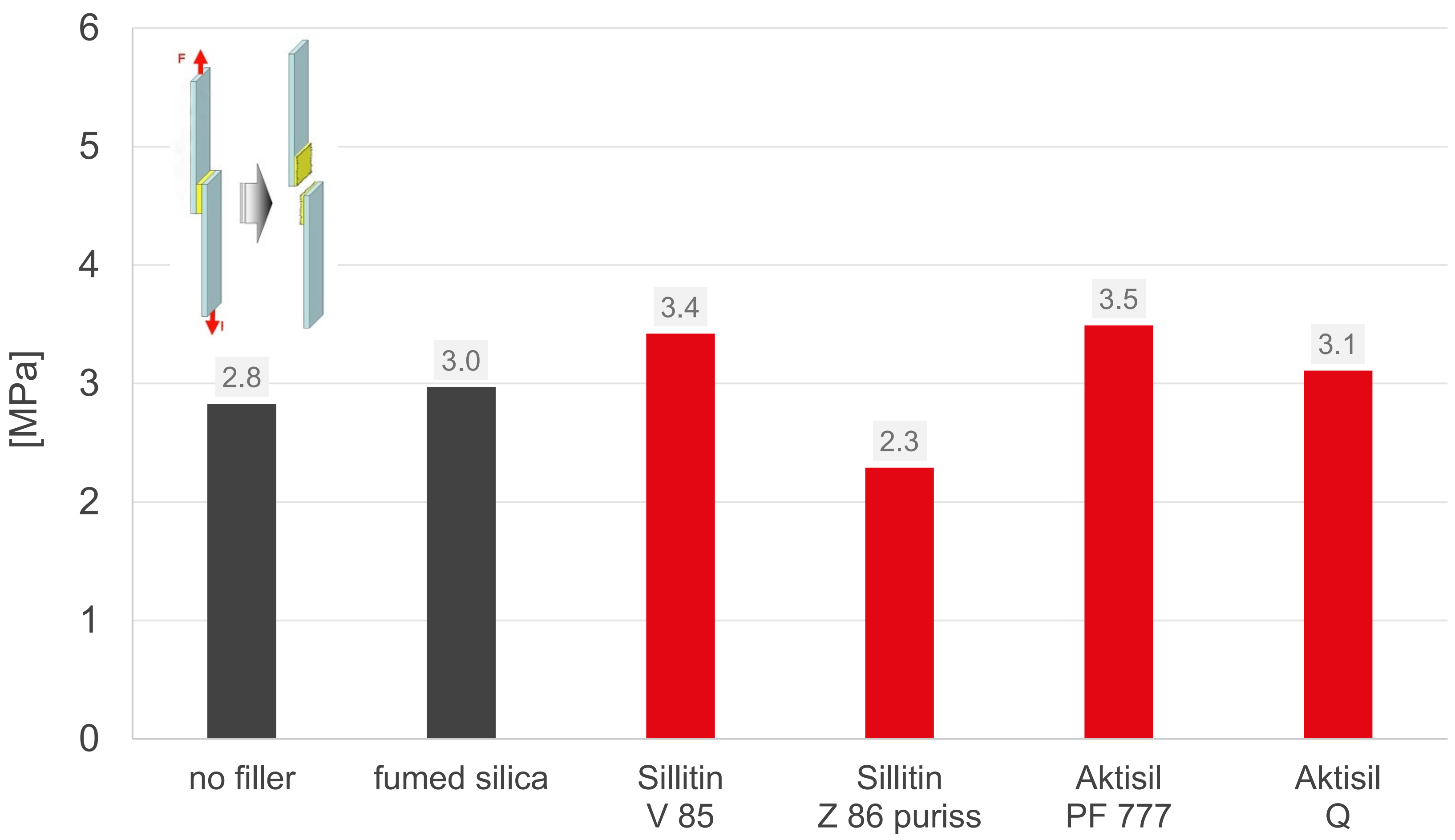
After adding the low-viscosity hardener:

Aktisil Q still shows the lowest viscosity, whereas the other fillers tested result in an ascending ranking of low shear viscosity or yield point.

Aktisil PF 777 marks the highest value after silica and thus the most pronounced shear thinning and thixotropy.

Lap Shear Strength

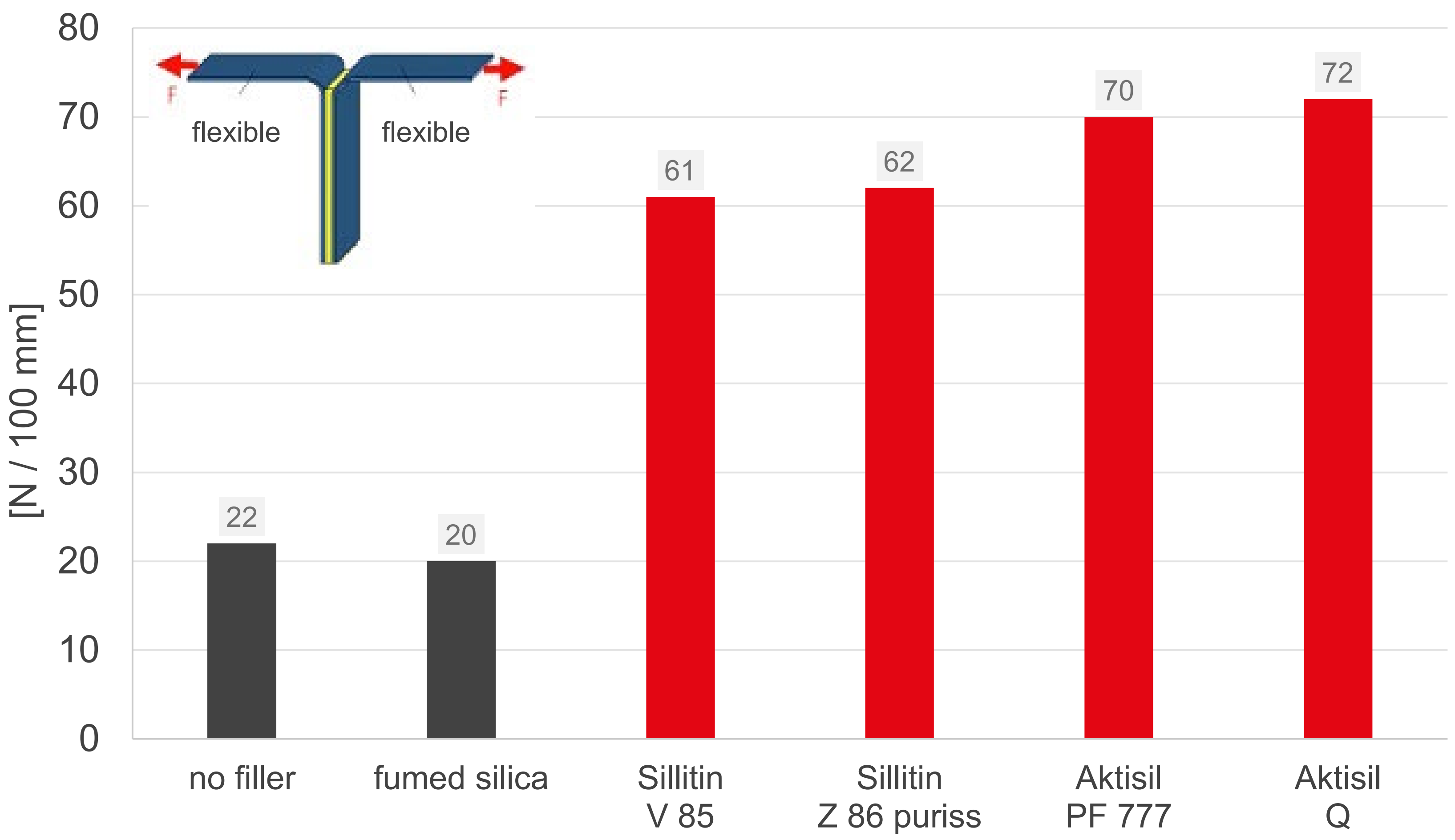
DIN EN 1465, Cr3 passivated aluminum, adhesive layer thickness 100 µm



Neuburg Siliceous Earth results in a similar to slightly increased lap shear strength compared to the unfilled formulation or the competitor silica.

T-Peel Test

DIN EN ISO 11339, Cr3 passivated aluminum, adhesive layer thickness 100 µm



With
Neuburg Siliceous Earth,
the peel resistance can be more than tripled.

Rating

	Fumed silica	Sillitin V85	Sillitin Z 86 puriss	Aktisil PF 777	Aktisil Q
Viscosity	↑↑	↑↑	↑↑	↑↑	0/↑
Lap shear strength	0	0 / +	0	0 / +	0
Peel resistance	0	++	++	++	++
Cost aspect	---	+++	+++	++	++
Note		standard product	easy to disperse	rheologically active	low viscosity

Benefits of
Neuburg Siliceous Earth
vs. unfilled formulation:

comparable or higher
lap shear strength

significantly higher
peel resistance

positive cost aspect
through the use of filler

System 1

Component A with epoxy-silicone block-copolymer

System 2

Component B with reactive liquid rubber (ATBN)

Formulation

Component A		Parts by weight [pbw]	
Epikote Resin 320	Epoxy-resin based on bisphenol A/F	100	100
Neuburg Silceous Earth	Filler	---	50
Total		**100**	**150**
Component B			
Epikure Curing Agent 05903	Hardener (mannich base)	34	34
Hypro 1300x16 ATBN	reactive liquid rubber	8.5	8.5
Total A + B		**142.5**	**192.5**

Preparation i

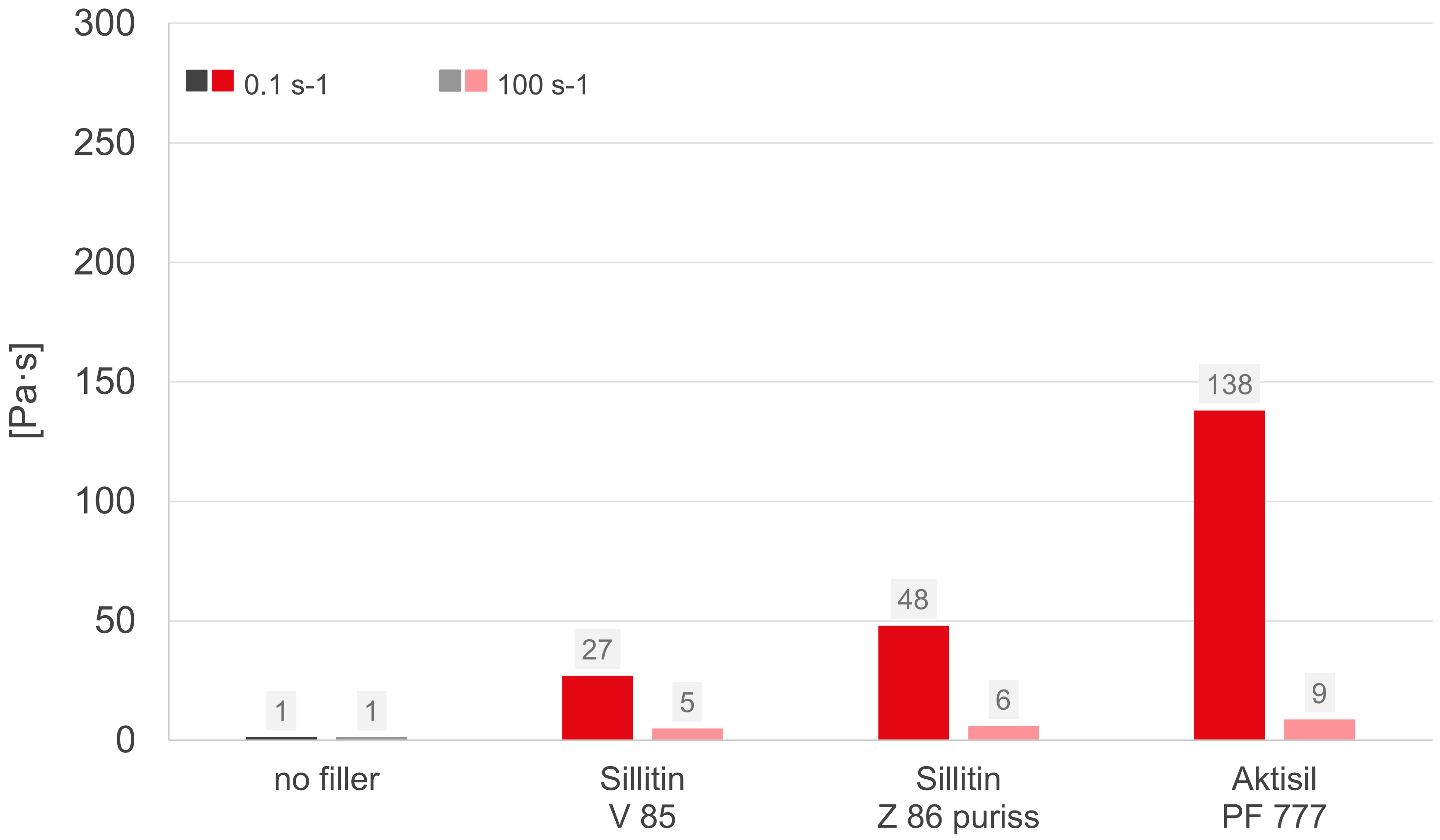
Filler characteristics

	Particle size		Color CIELab			Oil absorption	Density	Specific surface area BET	Surface treatment
	d_{50} [µm]	d_{97} [µm]	L* [-]	a* [-]	b* [-]	[g/100g]	[g/cm³]	[m²/g]	
Sillitin V 85	4.5	18	93.3	1.0	9.2	45	2,6	10	-
Sillitin Z 86 puriss	1.9	9	93.9	1.0	9.7	55	2,6	12	-
Aktisil PF 777	2.2	10	93.6	1.2	10.0	35	2,6	9	alkyl functionalized

Structure of Neuburg Siliceous Earth i

Viscosity Component A

MCR 300, platte/platte PP25, measuring 7d after preparation



The addition of the filler increases the viscosity noticeably, especially in the low shear range.

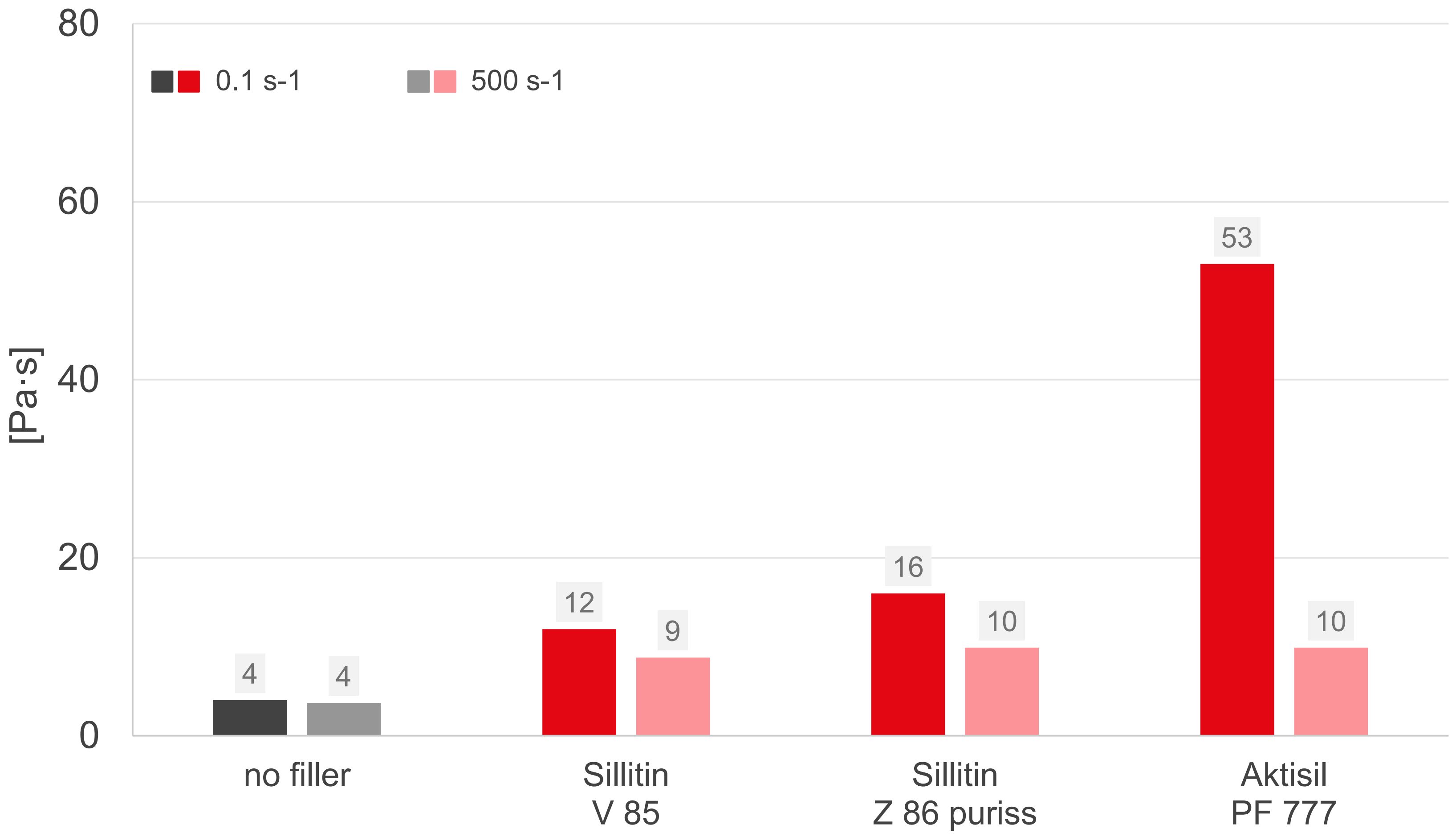
Sillitin V 85 remains at a moderate level despite the dosage of 50 GT, whereas Aktisil PF 777 gives the highest viscosity

Storage stability

Sedimentation	storage at room temperature for up to 8 weeks: no sediment	✓
Rheology	storage at room temperature for up to 12 weeks: no change in rheology	✓

Viscosity Component A+B

MCR 300, plate/plate PP25



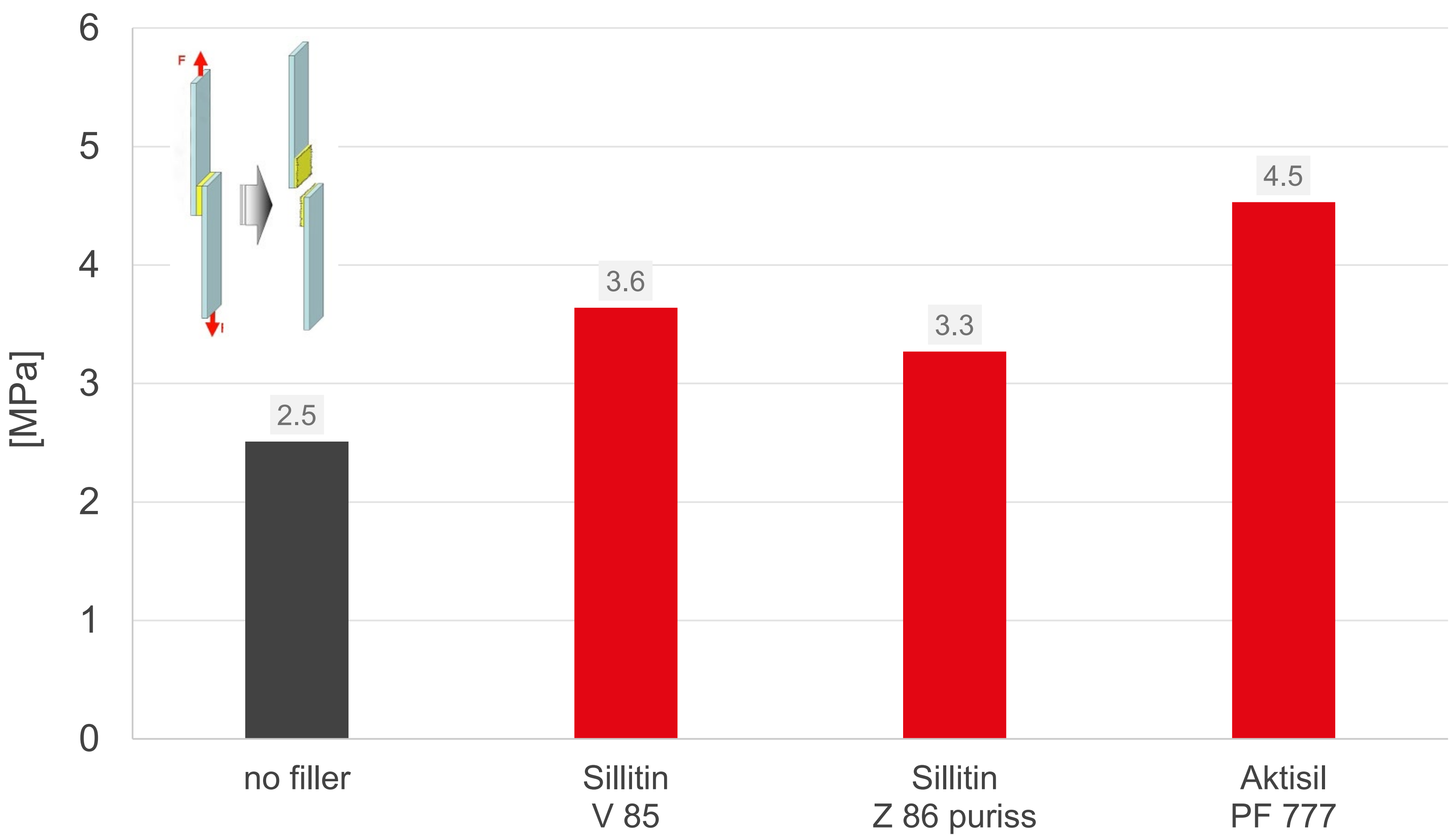
After adding the hardener:

Sillitin V 85 still shows the lowest viscosity, whereas the other fillers tested result in an ascending ranking of low shear viscosity or yield point.

Aktisil PF 777 marks the highest value and thus the most pronounced shear thinning and thixotropy.

Lap Shear Strength

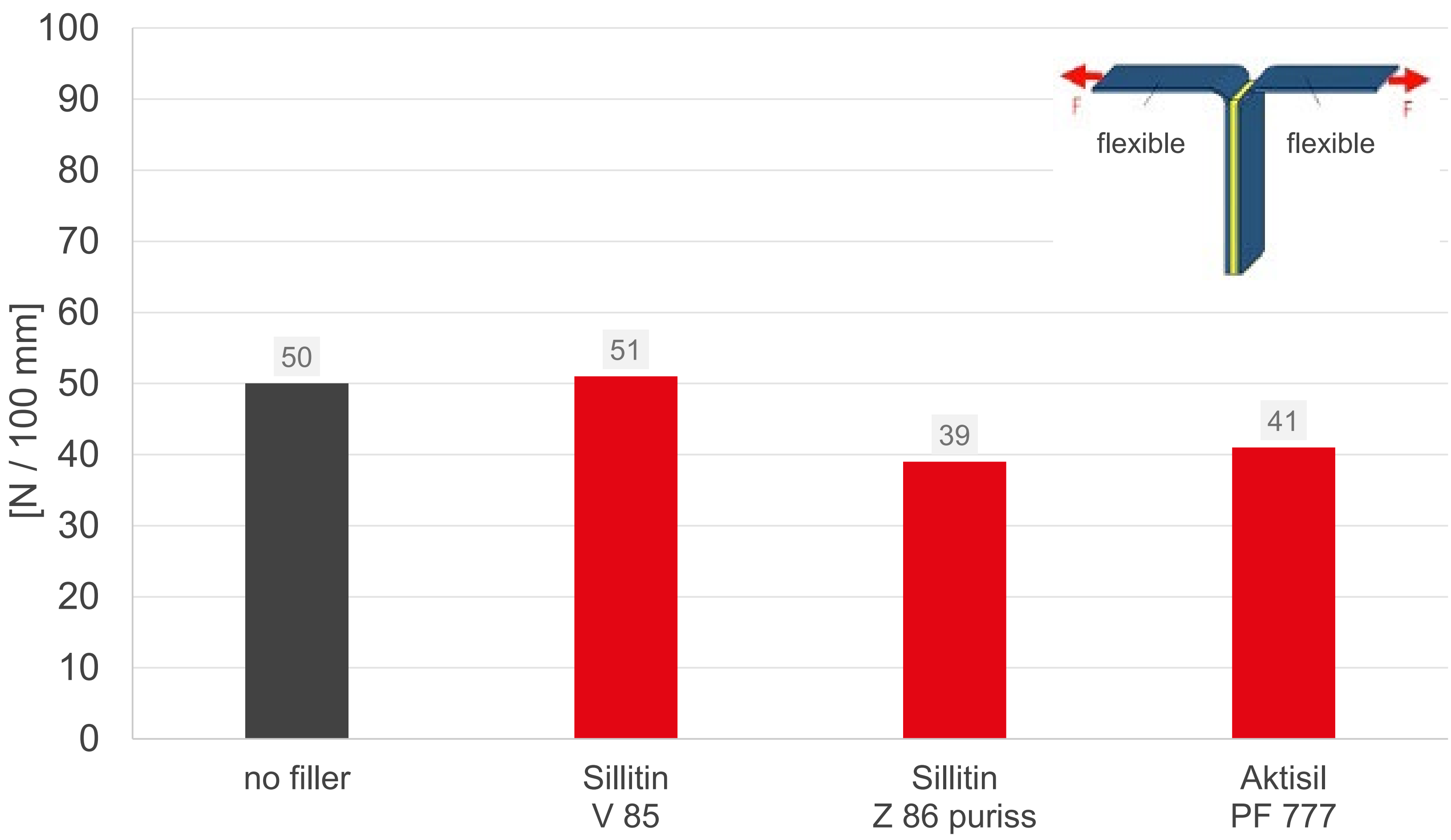
DIN EN 1465, Cr3 passivated aluminum, adhesive layer thickness 100 µm



Neuburg Siliceous Earth results in a noticeable increased lap shear strength.

T-Peel Test

DIN EN ISO 11339, Cr3 passivated aluminum, adhesive layer thickness 100 µm



The peel resistance with Neuburg Siliceous Earth is approximately at the level of the unfilled formulation.

Rating

	Sillitin V85	Sillitin Z 86 puriss	Aktisil PF 777
Viscosity	↑↑	↑↑	↑↑
Lap shear strength	+	0 / +	+
Peel resistance T-Peel	0	0 / -	0 / -
Cost aspect	+++	+++	++
Note	standard product	easy to disperse	rheologically active

Benefits of
Neuburg Siliceous Earth
vs. unfilled formulation:

comparable or higher lap shear strength

comparable peel resistance

positive cost aspect through the use of filler

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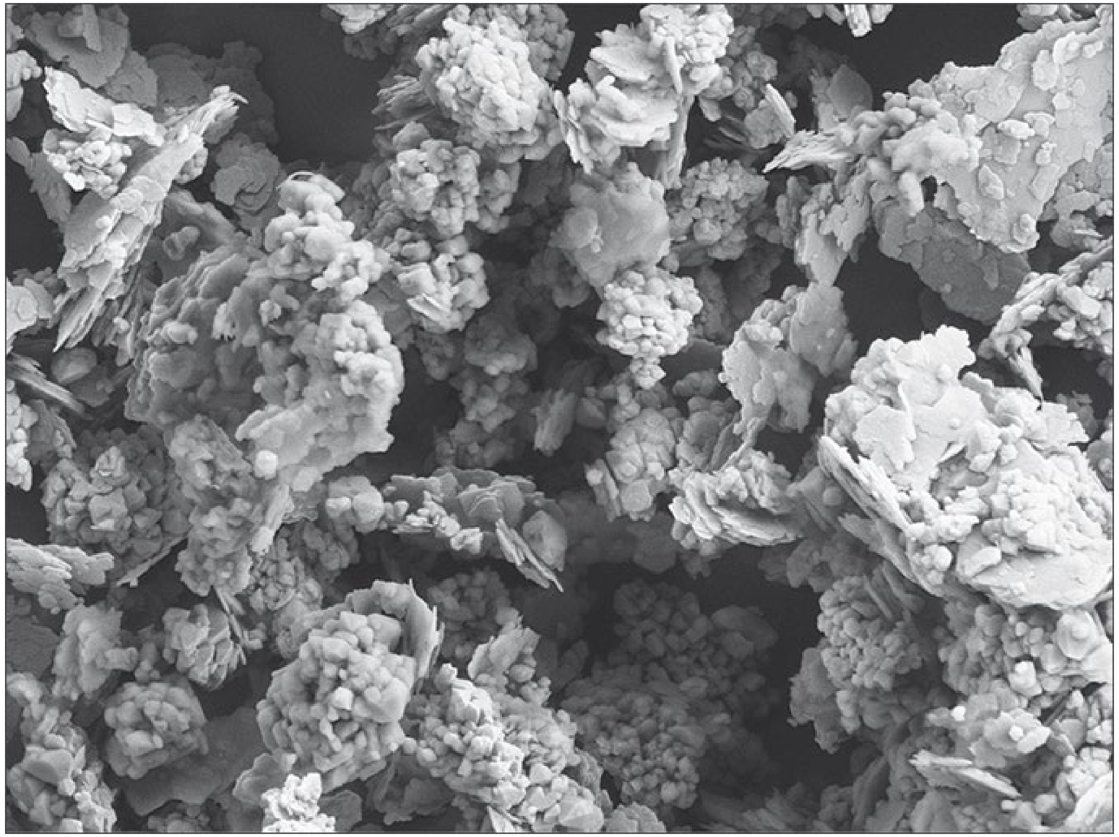
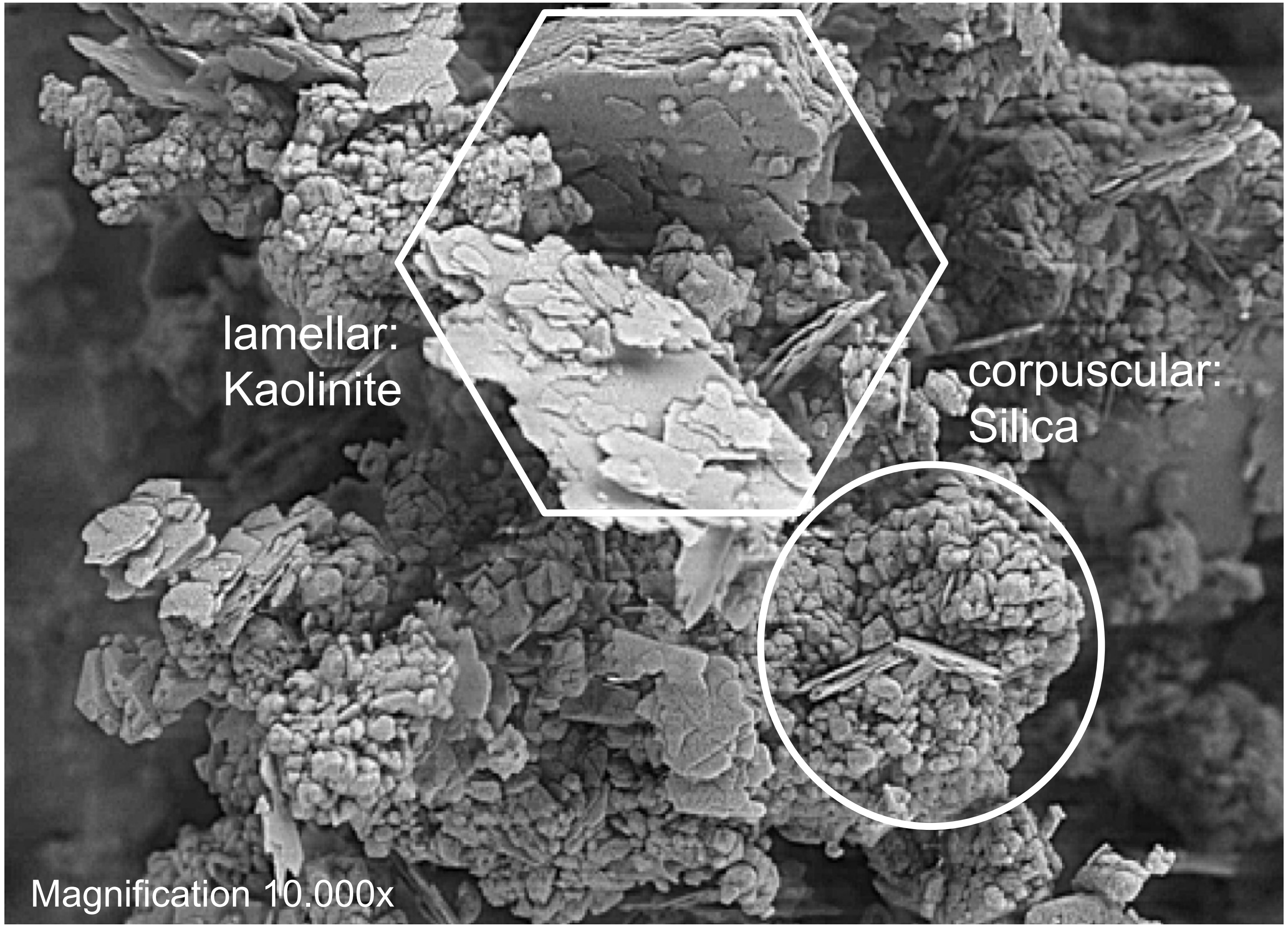
Neuburg Siliceous Earth



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.

Morphology of Neuburg Siliceous Earth



Preparation System 1

Component A	Speedmixer Resin, Abiflex and silane — 60 s @ 1000 rpm + 120 s @ 2000 rpm + first part of filler — 30 s @ 800 rpm + rest of filler — 30 s @ 800 rpm Clean lid and rim — 60 s @ 1000 rpm Clean lid, rim and bottom — 300 s @ 2000 rpm Clean lid, rim and bottom — 60 s @ 1000 rpm + 120 s @ 2000 rpm
Mixing Component A+B	Speedmixer 60 s @ 1000 rpm + 120 s @ 2000 rpm

Quelle: Hauschild

Results in tabular form – System 1

			no filler	fumed silica	Sillitin V 85	Sillitin Z 86 puriss	Aktisil PF 777	Aktisil Q
Rheology								
Component A	Viscosity @ 0.1 s^{-1} Viscosity @ 100 s^{-1}	Pa·s	17 11	463 22	118 21	213 24	325 21	50 20
Component A+B	Viscosity @ 0.1 s^{-1} Viscosity @ 100 s^{-1}	Pa·s	7 6	102 9	27 10	59 12	80 12	15 10
Storage stability Component A								
Sedimentation, 8 w @ RT Change in rheology, 12 w @ RT			-	without no	without no	without no	without no	without no
Mechanical properties								
Lap shear strength Cr3 passivated aluminum, 100 µm		MPa	2.8	3.0	3.4	2.3	3.5	3.1
Peel resistance T-Peel Cr3 passivated aluminum, 100 µm		N / 100 mm	22	20	61	62	70	72

Preparation – System 2

Component A	Speedmixer Resin + first part of filler — 30 s @ 800 rpm + rest of filler — 30 s @ 800 rpm Clean lid and rim — 60 s @ 1000 rpm Clean lid, rim and bottom — 300 s @ 2000 rpm
Mixing Component A+B	Speedmixer 60 s @ 1000 rpm + 120 s @ 2000 rpm

Quelle: Hauschild

Results in tabular form – System 2

			ungefüllt	Sillitin V 85	Sillitin Z 86 puriss	Aktisil PF 777
Rheology						
Component A	Viscosity @ 0.1 s^{-1} Viscosity @ 100 s^{-1}	Pa·s	1 1	27 5	48 6	138 9
Component A+B	Viscosity @ 0.1 s^{-1} Viscosity @ 100 s^{-1}	Pa·s	4 4	12 9	16 10	53 10
Storage stability Component A						
Sedimentation, 8 w @ RT Change in rheology, 12 w @ RT			-	without no	without no	without no
Mechanical properties						
Lap shear strength Cr3 passivated aluminum, 100 µm		MPa	2.5	3.6	3.3	4.5
Peel resistance T-Peel Cr3 passivated aluminum, 100 µm		N / 100 mm	50	51	39	41

Overview tests

Rheology	MCR 300, PP25, 1 mm gap, 23 °C, logarithmic flow curve from 0.05-500 s^{-1} (rotation)
Sedimentation	Storage 8 weeks at room temperature
Application	using a disposable syringe, immediately after mixing the two components
Lap shear strength	DIN EN 1465 Substrate: Aluminum 5005 H24 (AlMg1(B)) with Cr3 passivation Adhesive layer: 100 µm, adjusted by 0.2 or 0.5 Vol-% glass beads (on total batch) Curing: 14 days at standard climate 23/50 Test speed: 1 mm/min Evaluation: Average maximum stress (tensile shear strength)
T-peel test	DIN EN ISO 11339 Substrate: Aluminum 3003 H24 (AlMg1Cu) with Cr3 passivation Adhesive layer: 100 µm, adjusted by 0.2 or 0.5 Vol-% glass beads (on total batch) Curing: 14 days at standard climate 23/50 Test speed: 100 mm/min Evaluation: Average peel force, according to standard based on 100 mm sample width