

Calcined Neuburg Siliceous Earth

in thermoplastics:

Polypropylene (PP)

Author: Hubert Oggermüller
Petra Zehnder

Contents

- 1 Introduction
- 2 Experimental
 - 2.1 Neuburg Siliceous Earth
 - 2.2 Fillers and their characteristics
 - 2.3 Compounding and molding
- 3 Results PP Copolymer, 20 % filler content
 - 3.1 Melt volume-flow rate
 - 3.2 Impact strength Charpy
 - 3.3 Tensile test
 - 3.4 Heat deflection
 - 3.5 Indentation by writing and scratch resistance
- 4 Results PP Copolymer, 40 % filler content
 - 4.1 Melt volume-flow rate
 - 4.2 Impact strength Charpy
 - 4.3 Tensile test
 - 4.4 Heat deflection
 - 4.5 Indentation by writing and scratch resistance
- 5 Results PP Homopolymer, 40 % filler content
 - 5.1 Melt volume-flow rate
 - 5.2 Impact strength Charpy
 - 5.3 Tensile test
 - 5.4 Heat deflection
 - 5.5 Scratch resistance
- 6 Summary
- 7 Appendix: Summary tables

1 Introduction

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, in the electricity sector and in the construction sector; a large part is used for 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.

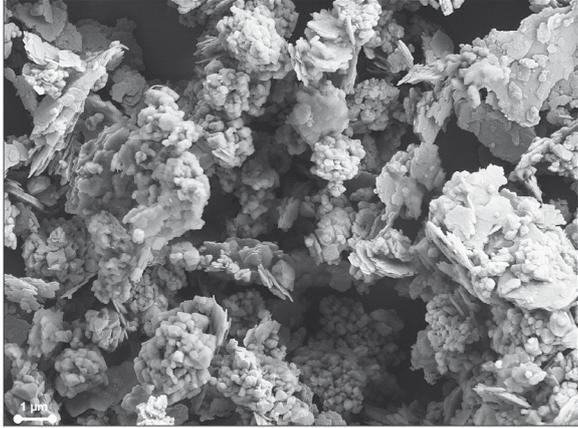
The present report will introduce calcined Neuburg Siliceous Earth grades as functional fillers for Polypropylene.

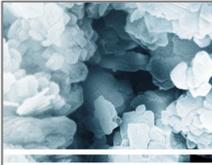
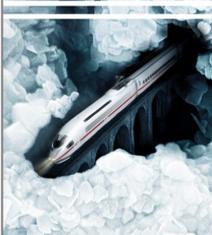
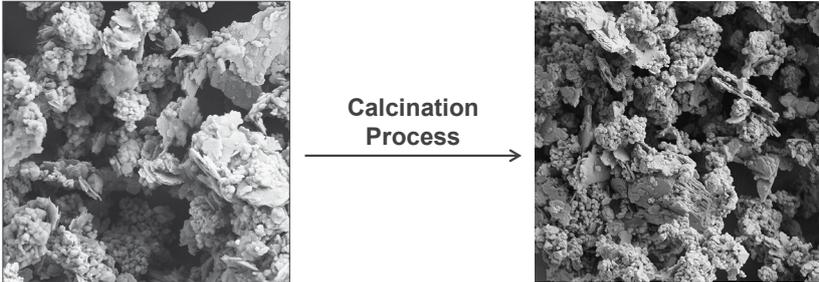
This includes a comparison of the property profiles of different grades of Calcined Neuburg Siliceous Earth with a talc grade appreciated for high scratch resistance and impact strength. The comparison looked above all at flow properties, dimensional stability at high temperature, mechanical properties and resistance against scratching and indentation by writing.

2 Experimental

2.1 Neuburg Siliceous Earth

The special morphological composition of Neuburg Siliceous Earth, which represents a class of minerals on its own, is illustrated here by a SEM photograph.

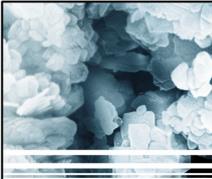
 INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY APPENDIX 	<h2>Structure</h2> 
	 <p>A natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods.</p> <p>The silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.</p> <p>VM-01/0416/07.2018</p>

 INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY APPENDIX 	<h2>Calcined Neuburg Siliceous Earth</h2> 
	<p>A downstream thermal process lead to the calcined products Silfit and Aktifit, based on SILLITIN Z 86.</p>  <p>Neuburg Siliceous Earth → Calcination Process → Calcined Neuburg Siliceous Earth</p> <p>Additional application benefits, as well as the removing of crystal water included in the kaolinite. The silica part remains inert.</p> <p>VM-01/0416/07.2018</p>

During calcination, Neuburg Siliceous Earth is subjected to a heat treatment. The components and the thermal process lead to a product that offers special performance benefits as a functional filler.

2.2 Fillers and their characteristics

The table shows a summary of the most important filler properties.

		Talc ultrafine*		Calcined Neuburg Siliceous Earth	
				Silfit Z 91	Aktifit AM
 INTRODUCTION <u>EXPERIMENTAL</u> RESULTS SUMMARY APPENDIX 					
	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	[m ² /g]	12	7.4	6.6
	Sieve residue > 40 µm	[mg/kg]	2	8	21
	Functionalization		none	none	Amino
	<small>*manufacturer information: d₅₀: 1 µm, d₉₅: 3 µm; special grade for good impact strength and scratch resistance</small>				
VM-01/0416/07.2018					

The comparison was made against a premium talc, an ultrafine dark-colored grade which is particularly recommended for high impact strength and scratch resistance.

From the portfolio of Neuburg Siliceous Earth grades, Silfit Z 91 and Aktifit AM were included. Silfit Z 91 is regarded as a cost-conscious filler without surface modification.

Aktifit AM is an activated Silfit Z 91 where the surface has been modified with an amino functional group.

2.3 Compounding and molding

The tests were carried out with a PP copolymer with a MFR of 16 g/10 min and a homopolymer with 17 g/10 min. The copolymer compound contained 40 resp. 20 weight percent of filler, the homopolymer was tested only at 40 %.

The compounding was made in a Werner & Pfleiderer twin-screw extruder ZSK 30 (screw diameter 30 mm).

The plastic pellets were mixed with the stabilizer and black color batch and fed into the main stream. The mass temperature was adjusted close to 250 °C. The filler was added to the melt via side feeder. The extruded strands were pelletized by cold-face cutting.

The preparation of the test specimens was made on a screw injection molding unit from Krauss Maffei, using a specimen tool according to ISO 294 with exchangeable inserts for the individual test specimens.

The PP-granules were injected with a melt temperature of 230 °C at a mold temperature of 40 °C without pre-drying.

Compounding, injection molding and subsequent tests were carried out at A. Schulman in Kerpen, Germany.

3 Results PP Copolymer, 20 % filler content

Formulations

INTRODUCTION <u>EXPERIMENTAL</u> • Copolymer 20 % Filler RESULTS SUMMARY APPENDIX	Formulations PP Copo 20 % Filler							HOFFMANN MINERAL
	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	

VM-01/0416/07.2018

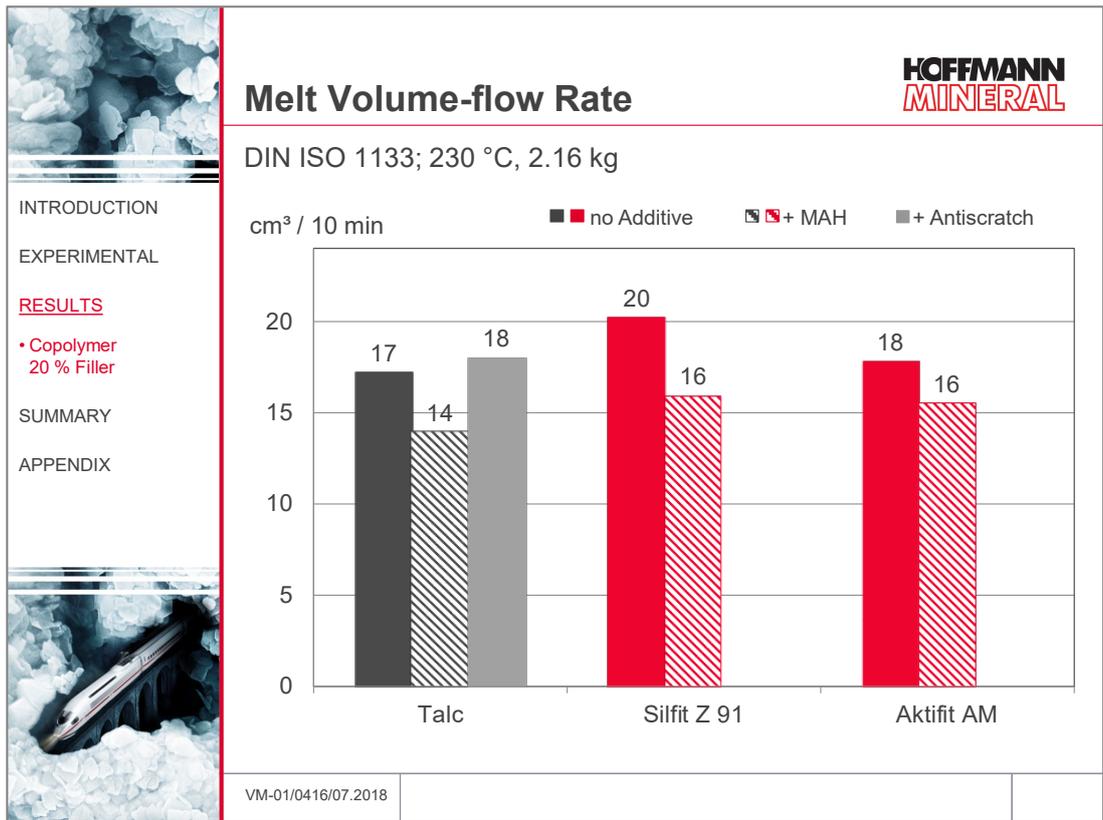
The PP was added with a stabilizer and a black color batch. For this part of the study, the filler loading was 20 weight percent.

For this test block, the filler choice comprised, among the calcined Neuburg Siliceous Earth grades, the products Silfit Z 91 and Aktifit AM, as well for comparison purposes a premium talc grade.

All fillers were also evaluated in combination with a PP grafted with maleic anhydride (MAH) for the chemical coupling of the filler to the PP matrix. Further controls were run with a compound loaded with the premium talc and a special siloxane based anti-scratch additive.

3.1 Melt volume-flow rate

Samples for this test were taken from the homogenized pellets ready for injection molding.



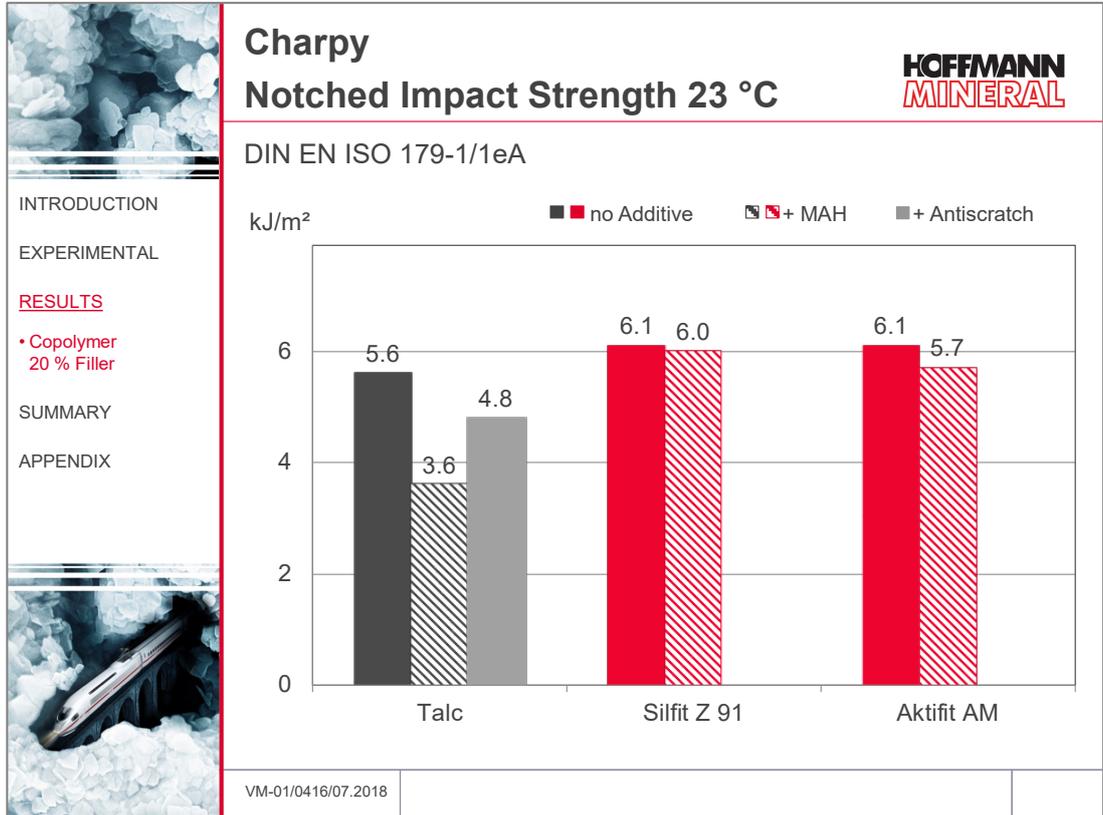
The flow properties of the compounds with Neuburg Siliceous Earth grades are found on a similar level with the talc loaded samples. MAH with all fillers exerts the same negative influence on the flow behavior.

3.2 Impact strength Charpy

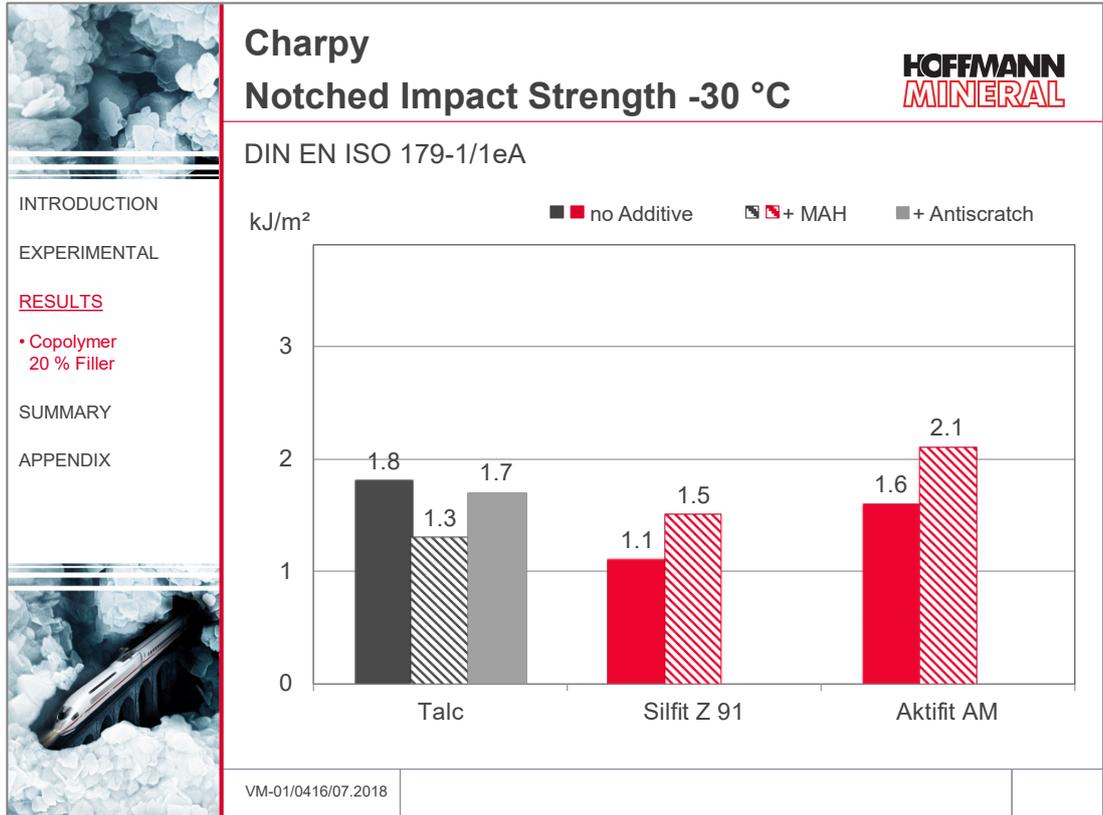
According to the Charpy method, the sample is supported unclamped at both ends and hit in the middle with a pendulum hammer.

Notched impact strength

For this test, the standard samples are provided in the middle with a single notch of the preferred kind A (notch root radius 0.25 mm, rest ground width 8.0 mm). The impact strength was determined according to the standard test with an impact on the narrow side opposite the notch (edgewise).



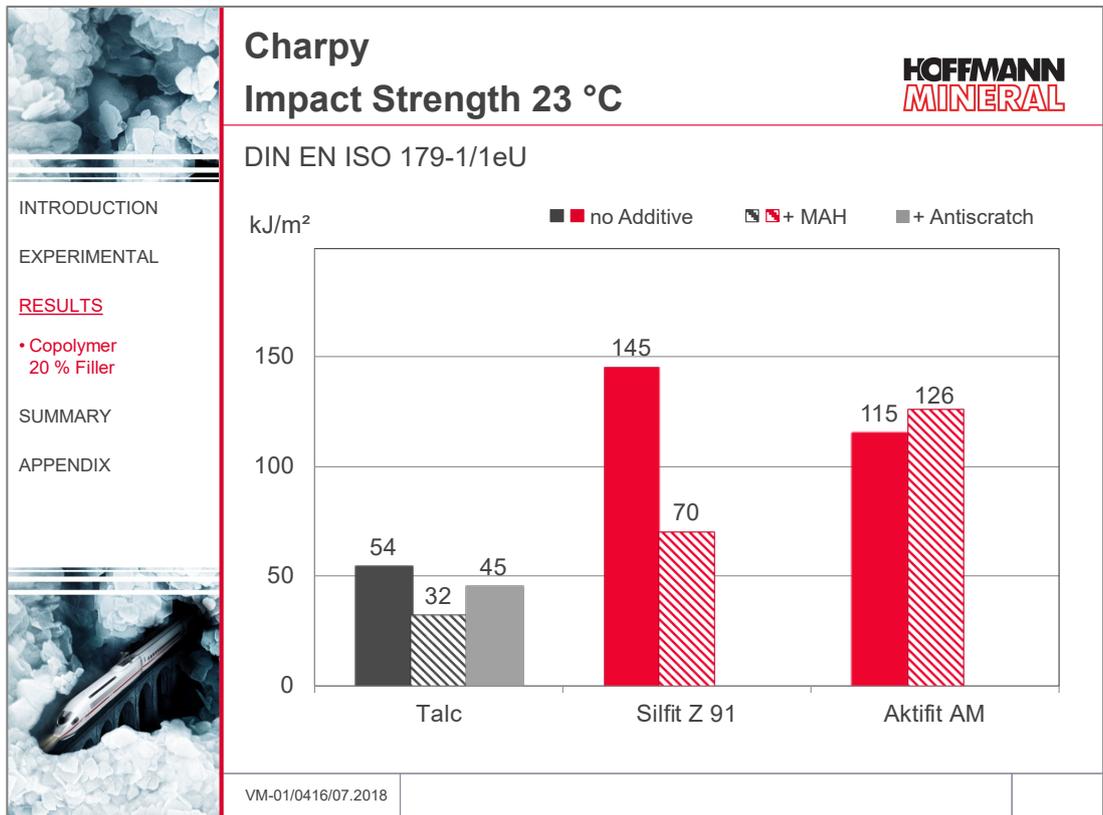
Here the Neuburg Siliceous Earth grades give rise to somewhat better results than the talc. With the talc, the addition of MAH or the anti-scratch agent leads to a decrease of the notched impact strength. By contrast, in case of the Neuburg Siliceous Earth grades the notched impact strength remains almost unchanged with values around 6 kJ/m².



At low temperature, the level of the notched impact strength, according to expectations, overall turns out markedly lower. The addition of MAH with Calcined Neuburg Siliceous Earth grades leads to a slight increase, with Aktifit AM giving the relatively best results.

Unnotched impact strength

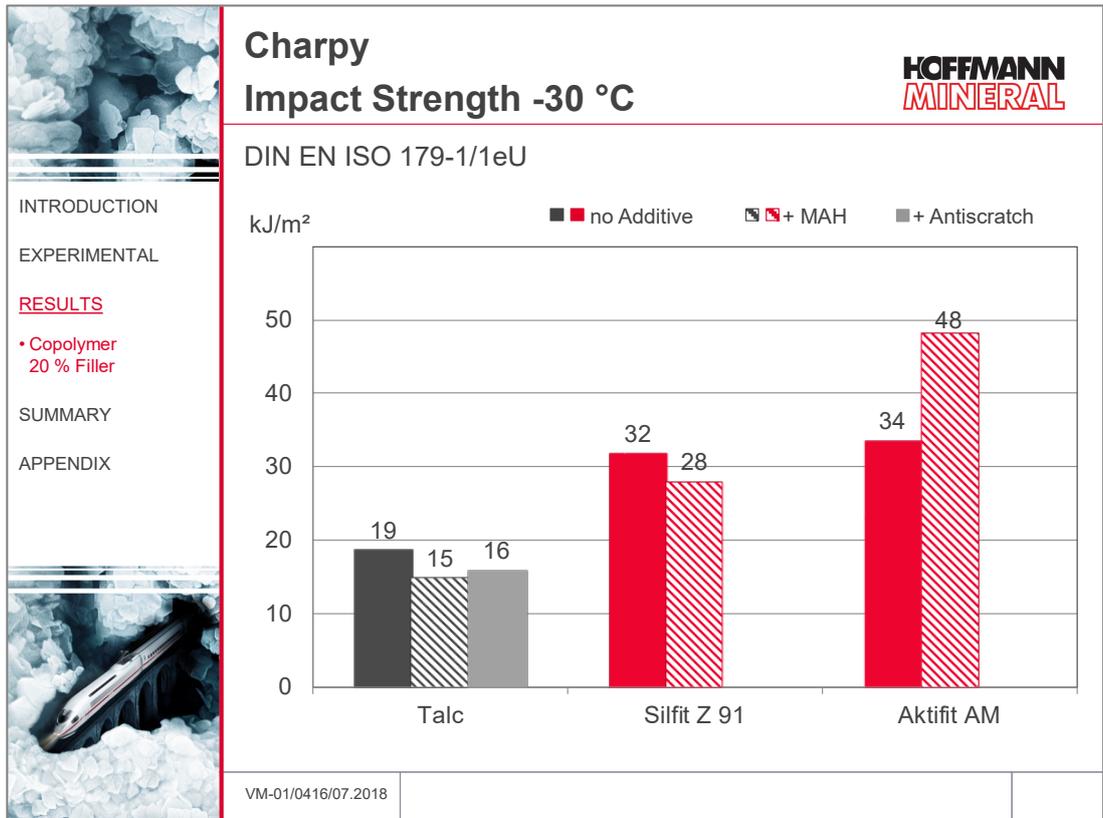
The test was run on unnotched standard samples, also with the impact hitting the narrow side of the sample.



With respect to impact strength, Calcined Neuburg Siliceous Earth can readily outperform the talc. The results obtained are two to three times higher than with the talc. This is all the more

remarkable as the talc grade used is recommended by the manufacturer for very good impact strength.

The addition of MAH with Aktifit AM gives rise to a further improvement of the impact resistance, while with Silfit Z 91 the contrary is true.

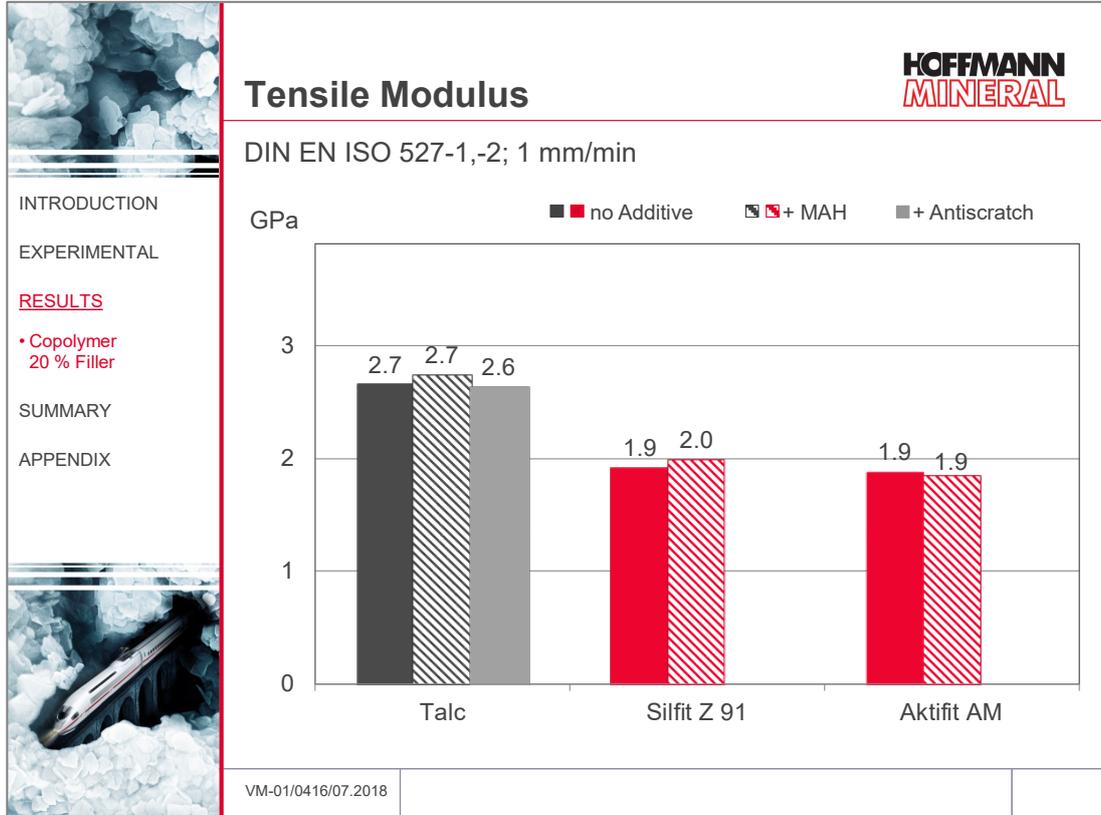


Also at low temperature the Neuburg Siliceous Earth grades yield markedly higher impact strength compared with the talc. The by far best result is obtained with Aktifit AM in combination with MAH.

3.3 Tensile test

Tensile modulus

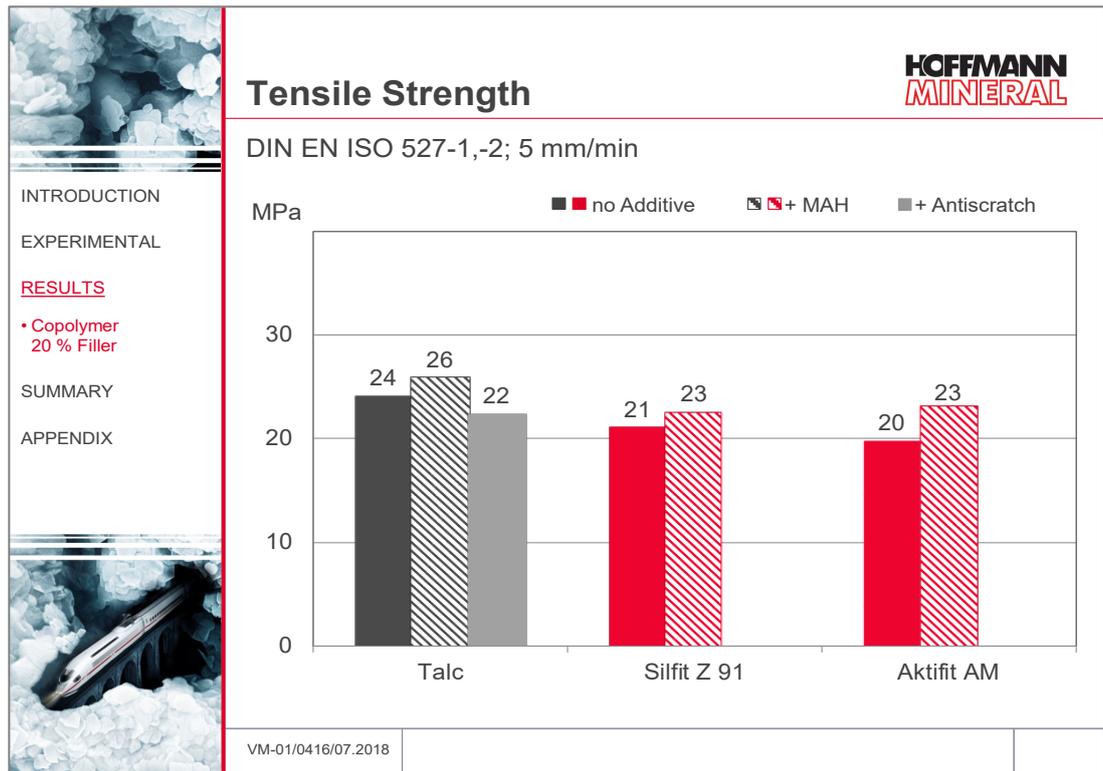
In place of the stiffness of the material, the tensile modulus was determined in a tensile test at an extension rate of 1 mm/min.



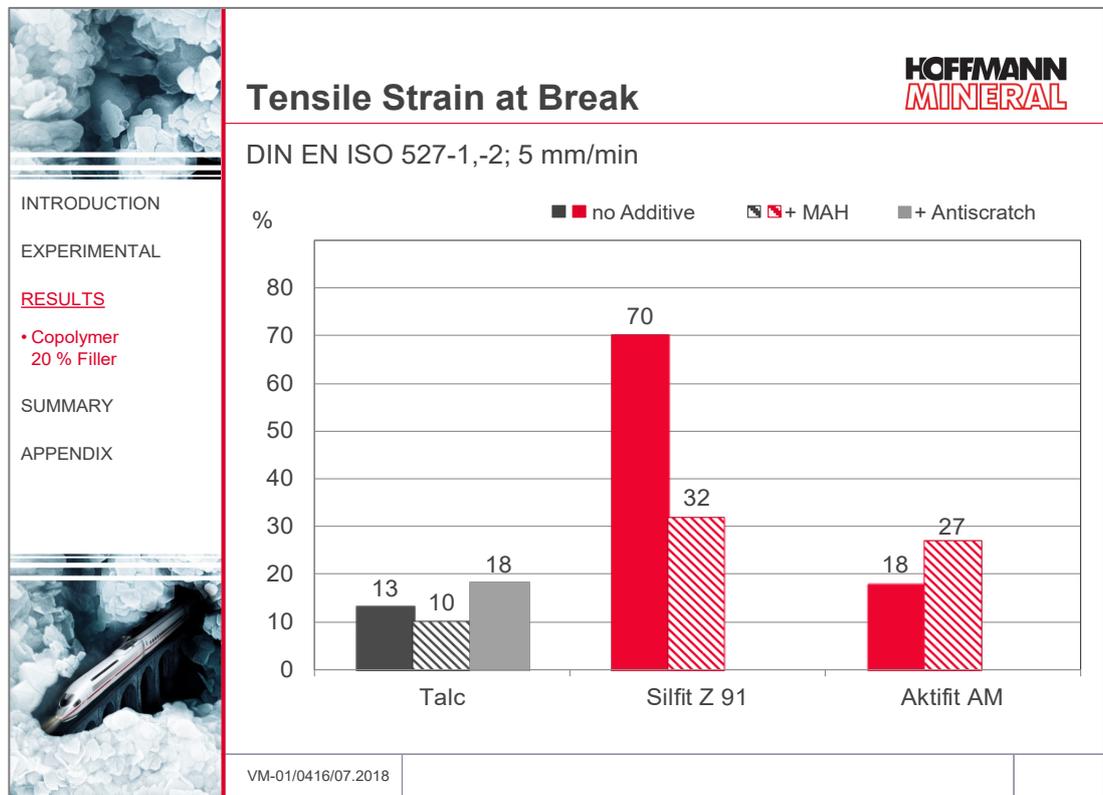
In view of the different morphological structure – corpuscular/lamellar Neuburg Siliceous Earth vs. straight lamellar talc – the Siliceous Earth grades come out with a lower stiffness than the talc. The use of additives has no significant influence on the stiffness results.

Tensile strength and tensile strain at break

The test was run with specimens type 1A at an extension rate of 5 mm/min up to break.



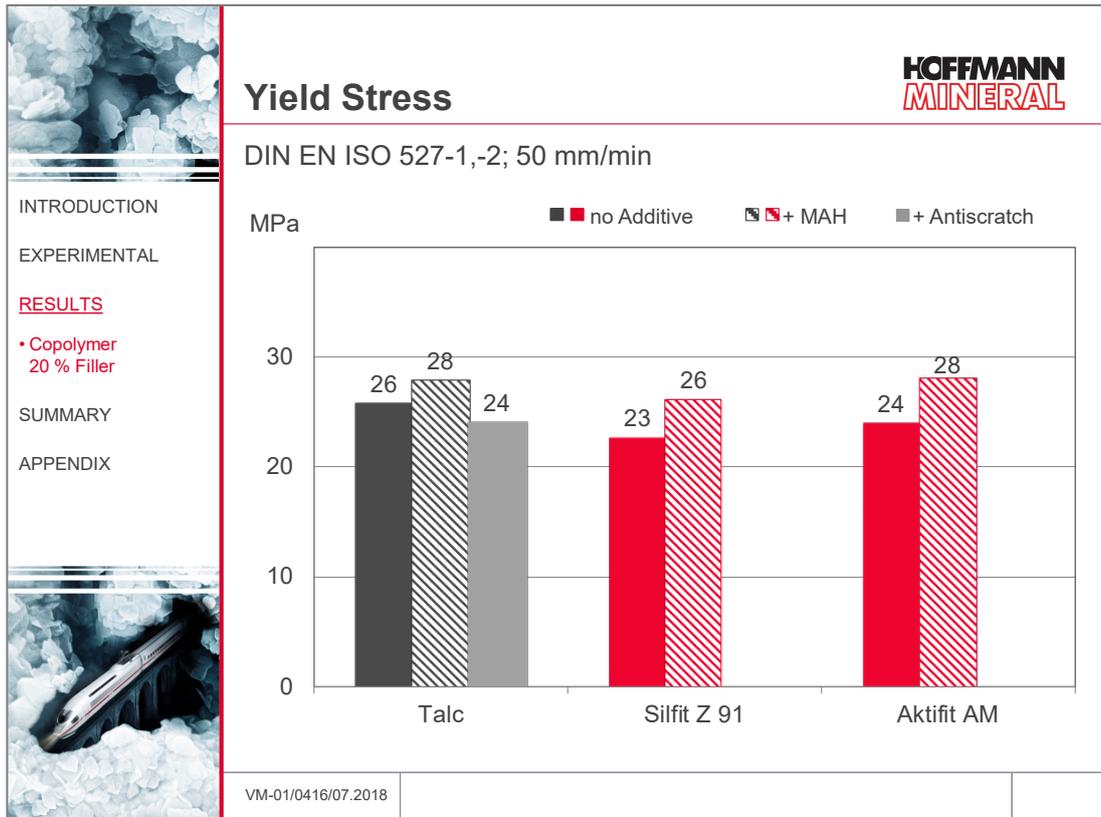
Looking at the compounds without additives, the talc shows a tendency towards higher strength. The addition of MAH increases the strength level with all fillers tested.



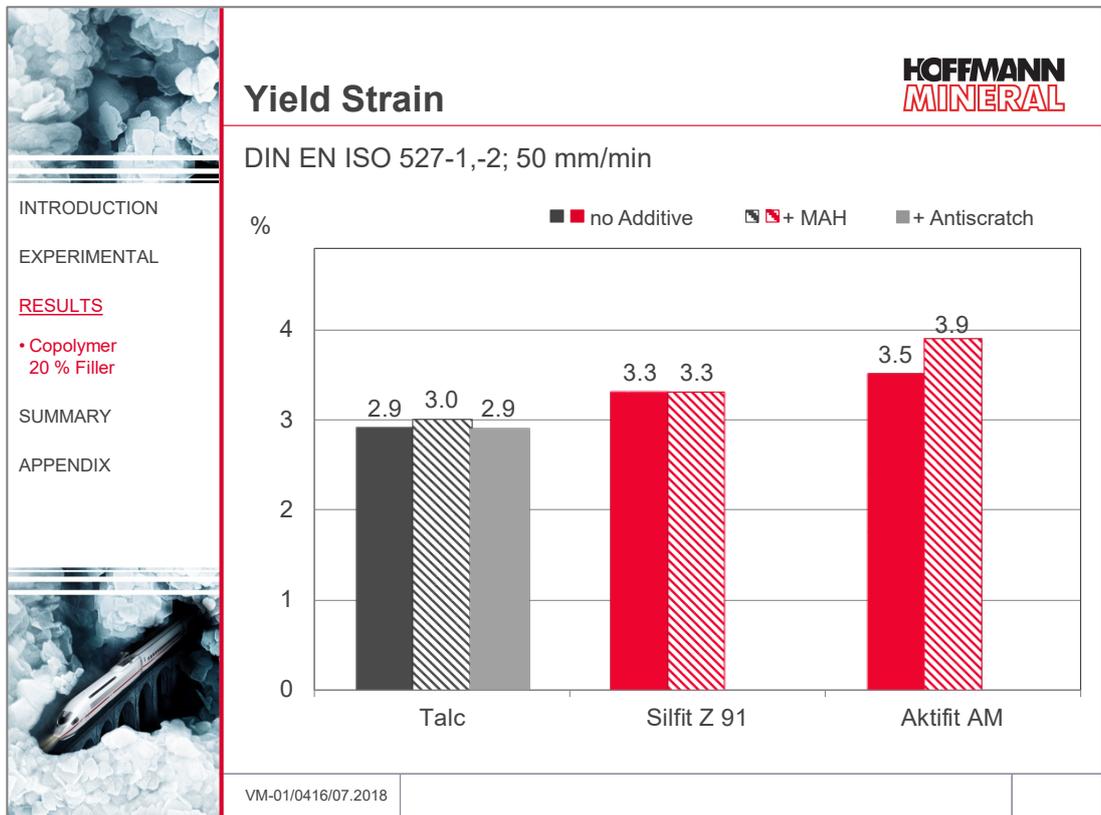
Strain at break comes out with the Neuburg Siliceous Earth grades markedly higher than with talc. Especially high values were obtained with Silfit Z 91 – where also the combination with MAH despite fairly strong negative effects remains distinctly higher in comparison with the talc compounds. By contrast, Aktifit AM only offers a slightly increased strain at break, which however by adding MAH can be raised above the level of talc plus anti-scratch additive. In the talc compounds, the use of additives gives only a moderate effect on the strain at break; MAH acts predominantly decreasing, the anti-scratch additive moderately increasing.

Yield stress, yield strain an nominal strain at break

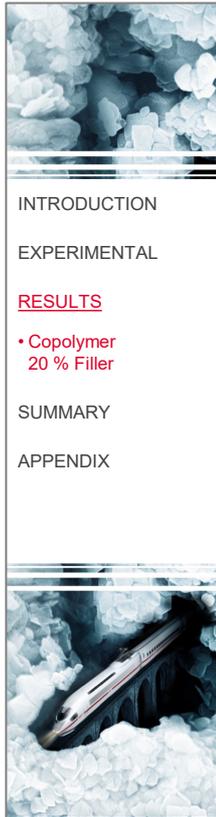
The test was also run with specimens type 1A, but at an extension rate of 50 mm/min up to break.



With the higher test speed, the yield stress shows a similar picture as the tensile strength. Talc shows a tendency towards higher values, and the addition of MAH with all fillers tested gives positive effects, in particular with Aktifit AM.



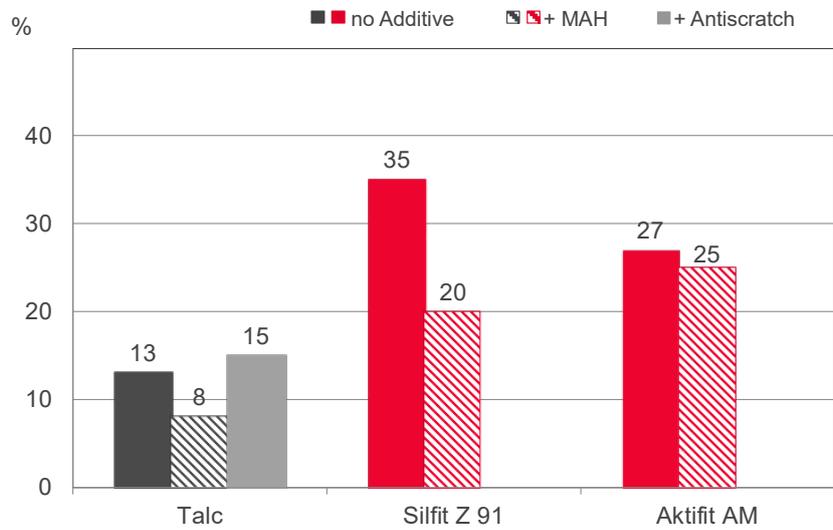
Yield strain with the Neuburg Siliceous Earth grades is higher than with talc. Especially in combination with MAH, Aktifit AM outperforms Silfit Z 91, and achieves with an increase of almost 1 % absolute compared to talc the highest level.



Nominal Strain at Break

**HOFFMANN
MINERAL**

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



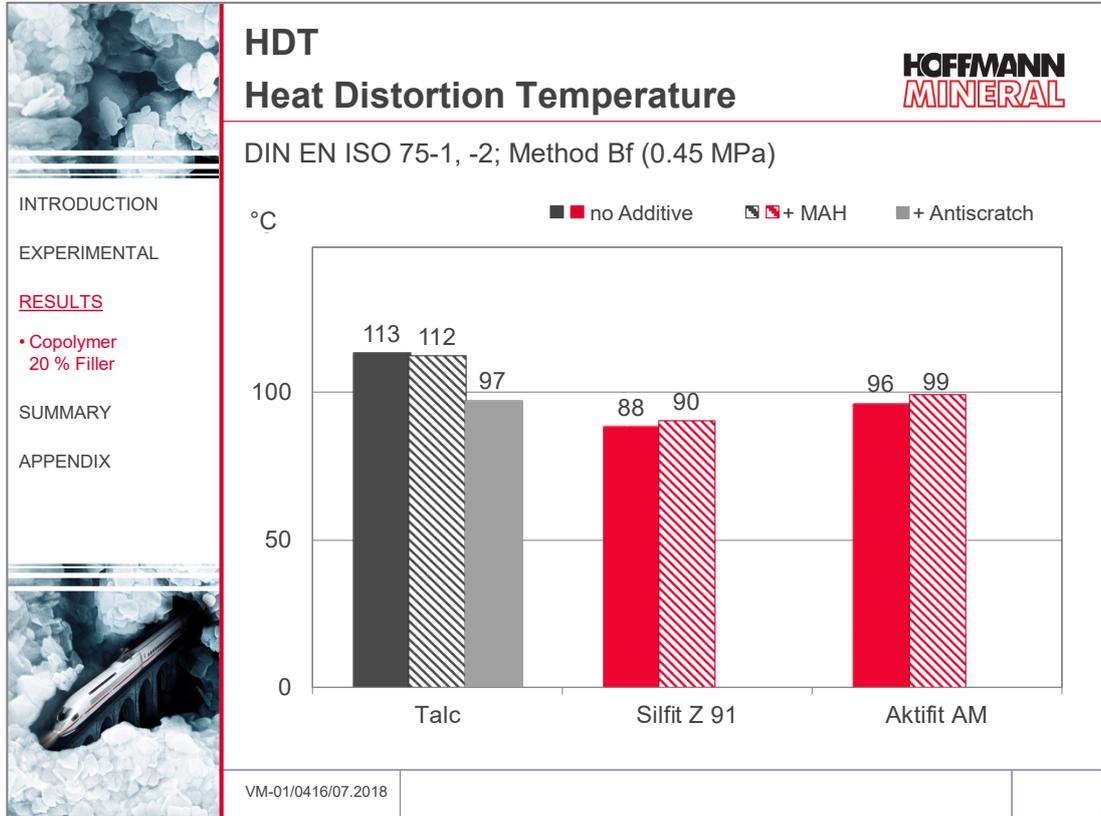
VM-01/0416/07.2018

Silfit Z 91 and Aktifit AM both arrive at a markedly higher nominal strain at break than the talc. The addition of MAH with all fillers gives rise to a decrease of varying degree. All the same the elongation of the Neuburg Siliceous Earth grades in combination with MAH comes out markedly higher compared with the talc variants. Aktifit AM again offers the very best result because the addition of MAH here hardly brings a negative effect.

3.4 Heat deflection

HDT

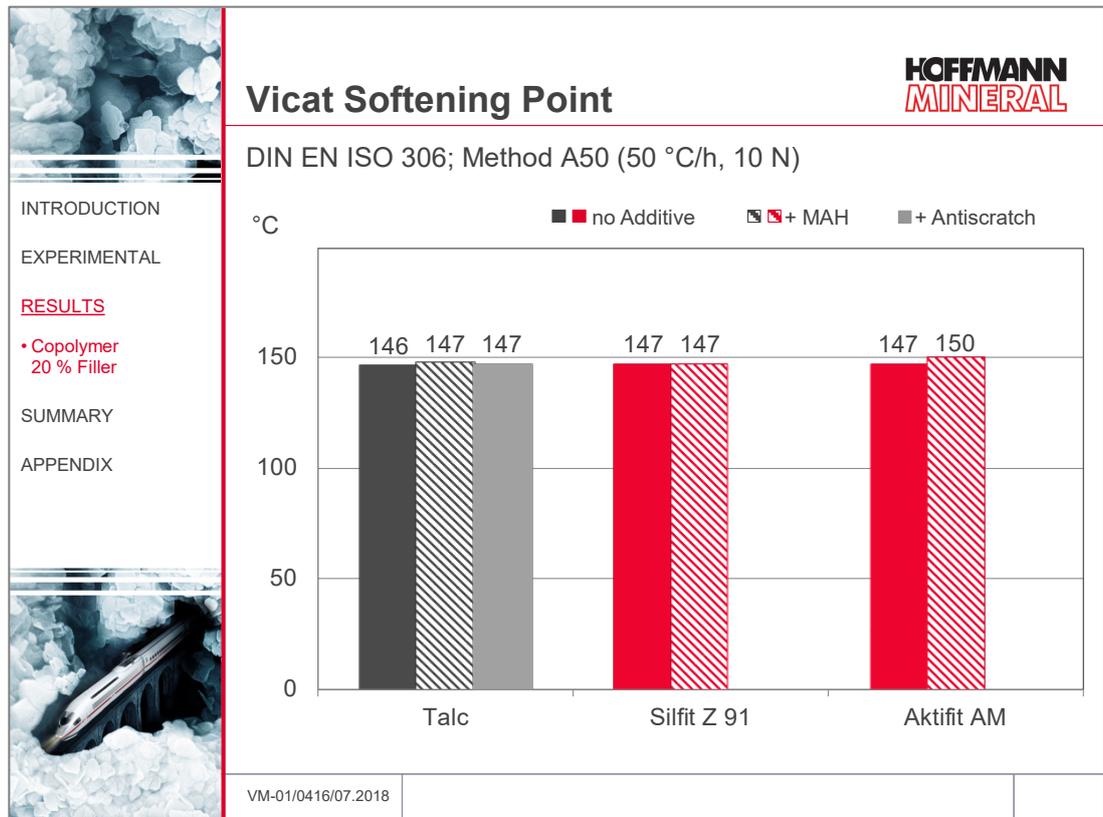
For the determination of the Heat Distortion Temperature (HDT), the sample was bent according to the 3-Point bending principle, charged with a constant load and heated with a rate of 120 K/h. The required load as a function of the sample thickness is calculated in order to arrive at an outer fiber stress of 0.45 MPa (Method B). The HDT then is the temperature that leads to a defined standard bending corresponding to 0.2 % outer fiber strain.



The heat deflection temperature with the Neuburg Siliceous Earth grades is somewhat lower than with the talc. The reason is probably the different E-modulus of the compounds which is caused by the different morphology of the fillers. All the same Aktifit AM arrives at a HDT comparable with talc in combination with the anti-scratch additive, which can even still slightly be increased by adding MAH.

Vicat softening point

The Vicat method measures the temperature at which a defined steel marker under a determined weight load and regular temperature increase penetrates 1 mm into the sample. The test followed the submethod A50, i.e. with a heating rate of 50 K/h and a load of 10 N.



If the dimensional stability under heat is determined according to the Vicat method, which is a different test method for the same property, a significant difference between the tested fillers can no longer be observed. Now Aktifit AM with MAH reaches even the highest level.

3.5 Indentation by writing and scratch resistance

The test method with the pertinent parameters is based on the Volkswagen test method PV 3074 for the writing resistance, and PV 3952 for the scratch resistance.

The test samples were platelets with two different surface structures. The coarser grain size is designed as K 09, the finer as K 31.

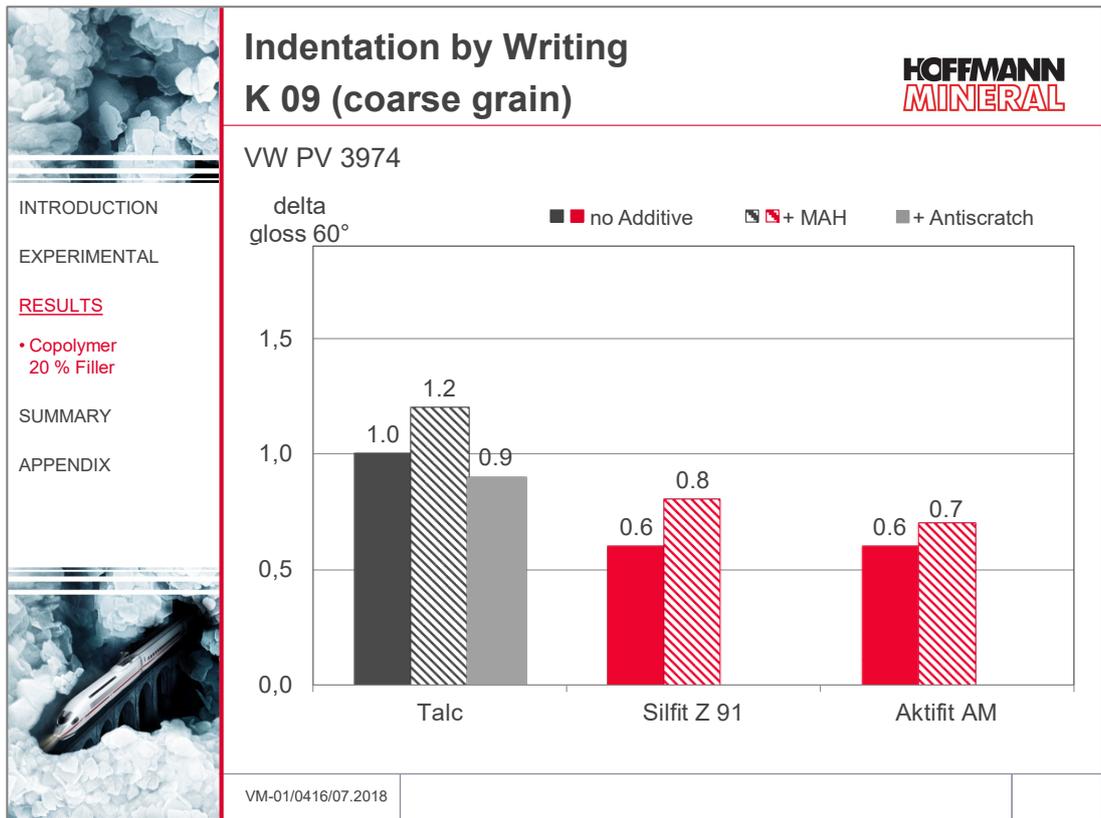
For the determination of the writing resistance, a rounded hard metal plate, adjusted vertically to the pull direction, is pulled over the sample surface with a weight load of 10 N. During the time of testing, 80 inscriptions are applied each in longitudinal and transverse direction (speed 1000 mm/min, distance between lines 0.5 mm).

The low distance between the inscriptions gives rise to the impression of a “polished surface” – which seems to exhibit more gloss than prior to the inscriptions. The test measures the difference of the gloss before and after the inscriptions using the 60° geometry.

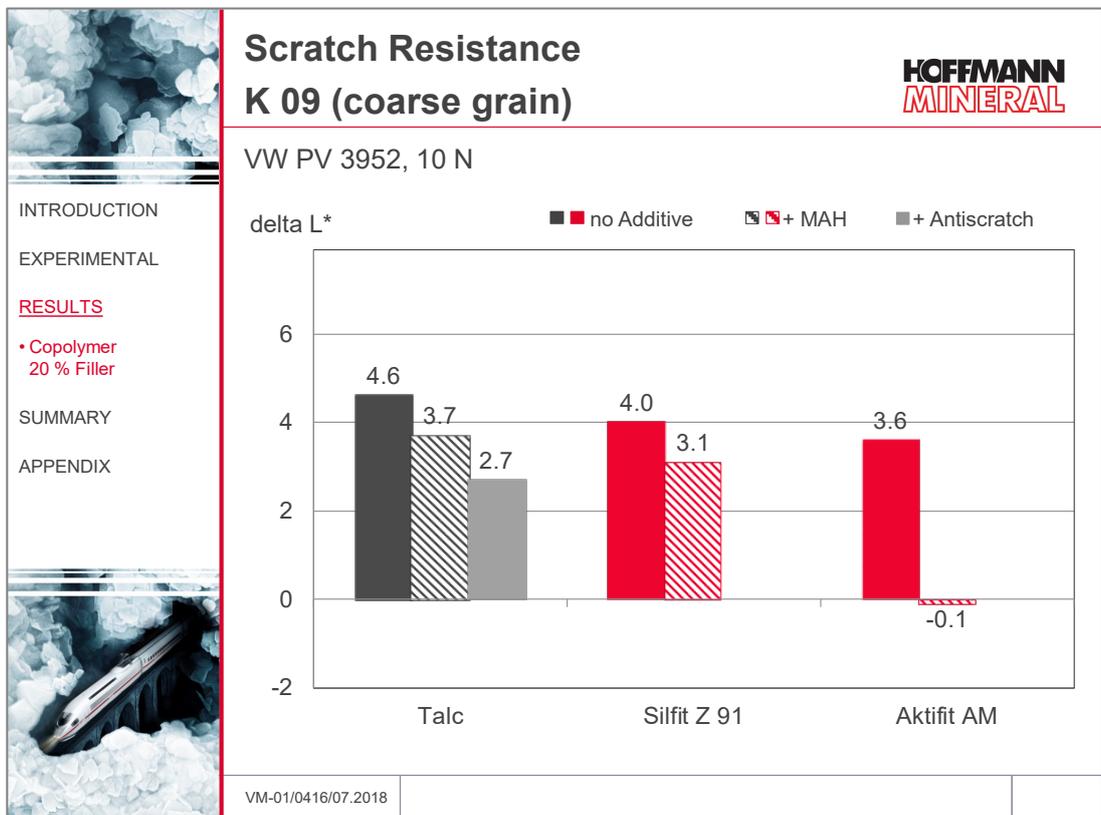
For the scratch resistance, a scratch marker with a ball point of 1 mm in diameter is led across the sample surface with a load of 10 N. In total, a grid-type surface pattern of 20 scratch marks in longitudinal and transverse direction is applied (speed 1000 mm/min, grid line distance 2 mm).

The resulting scratch marks appear brighter than the undamaged sample surface. The test measures the difference of the brightness L^* with a 45/0 measuring geometry prior to and after the scratching.

Coarse-grained surface (K 09)

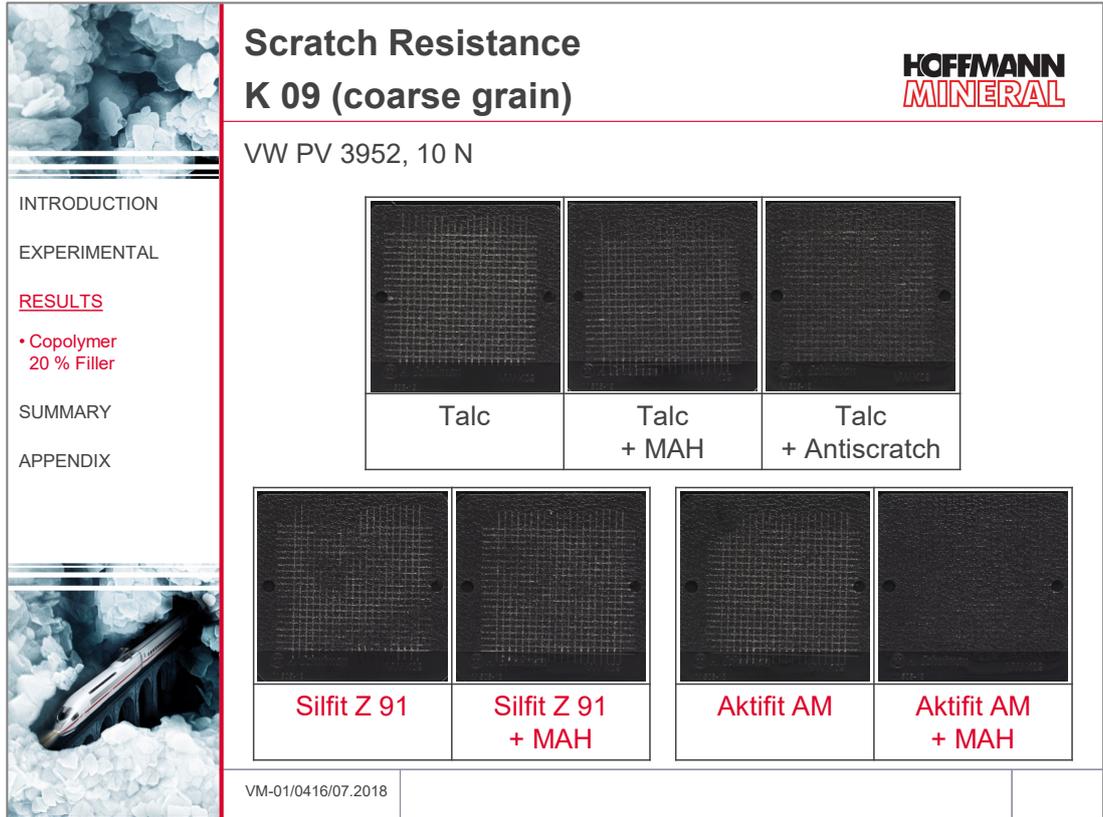


On surfaces with coarse grain size the writing resistance with Calcined Neuburg Siliceous Earth fillers is generally better than with talc. At the filler loading of 20 %, the addition of MAH does not cause any further improvement of the writing resistance.



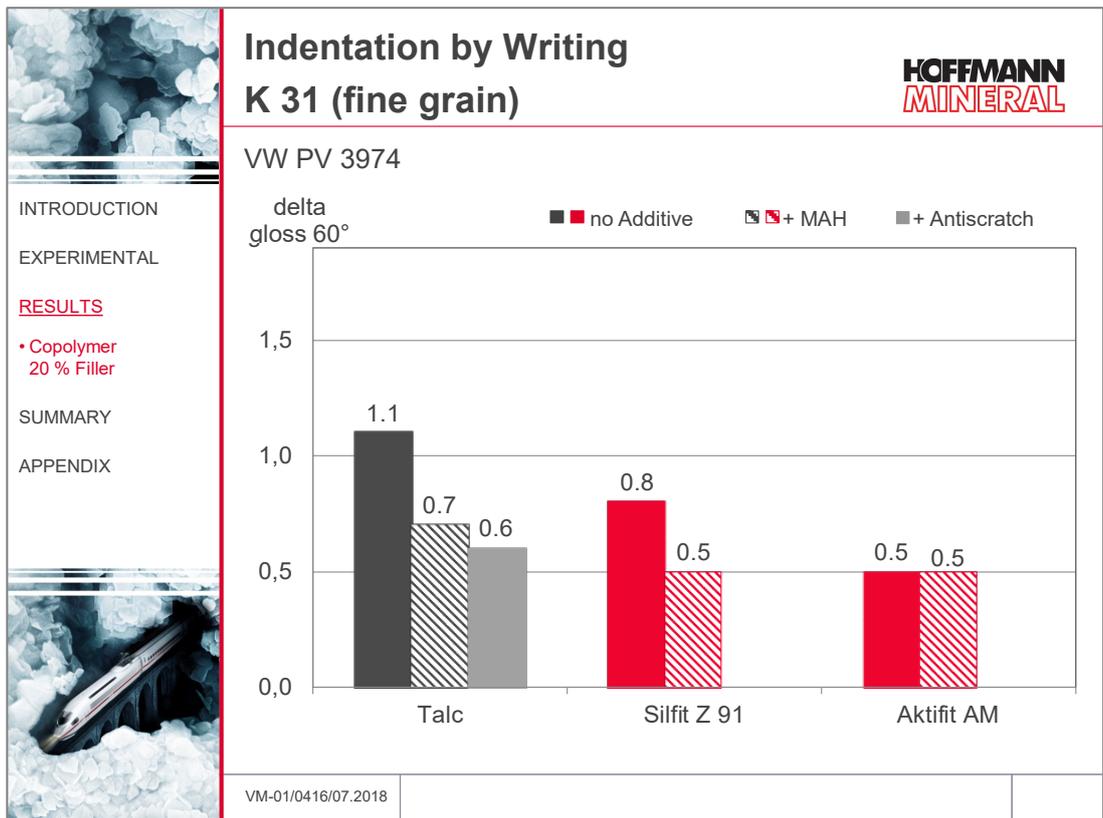
The suitable choice of the filler is already capable of influencing the scratch resistance. Although the talc grade used is praised for very good scratch resistance, the Neuburg Siliceous Earth grades even without additives offer somewhat better results. The addition of MAH results with all fillers in an improvement, but to a different extent. The combination of Silfit Z 91 with MAH already comes off somewhat better than the talc with the same additive.

Particularly noteworthy, however, is the combination of Aktifit AM plus MAH: here practically no brightening of the surface by scratch marks is any longer observed.

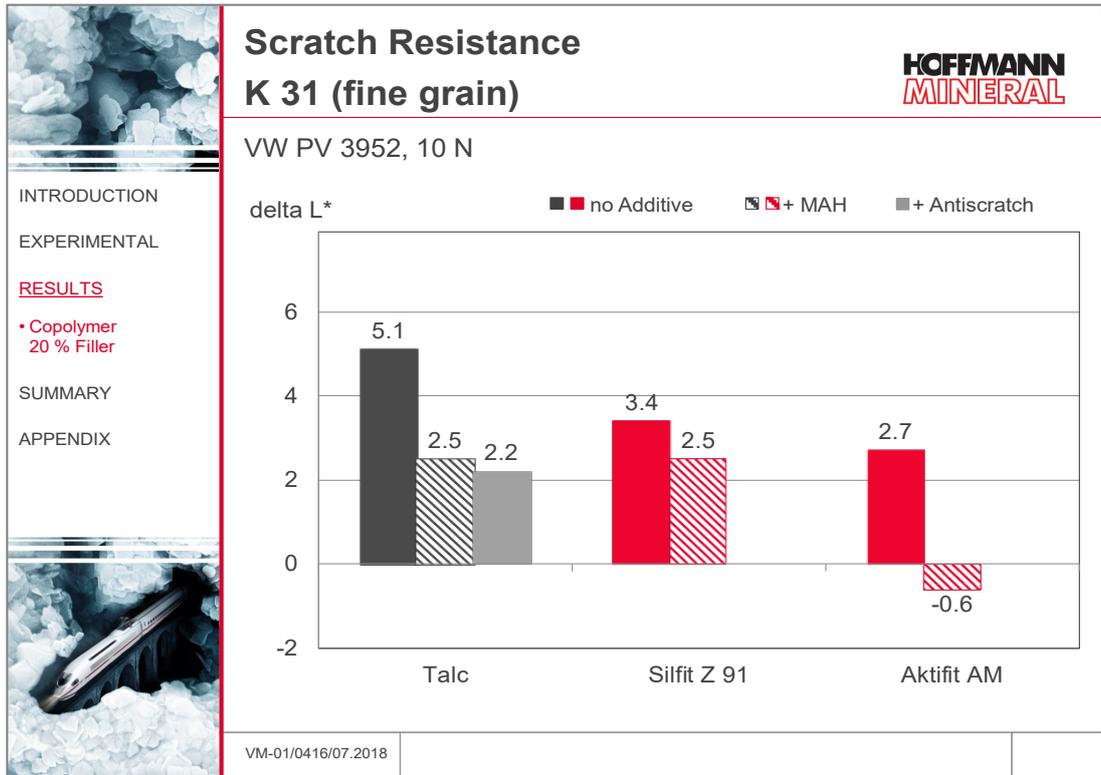


The images of the samples after scratching confirm the brightness differences measured. Aktifit AM with MAH practically does not show any brightening and by far gives the best result on the coarse grained surface.

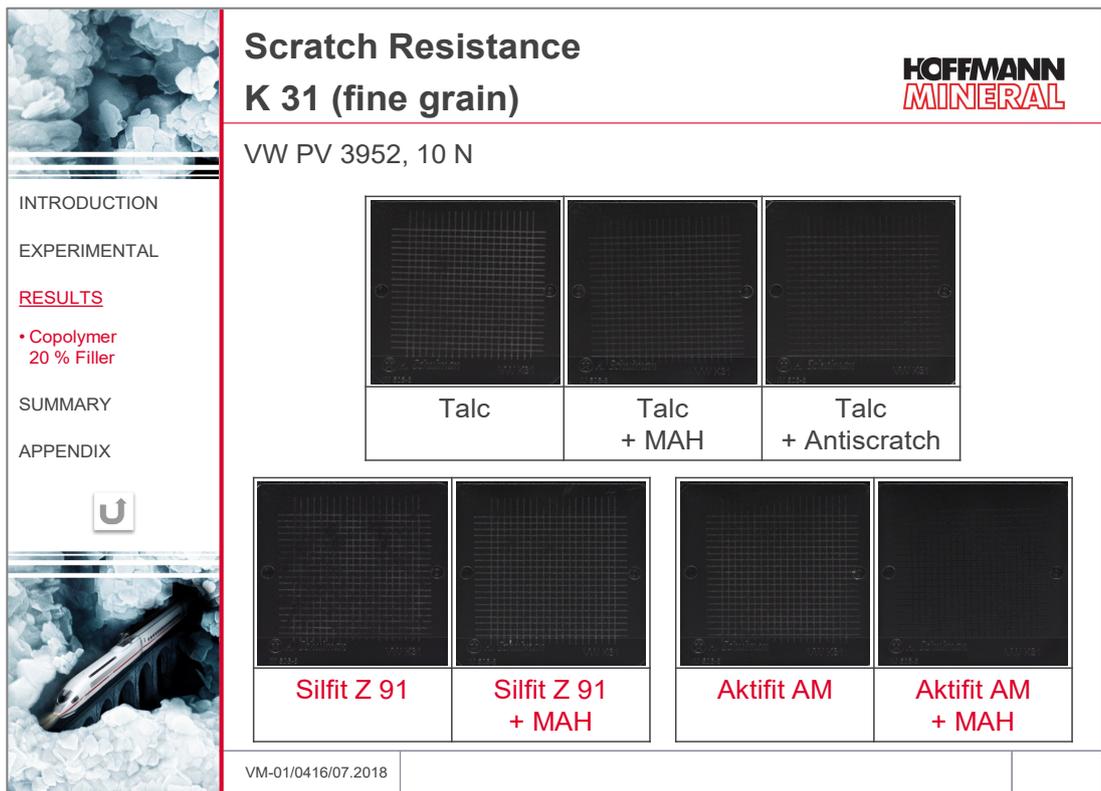
Fine-grained surface (K 31)



With fine grain size, the Calcined Neuburg Siliceous Earth grades already offer results which with talc can only be obtained with the addition of additives. The use of MAH with all fillers leads to a further improvement of the inscription resistance. Silfit Z 91 with MAH and Aktifit AM (with or without MAH) here lead to the best results.



Also in the case of finely grained sample sheets the surface with the Neuburg Siliceous Earth grades is better scratch resistant than with the talc. The addition of the anti-scratch agent or of MAH tends to further improve the scratch resistance, although to a different degree. Silfit Z 91 with MAH places itself close to the level of the talc with MAH resp. of Aktifit AM without additive. Here again it is the combination of Aktifit AM with MAH which offers an impressive result.



The images tend to confirm the test results. The combination of Aktifit AM with MAH gives rise to an almost uniform dark surface without eye-catching scratch marks.

4 Results PP Copolymer, 40 % filler content

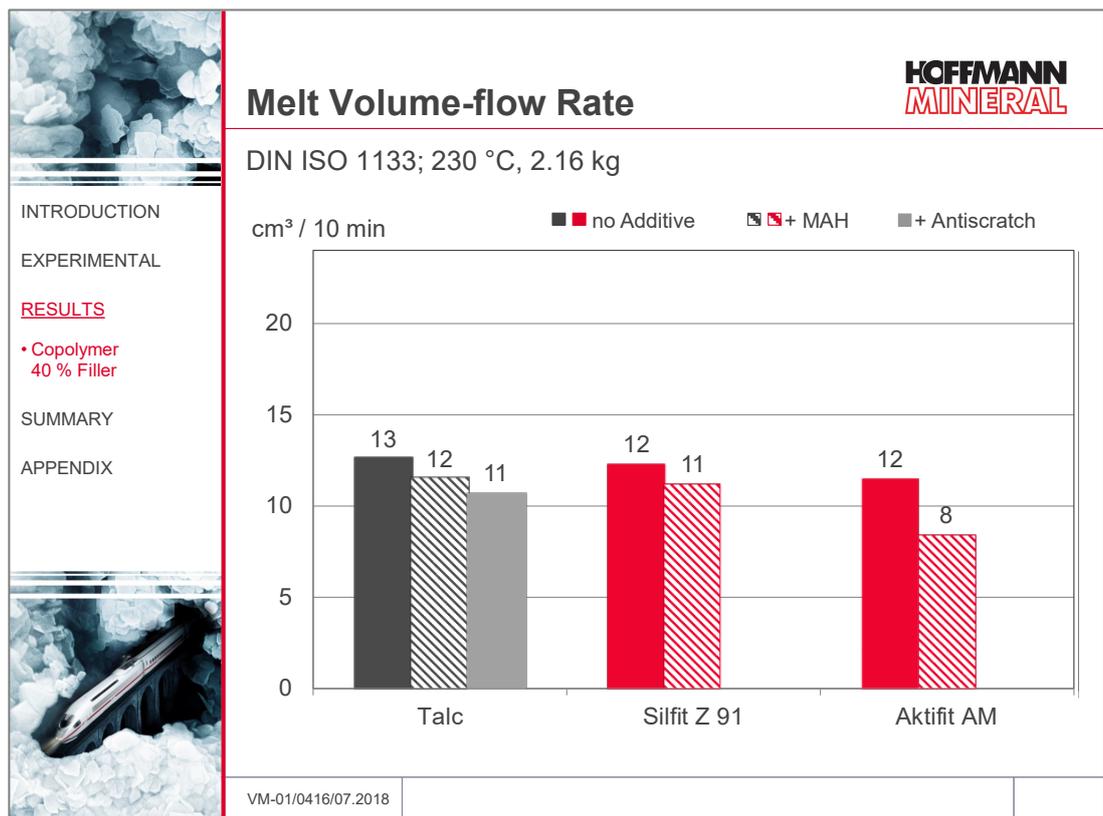
Formulations

	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

VM-01/0416/07.2018

The test program for this series was analogous to the preceding studies, with only a higher filler loading of 40 weight percent. The fillers used again were a talc as well as Silfit Z 91 and Aktifit AM. For the compounds with MAH modified PP and anti-scratch agent, the additive concentration was adjusted to the higher filler content. The tests executed were the same as in the lower loaded compound series.

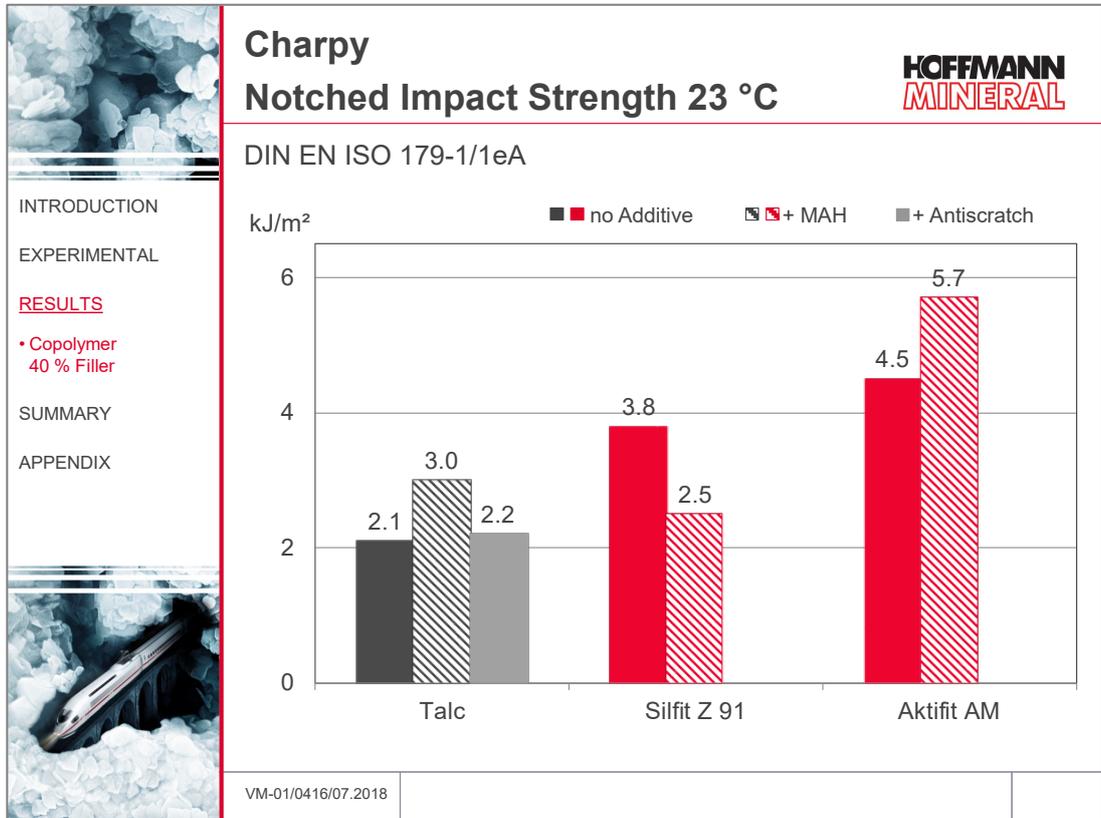
4.1 Melt volume-flow rate



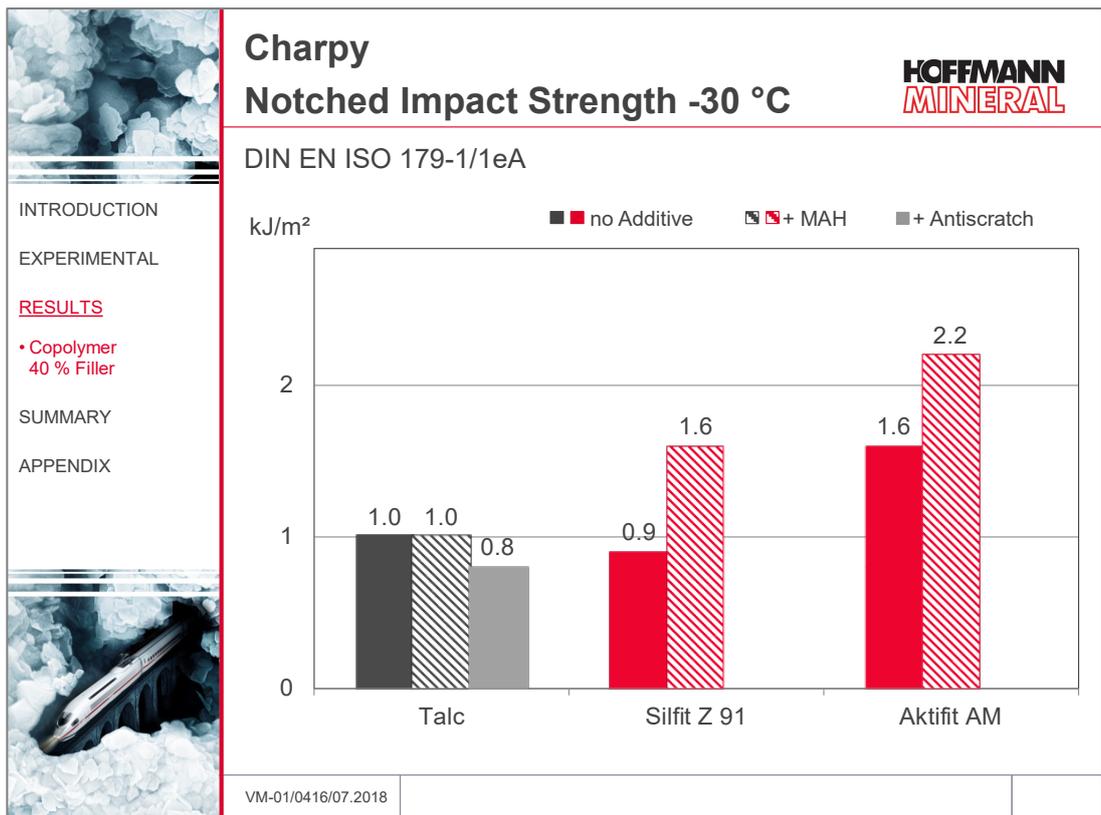
The flow behavior of the compounds tested was largely comparable. The addition of additives in all cases leads to a slight decrease of the flow rate. This effect comes out most clearly with the combination of Aktifit AM with MAH.

4.2 Impact strength Charpy

Notched impact strength



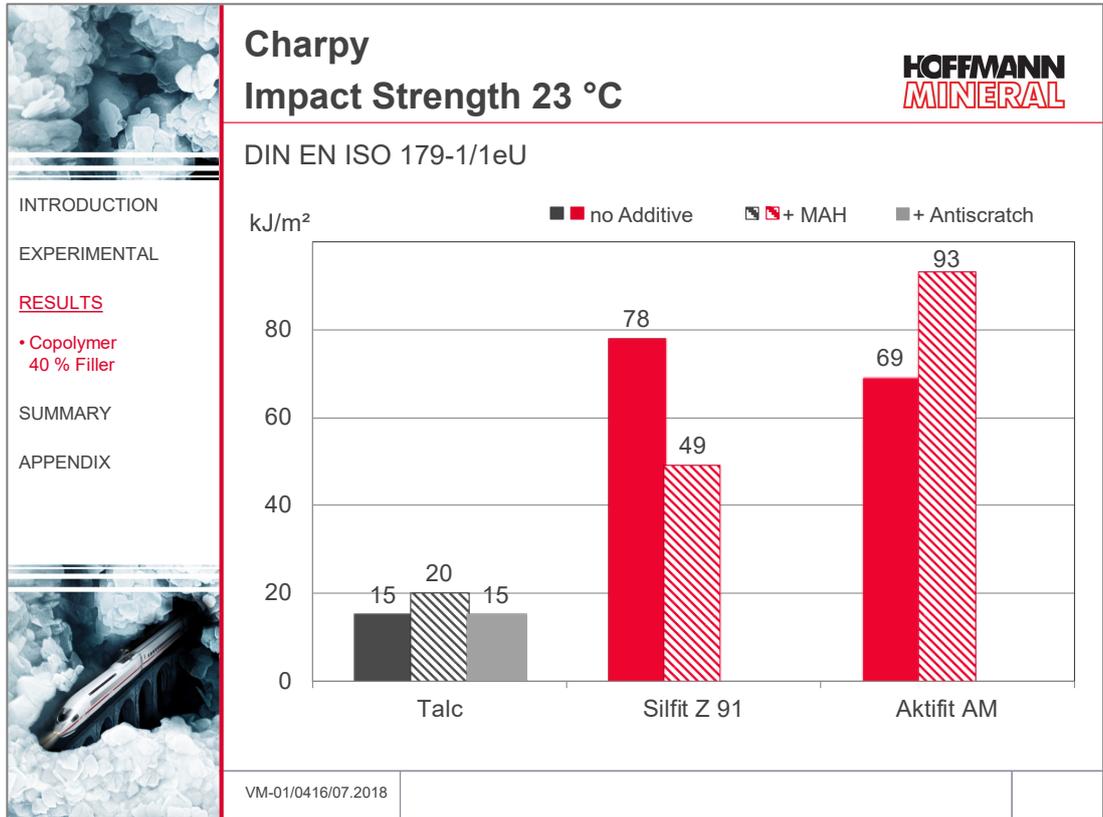
Silfit Z 91 gives a better notched impact strength than the talc, in combination with MAH fairly at level. Already without additives, Aktifit AM reaches a markedly higher notched impact strength, and this can be further increased by the addition of MAH.



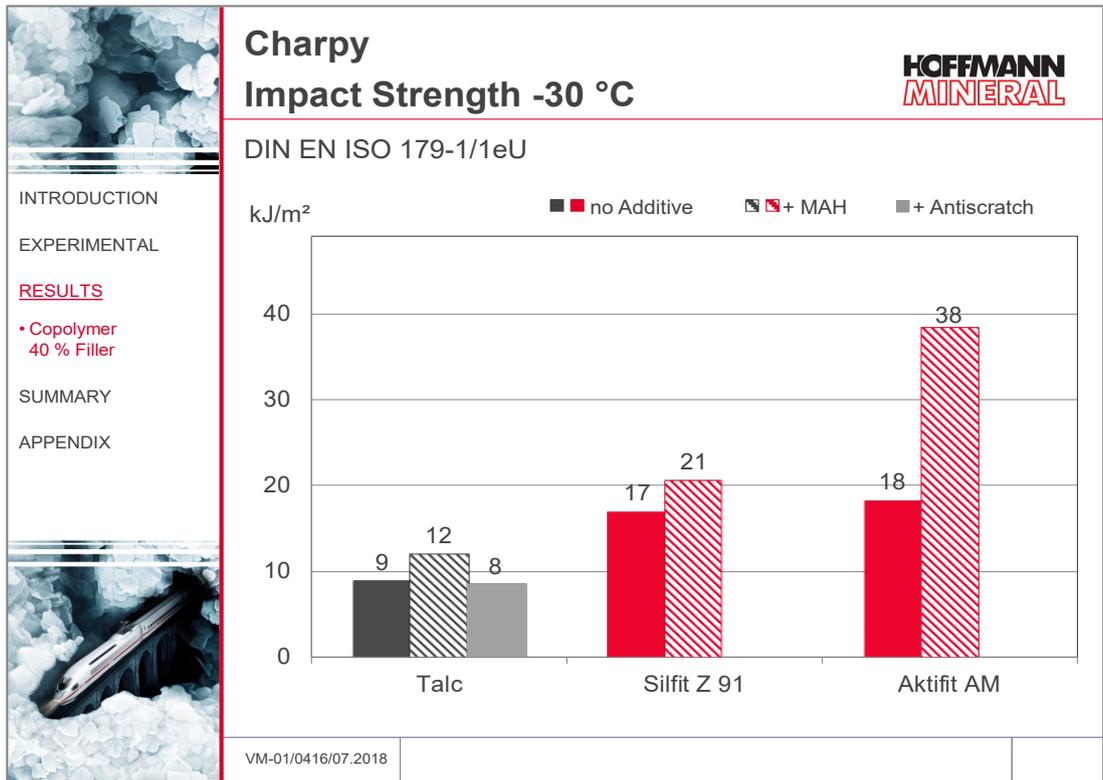
At low temperature, Silfit without additives is around the talc compounds, Aktifit AM already reaches a higher level.

With the help of the combination with MAH, the notched impact strength with the Calcined Neuburg Siliceous Earth grades can be further improved, while such an effect is not found with the talc, and the anti-scratch agent rather gives a negative result. Overall Aktifit AM with MAH offers the optimum level.

Unnotched impact strength



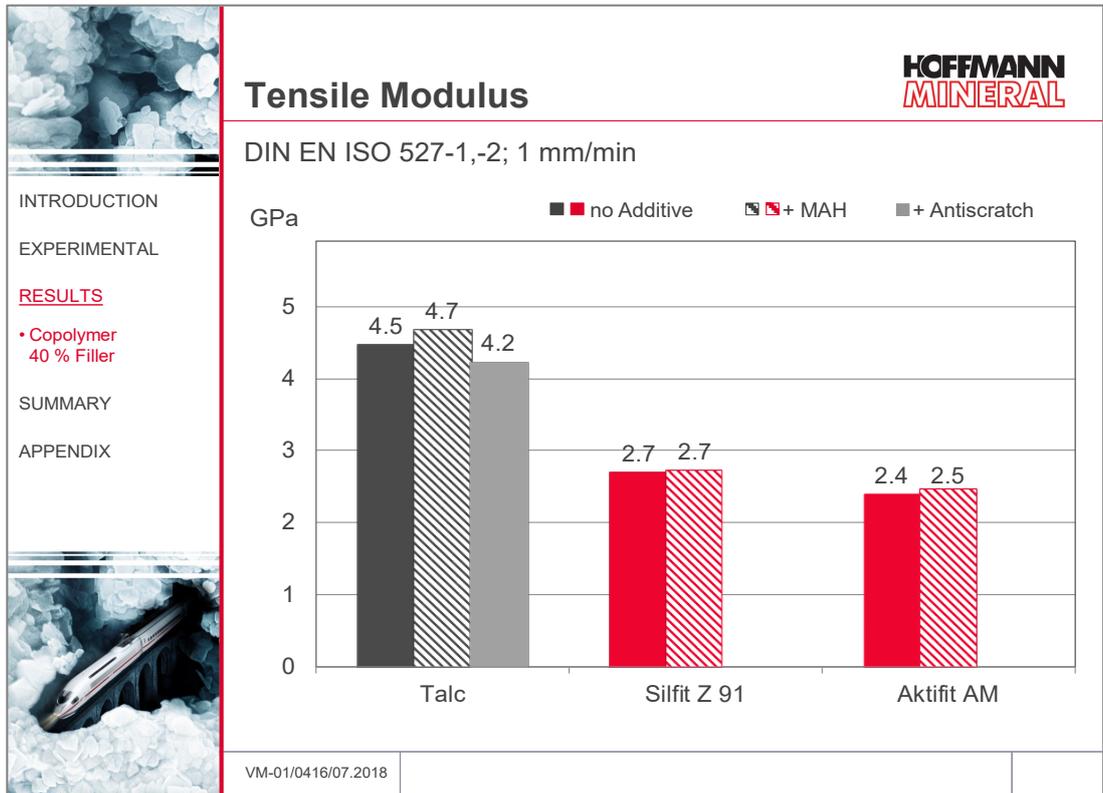
For the unnotched samples, the Neuburg Siliceous Earth grades even at the higher filler loading clearly outperform the talc grade that has been praised for high impact strength: four times the level can be reached. The best result here too is obtained with Aktifit AM in combination with MAH with an increase over talc to approximately 500 %.



Noteworthy, although not to the same degree, are the results at low temperature, where the Calcined Neuburg Siliceous Earth grades again lead to a marked increase of the impact strength and the combination of Aktifit AM with MAH by far offers the highest level. When looking at all impact results, with the filler loading of 40 % the benefits of Aktifit AM with MAH compared with the talc are still more evident.

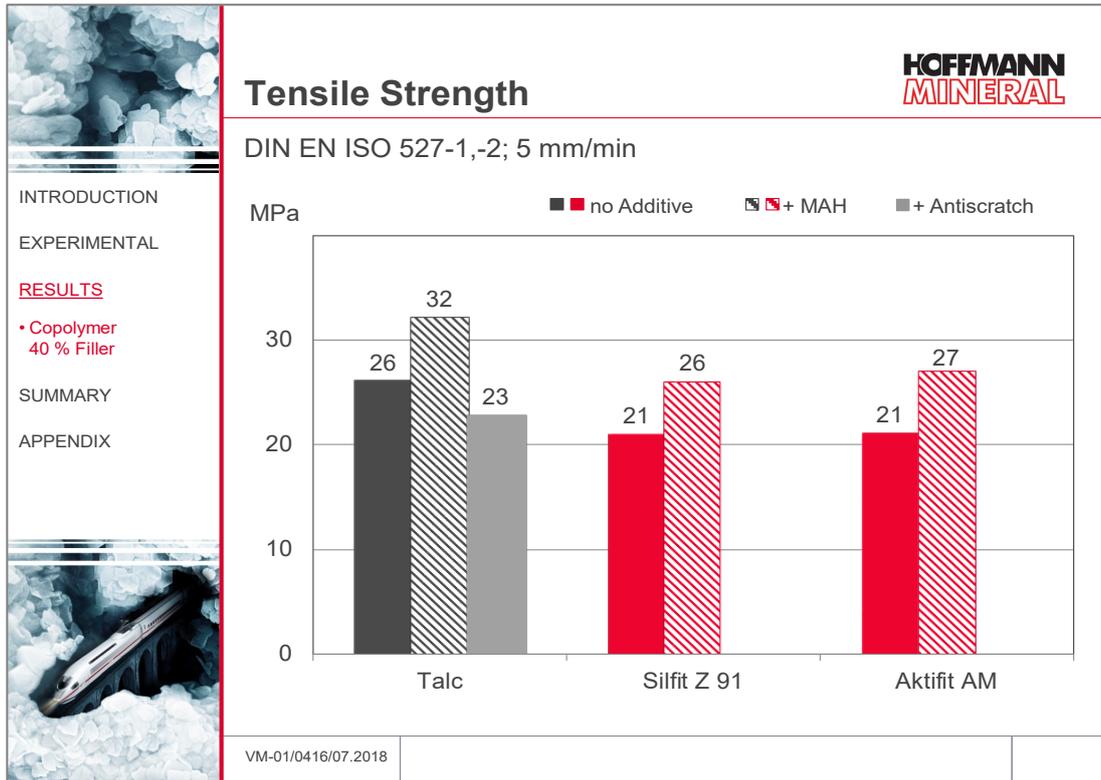
4.3 Tensile test

Tensile modulus

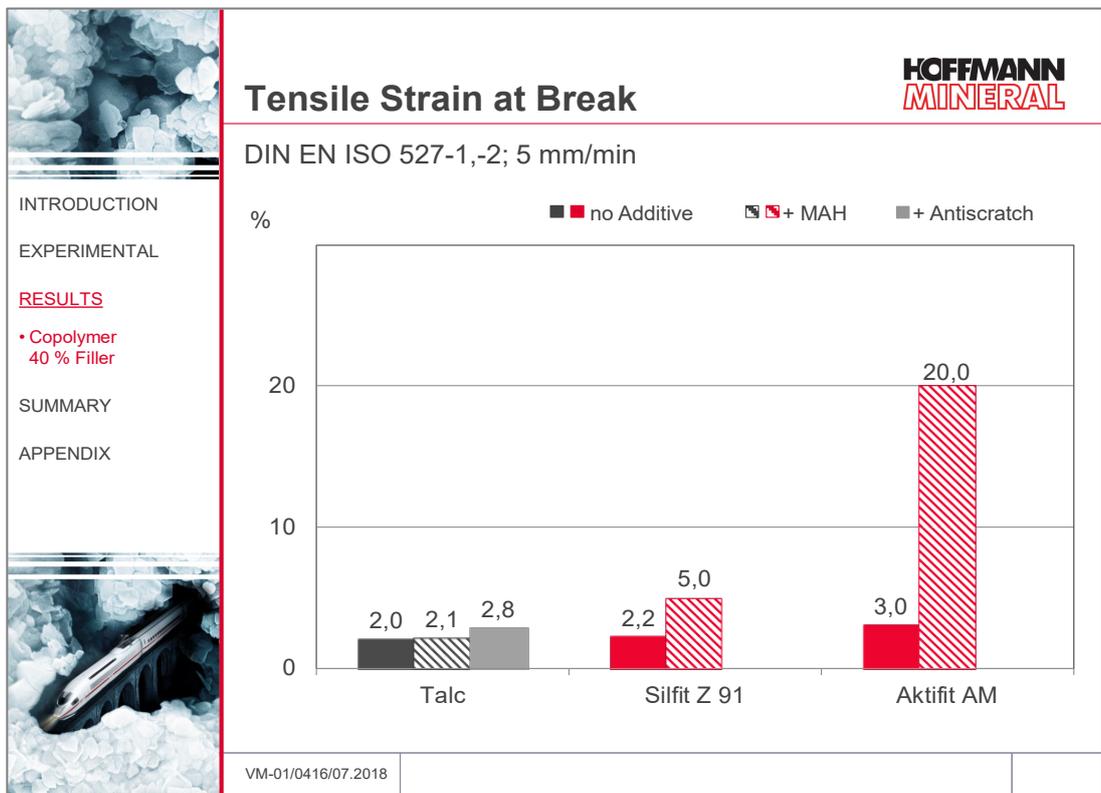


Because of their morphological structure, the stiffness with the Neuburg Siliceous Earth grades is lower than with the ultrafine platelet-shaped talc. This is particularly true with the high filler loading of 40 weight percent.

Tensile strength and tensile strain at break

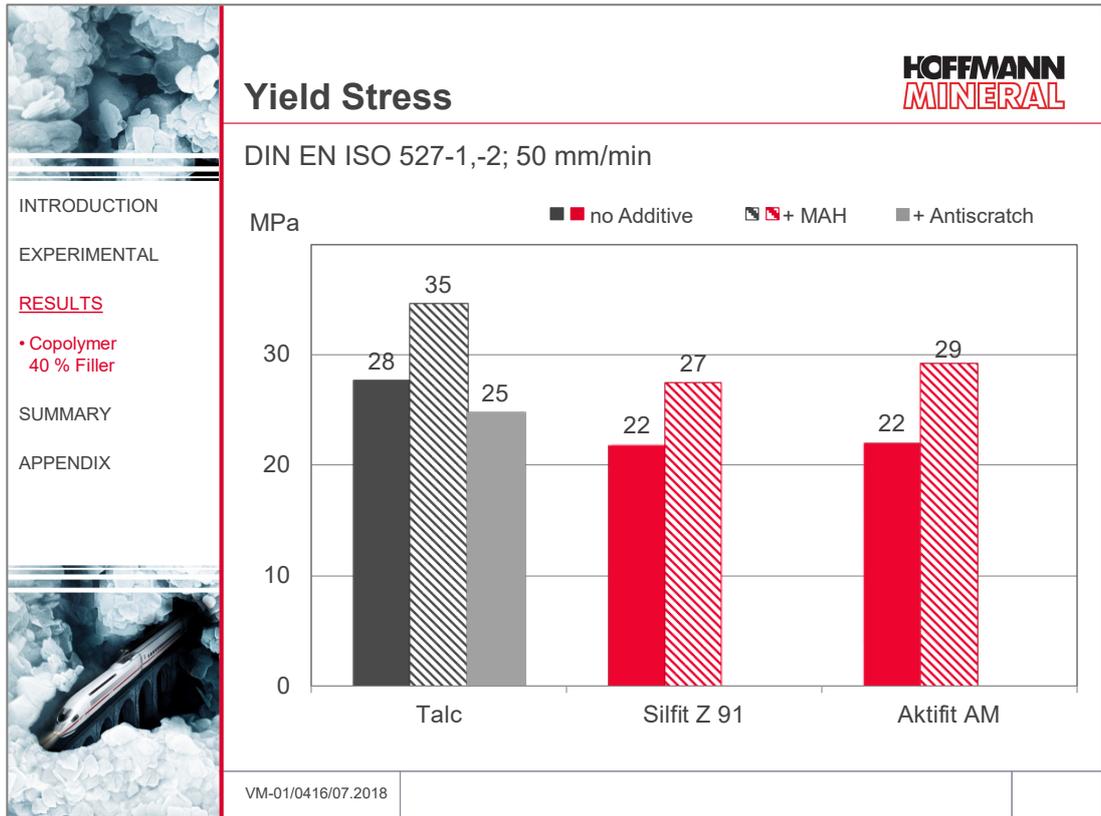


The talc helps to offer a somewhat higher tensile strength than the Neuburg Siliceous Earth grades. The addition of MAH with all fillers tends to increase the strength which then arrives at similar values as the talc without additives. Likewise, the result of the talc with the anti-scratch agent is exceeded with the MAH containing compounds loaded with Calcined Neuburg Siliceous Earth.

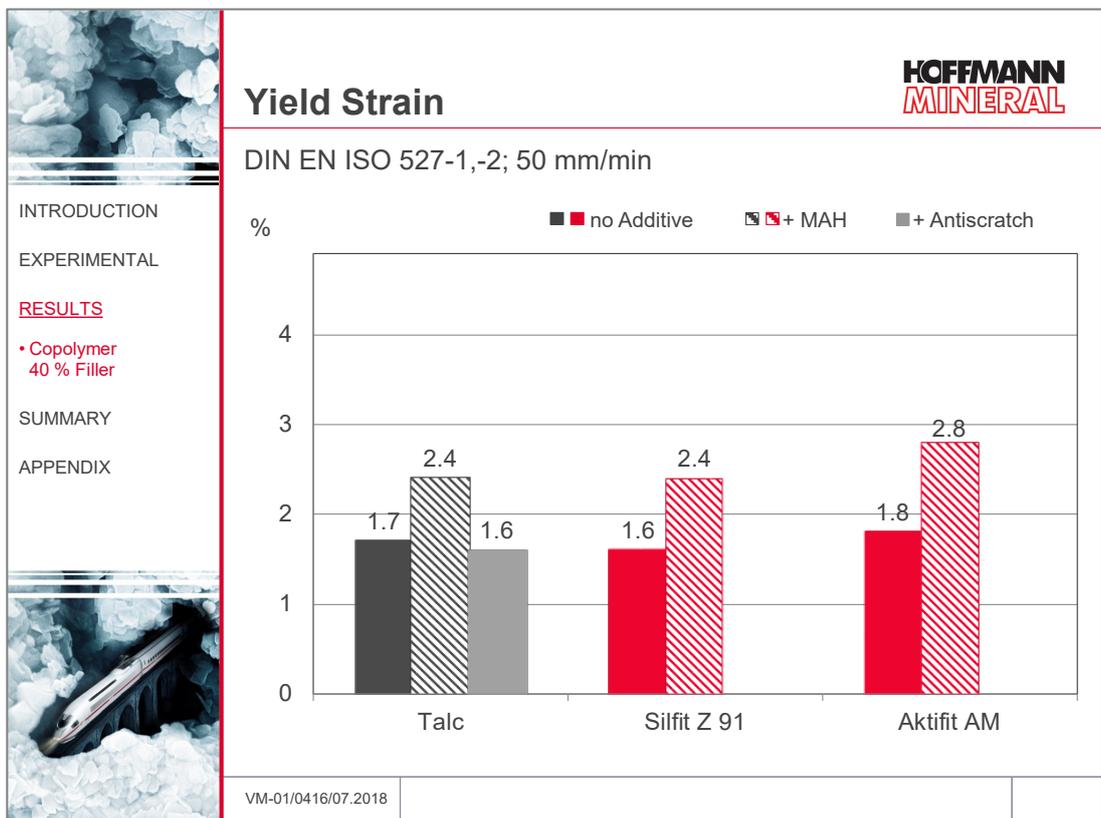


The strain at break with all fillers without additives comes out at around 2 to 3 %. Aktifit AM already here shows a tendency towards somewhat higher strain levels. While the addition of MAH to the talc does not produce in a significant effect, the elongation of the Calcined Neuburg Siliceous Earth compounds will be markedly increased. Of particular interest proves the combination of Aktifit AM with MAH which in the tests came off with 10 times the elongation level of the talc compounds.

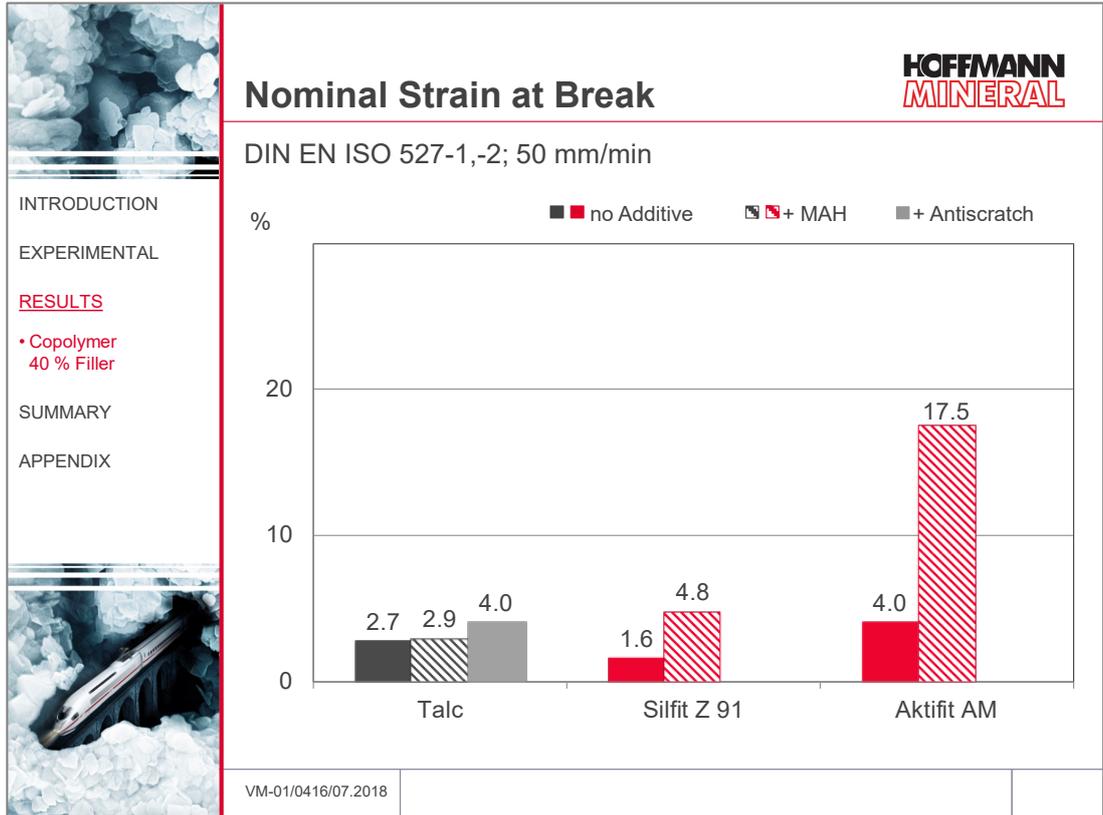
Yield stress, yield strain and nominal strain at break



The faster test rate causes a shift of the strength level to somewhat higher values. The basic conclusion, however, remains the same as the talc tends to offer higher results than the Neuburg Siliceous Earth grades, and the strength level can generally be improved by the addition of MAH.



Without the addition of additives the yield strain for all fillers tested is largely at a comparable level. By adding MAH, the yield strain increases with the talc and Silfit Z 91 about to the same degree; with Aktifit AM, the increase comes out somewhat more important.



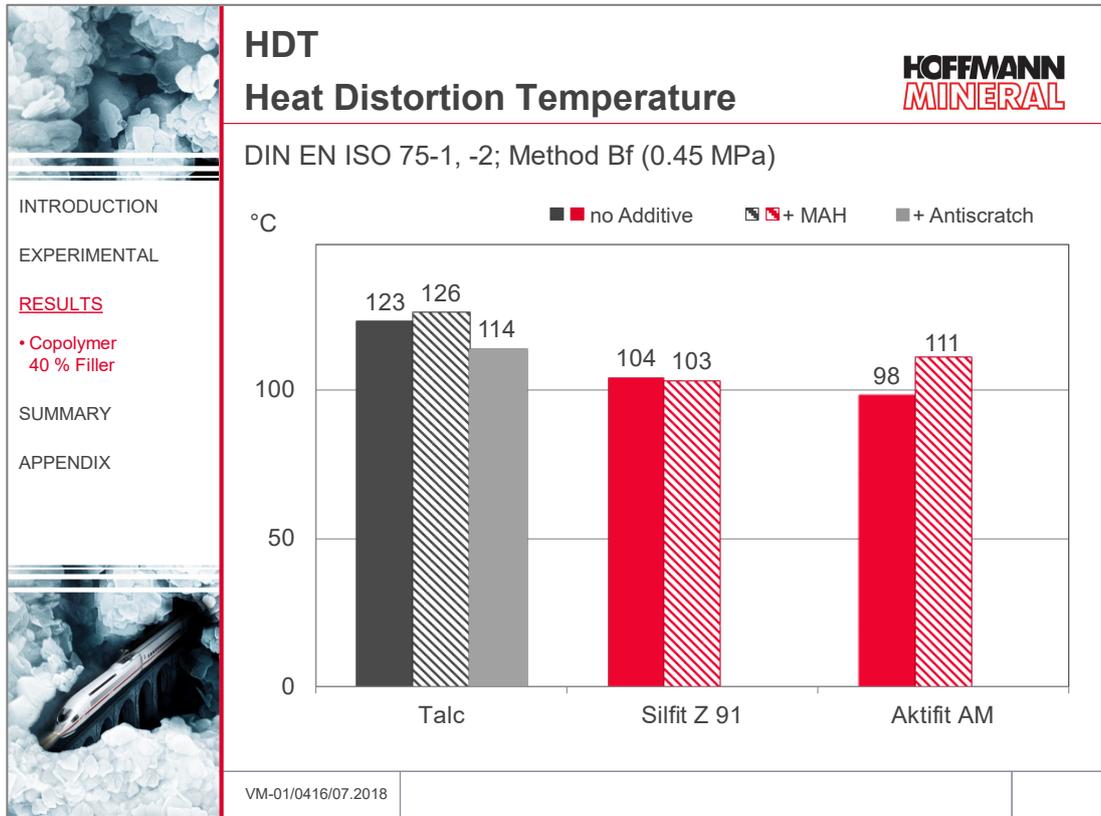
In comparison with the talc, the compounds filled with Silfit Z 91 undergo breakage already at somewhat smaller deformation. By contrast, the nominal strain at break with Aktifit AM already without additive is higher.

The use of MAH with talc does not produce a significant effect. With Silfit Z 91 the elongation will be markedly increased. The combination of Aktifit AM with MAH results in a remarkably higher nominal strain at break, and this despite the filler content of 40 weight percent.

4.4 Heat deflection

HDT

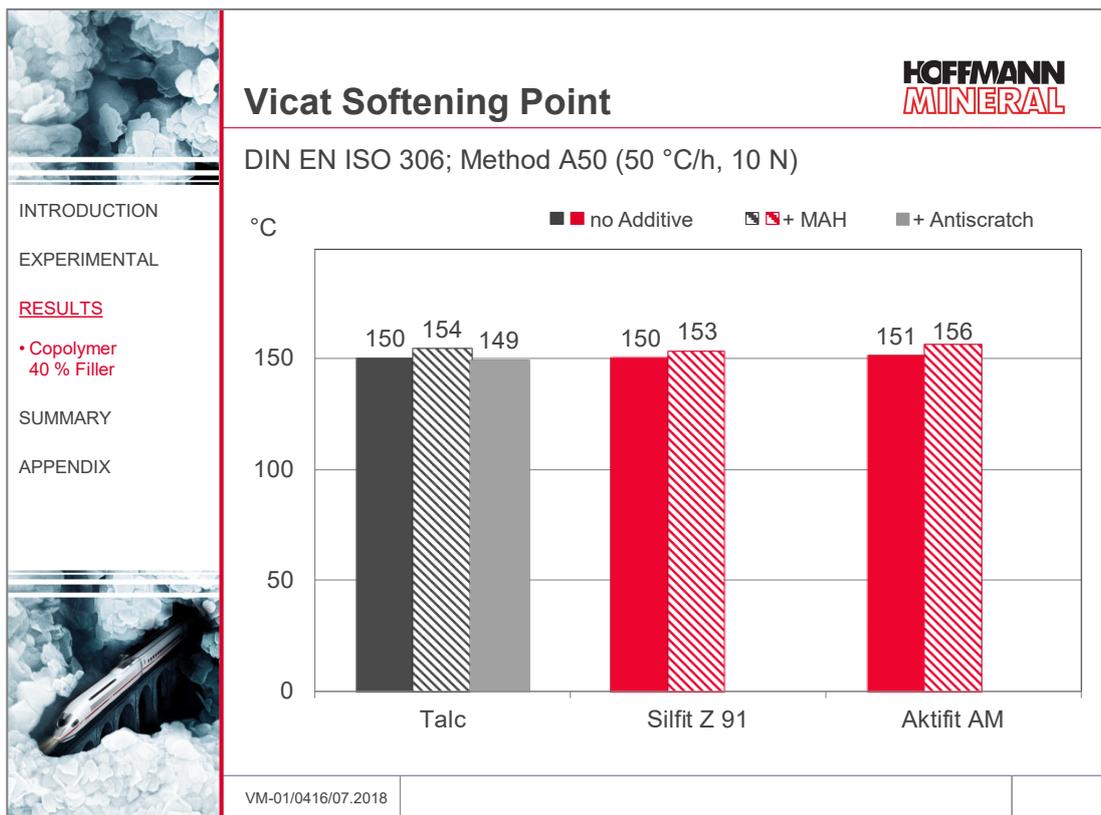
The heat deflection temperature was determined with the same parameters as for the filler loading of 20 %.



With the higher filler loading too, because of the different E-modulus, differences in the HDT can be observed.

Interestingly, the addition of MAH with Aktifit AM leads to an increased HDT.

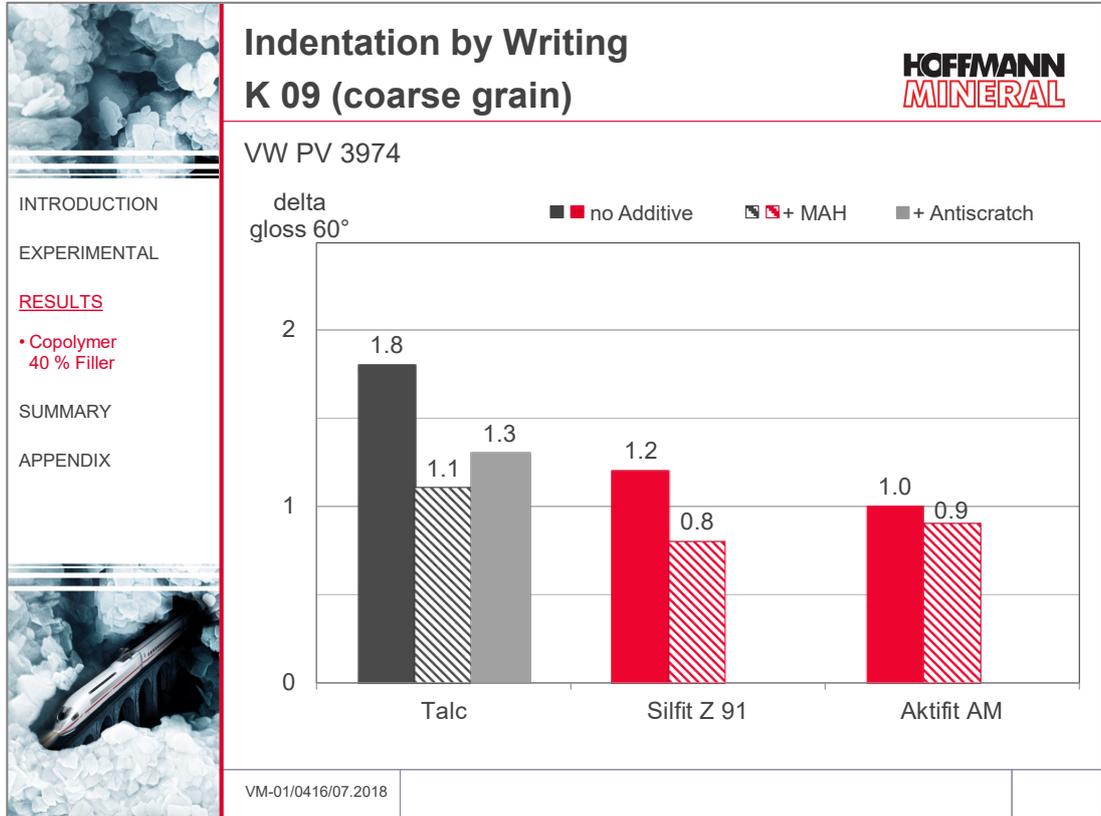
Vicat softening point



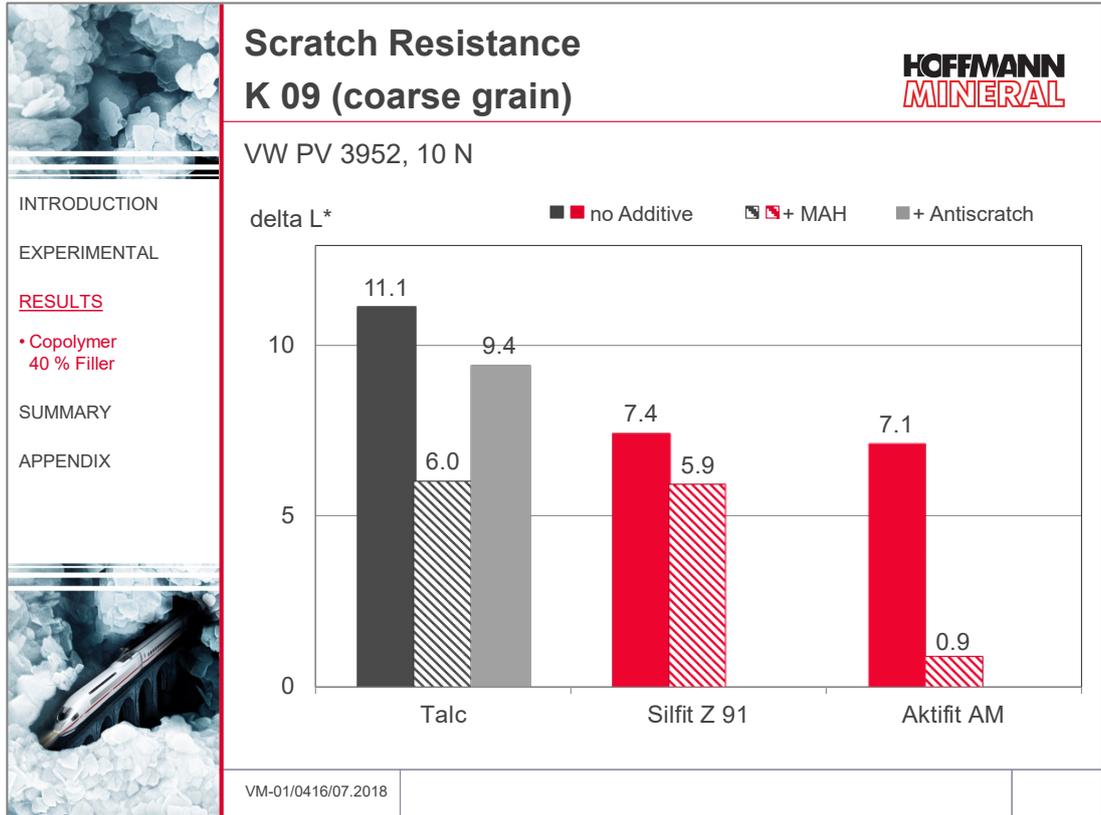
If the heat distortion temperature is determined according to the Vicat method, then no appreciable difference is detectable between the fillers used; Aktifit AM with MAH now even obtains the highest value.

4.5 Indentation by writing and scratch resistance

Coarse-grained surface (K 09)

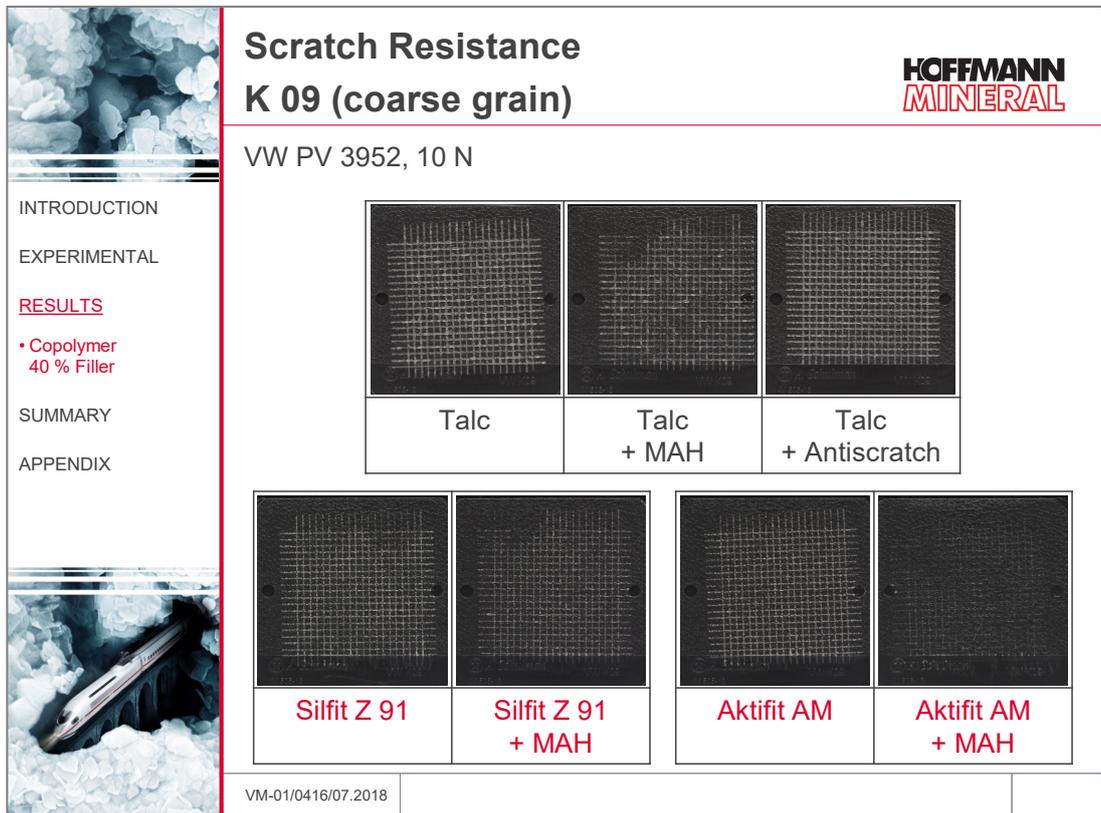


The resistance against writing with the Calcined Neuburg Siliceous Earth grades without any additives is already at least comparable with talc plus additives. The addition of MAH to the Siliceous Earth compounds helps to further improve this property.



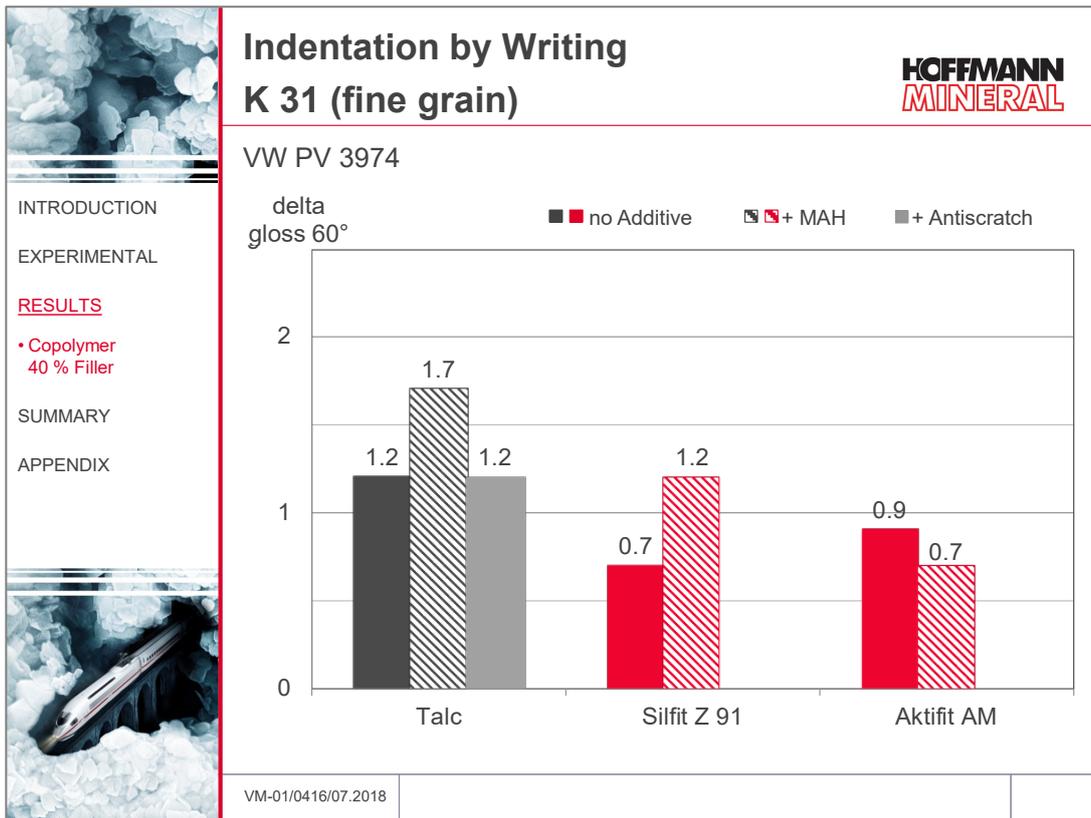
At the high filler loading of 40 % the difference in the scratch resistance of the fillers is markedly more evident. Silfit Z 91 and Aktifit AM already without MAH addition place themselves on a distinctly better level than the talc. Like with the lower filler loading, MAH addition gives rise to an improvement with all fillers tested to a varying degree.

Talc with MAH and Silfit Z 91 with MAH lastly show the same scratch picture. Again the combination of Aktifit AM with MAH gives an outstanding result: the brightening by scratches comes out extremely reduced. The (expensive) anti-scratch agent incidentally on the coarsely grained surface only gives evidence of a marginal effect at the high filler content.

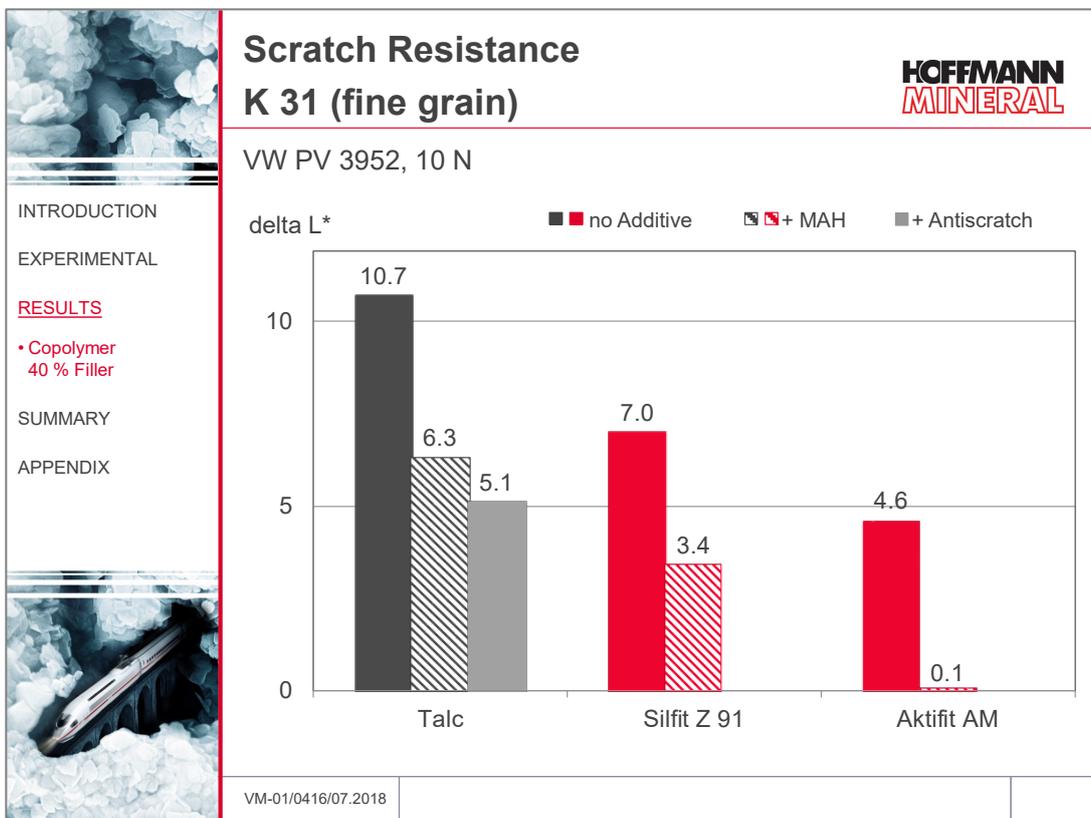


This is confirmed by the photographic images of the scratched sample sheets. Aktifit AM with MAH differs clearly from the other samples and offers by far the best result.

Fine-grained surface (K 31)



The resistance against writing with the Calcined Neuburg Siliceous Earth grades without additives is at least at level with the talc with additives. The addition of MAH to Aktifit AM reduces the difference in the degree of gloss prior to and after writing on fine-grained surfaces.



Already without additives a big difference can be observed between the fillers tested. The Neuburg Siliceous Earth grades give rise to markedly lower brightening through scratch marks compared with the competitive talc. The use of MAH helps to improve the scratch resistance with all three fillers to a similar degree. Already the combination of Silfit Z 91 with MAH gives rise to a better scratch resistance than the talc with MAH or with the (expensive) anti-scratch agent.

Again an outstanding result is obtained even at the fine-grained surface with the combination of Aktifit AM and MAH – the difference in the brightness of 0.1 units practically does not prove an effect brought about by the scratch marks.

Scratch Resistance

K 31 (fine grain)



VW PV 3952, 10 N

INTRODUCTION

EXPERIMENTAL

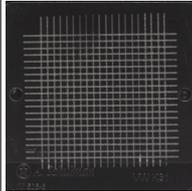
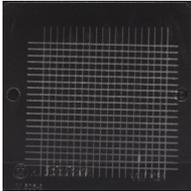
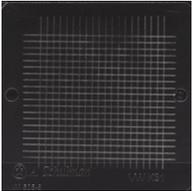
RESULTS

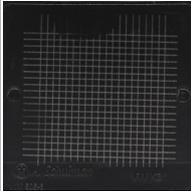
• Copolymer
40 % Filler

SUMMARY

APPENDIX



		
Talc	Talc + MAH	Talc + Antiscratch

			
Silfit Z 91	Silfit Z 91 + MAH	Aktifit AM	Aktifit AM + MAH

VM-01/0416/07.2018

Here again the differences are easy to recognize in the photographic images. With the combination of Aktifit AM and MAH, the grid lines can hardly be made out any longer – no brightening is in evidence.

5 Results PP Homopolymer, 40 % filler content

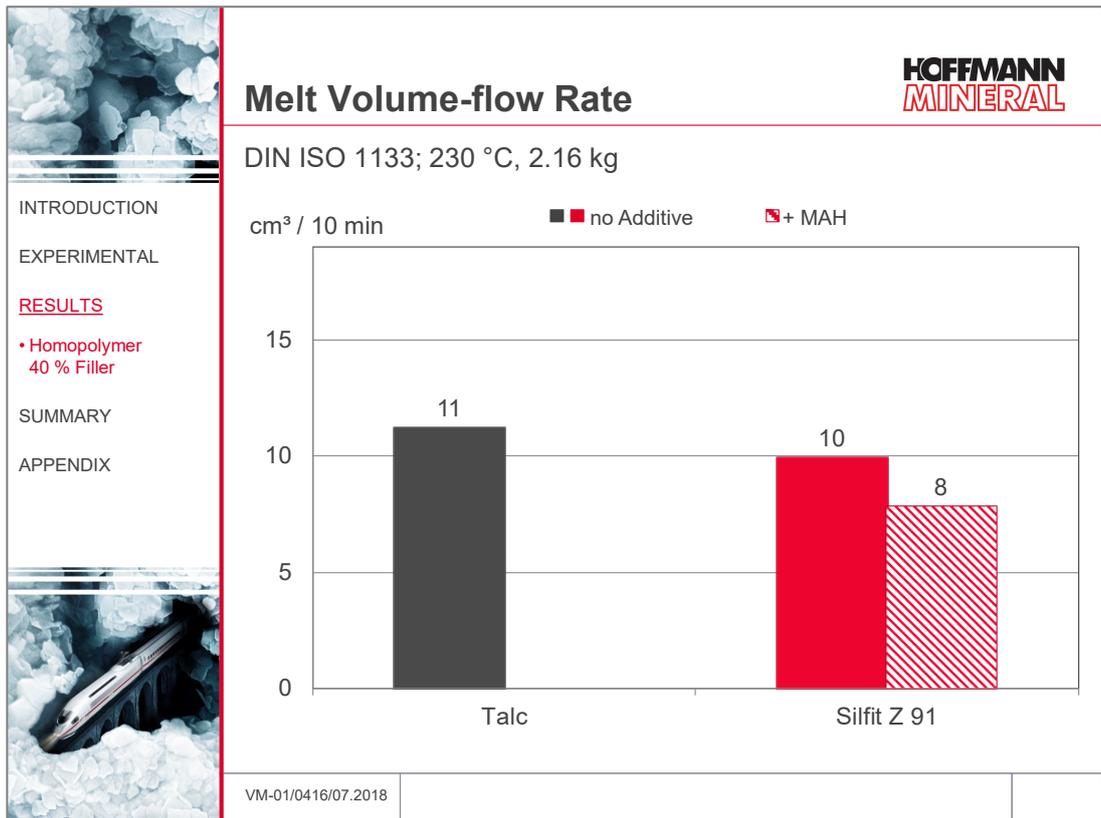
Formulations

In view of the fact that PP homopolymer largely is used for low-cost applications with minor requirements concerning impact strength, the number of compound formulations and tests in this section was correspondingly reduced.

		Talc		Silfit Z 91	
		---	---	MAH	
 INTRODUCTION <u>EXPERIMENTAL</u> • Homopolymer 40 % Filler RESULTS SUMMARY APPENDIX 	Formulations PP Homo 40 % Filler		HOFFMANN MINERAL		
	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	
VM-01/0416/07.2018					

The starting point was a standard PP homopolymer added with a stabilizer and a black color batch. The filler loading in this block was chosen at 40 weight percent. The fillers included the premium talc, but from the calcined Neuburg Siliceous Earth grades only Silfit Z 91. With the Silfit Z 91, also a compound with a MAH-modified PP was examined.

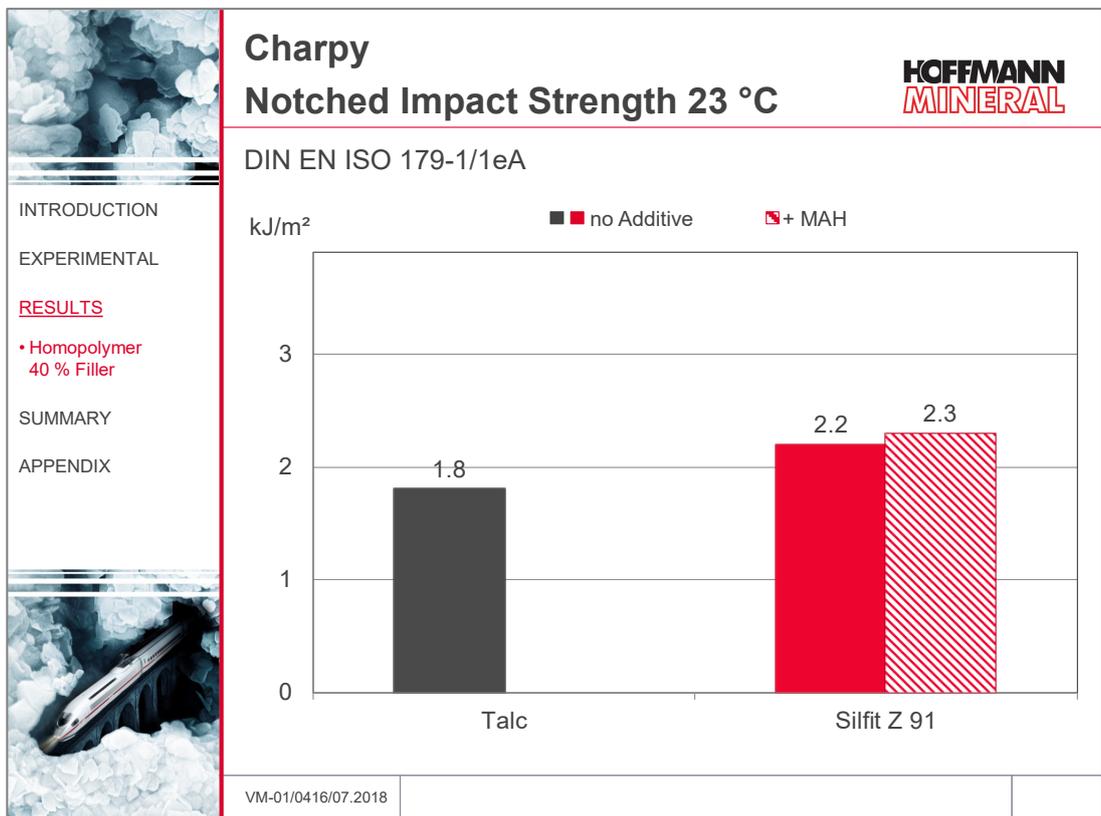
5.1 Melt volume-flow rate



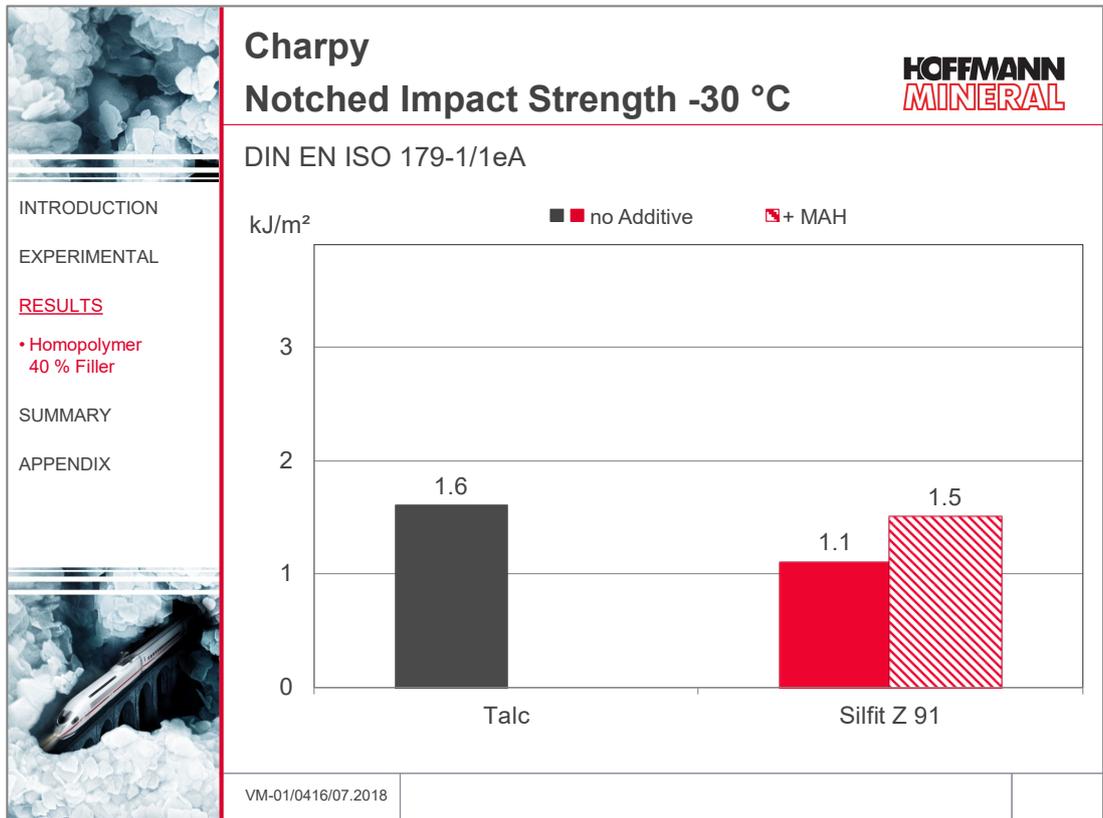
Similar to the results with the PP copolymer, the flow behavior of the compound with Silfit Z 91 is largely comparable with the talc compound. The flow properties suffer somewhat by the addition of MAH to the Silfit Z 91 compound.

5.2 Impact strength Charpy

Notched impact strength

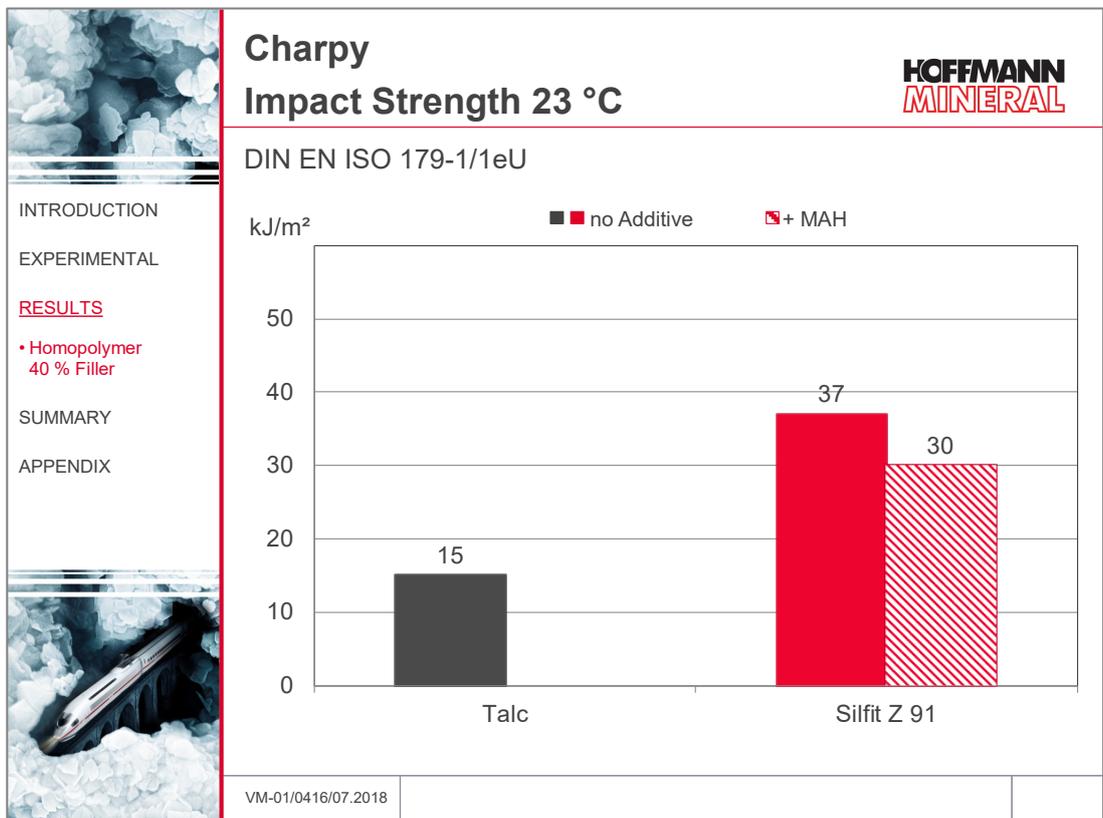


In notched impact strength, the Silfit Z 91 compounds came out somewhat higher compared with the talc compound. The addition of MAH here does not show any effect.

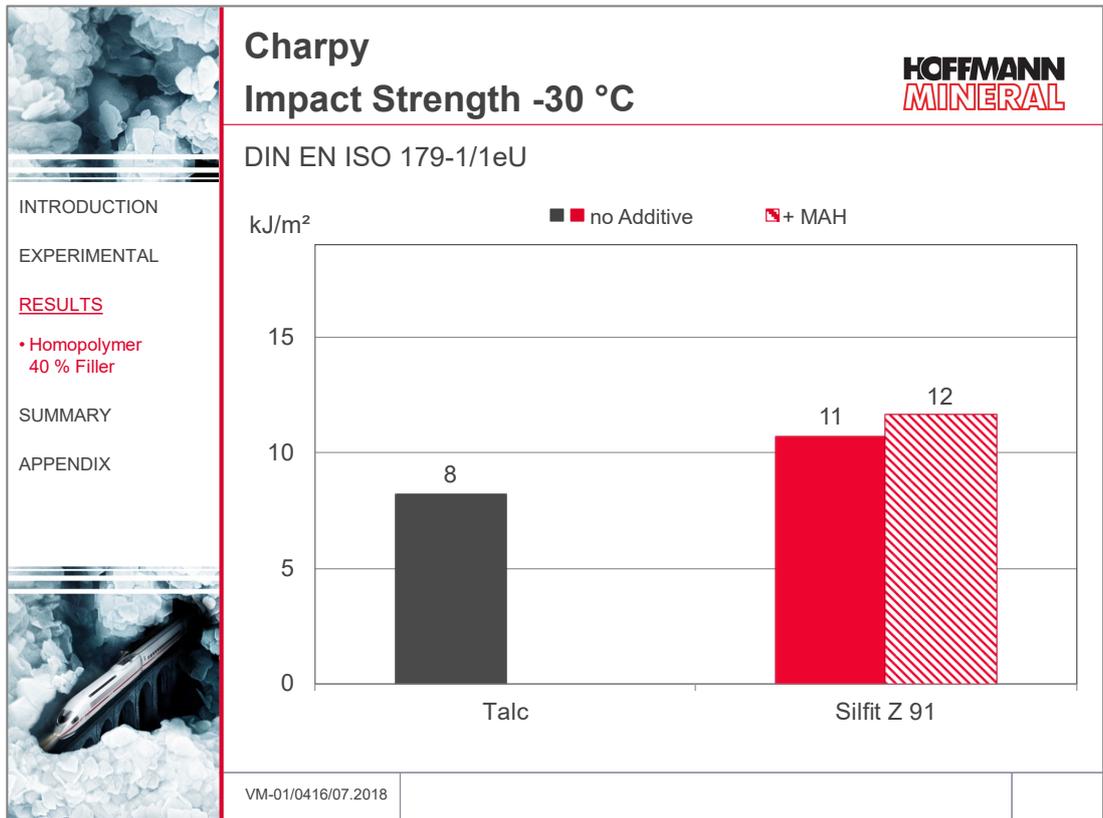


At low temperature, the notched impact strength with Silfit Z 91 drops more strongly than for the talc compound. Adding MAH brings the result back to the level of the talc compound.

Unnotched impact strength

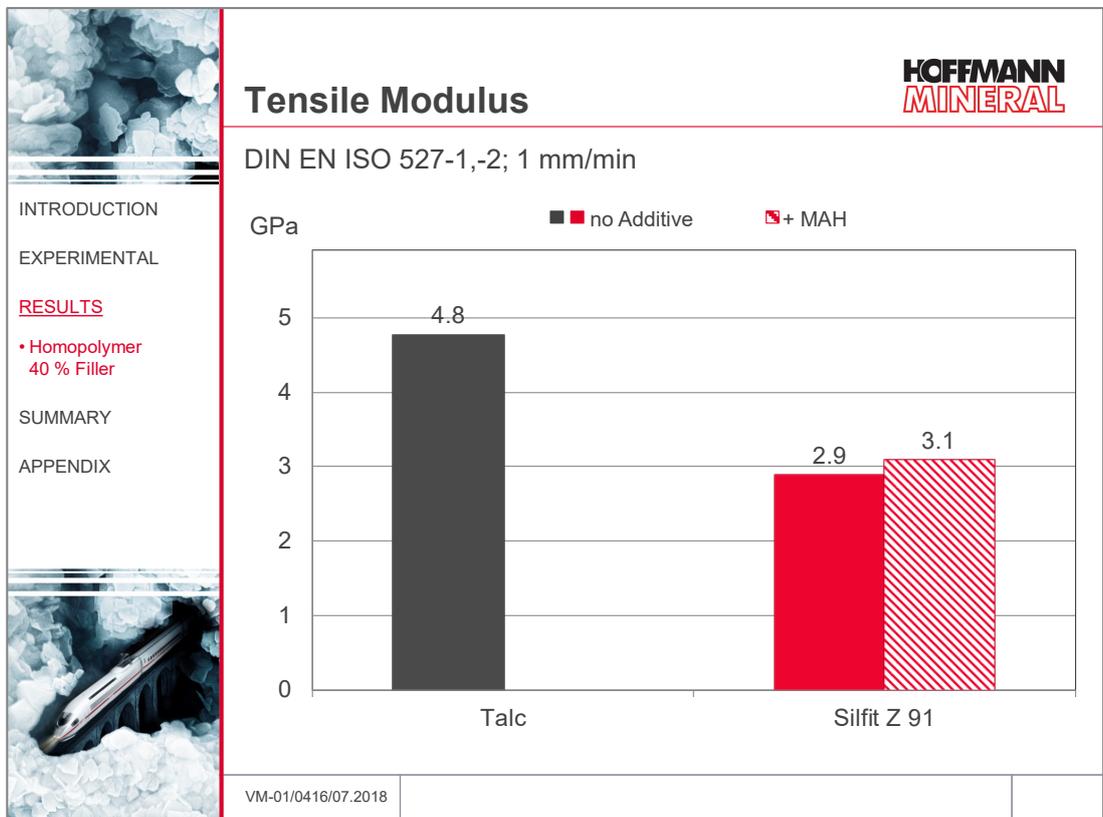


In the tests with unnotched samples, the impact strength is at least multiplied by a factor of two with Silfit Z 91.



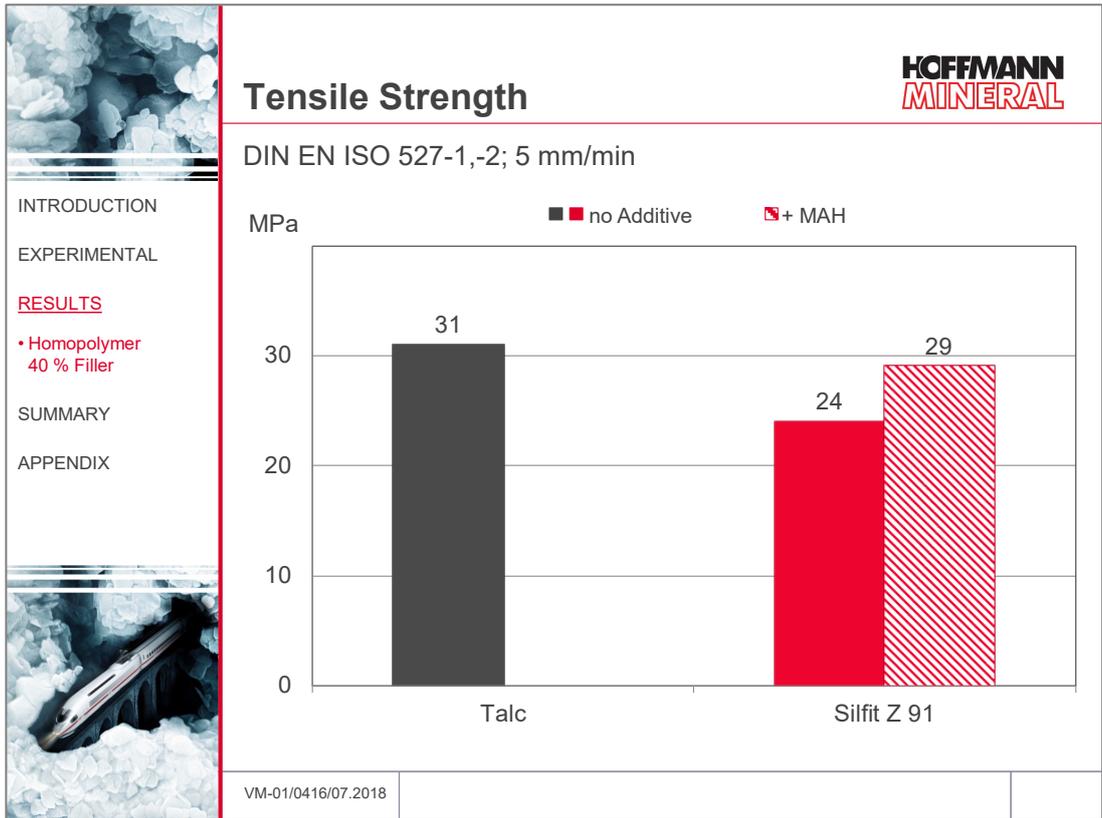
Even at low temperature an advantage is still observed with Silfit Z 91.

5.3 Tensile test Tensile modulus

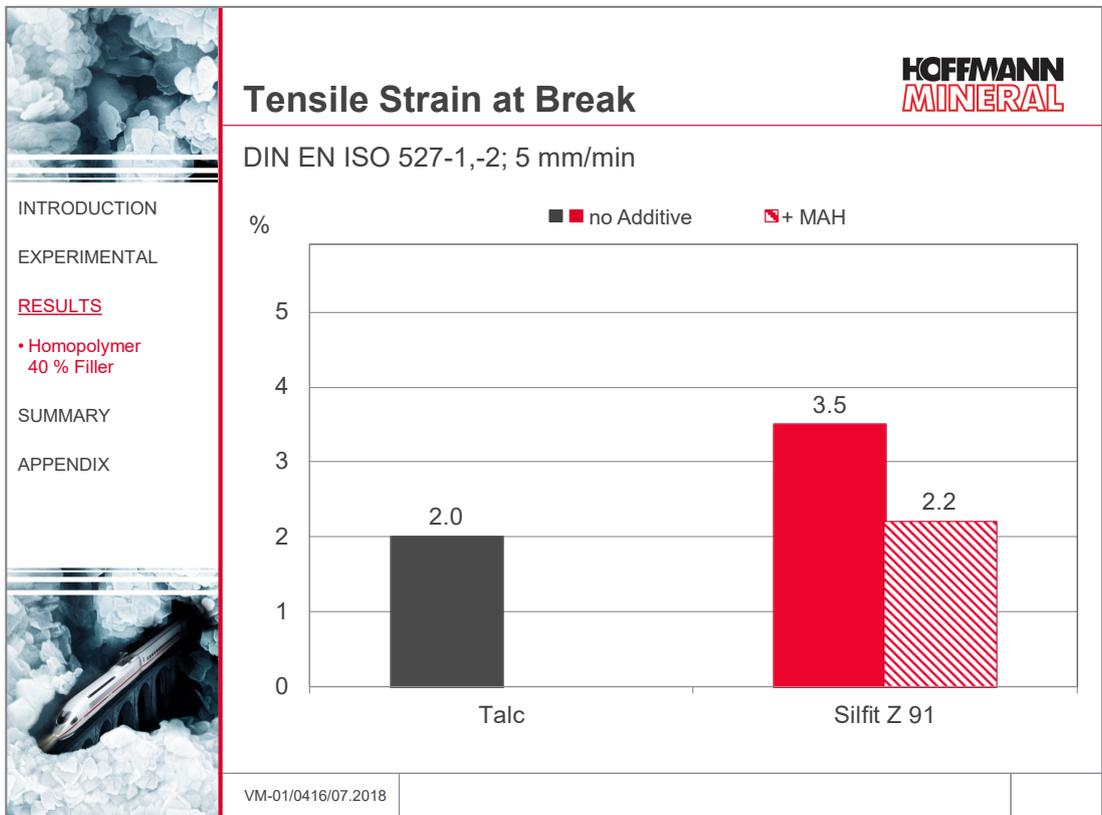


As already observed for the PP copolymer, the stiffness with the Calcined Neuburg Siliceous Earth grades, as a result of their morphological structure, comes off lower than with the ultrafine platelet-shaped talc.

Tensile strength and tensile strain at break



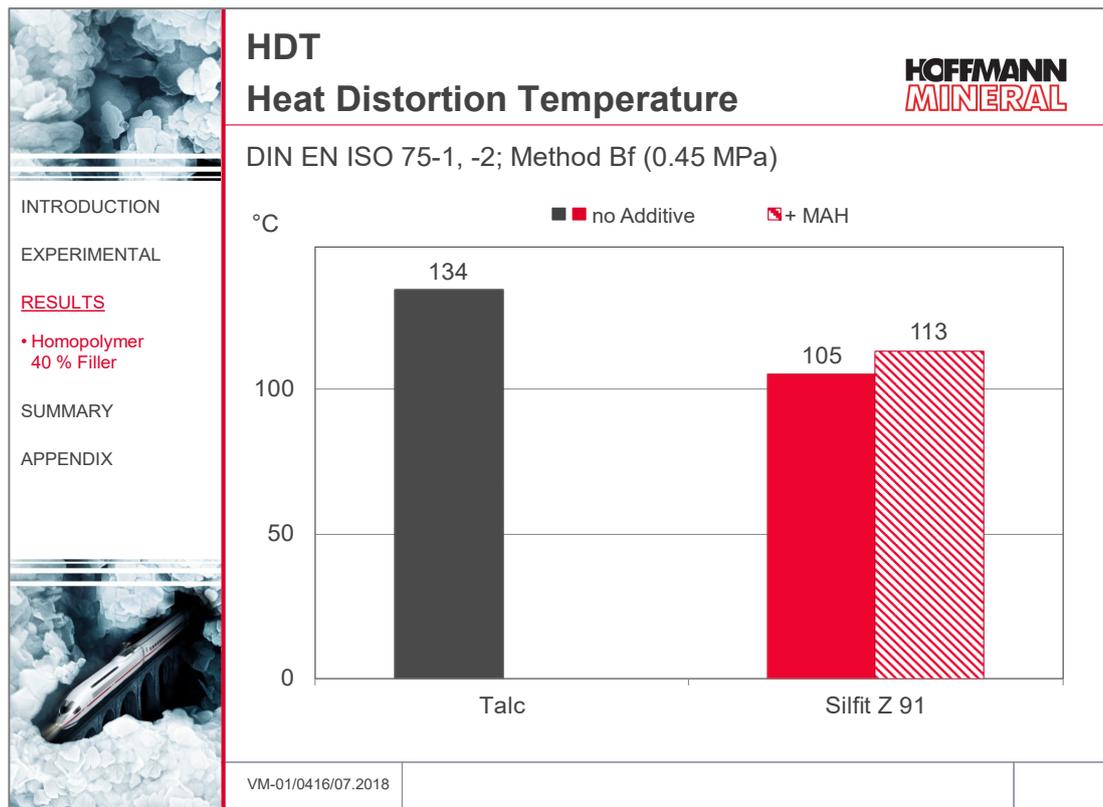
Like with the copolymer, also the tensile strength with Silfit Z 91 is somewhat lower than with the talc, but can be raised with MAH back to a level nearly comparable to talc.



An advantage remains for Silfit Z 91 with the higher strain at break, which also in combination with MAH does not come out poorer than with the talc without additive.

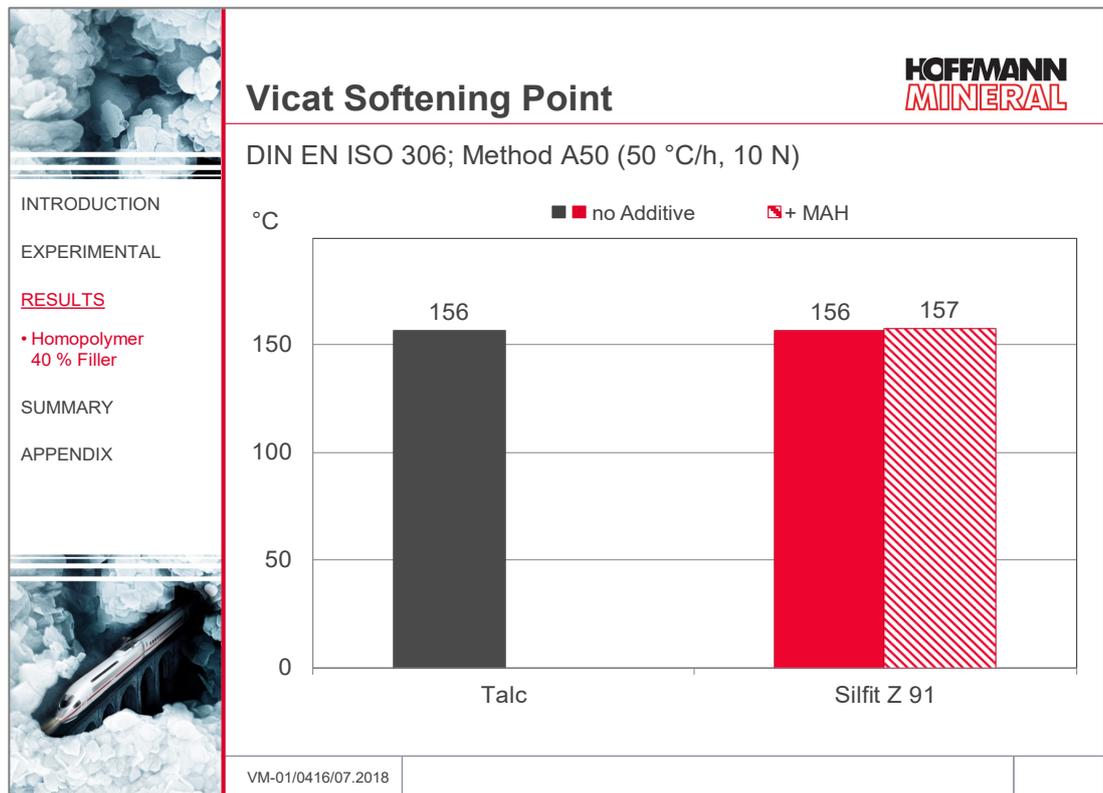
5.4 Heat deflection

HDT



The evaluation of the heat deflection temperature in the flexural mode again shows the influence of the lower E-modulus of the Silfit compounds insofar as the temperature comes out lower. Adding MAH here too leads to an improvement.

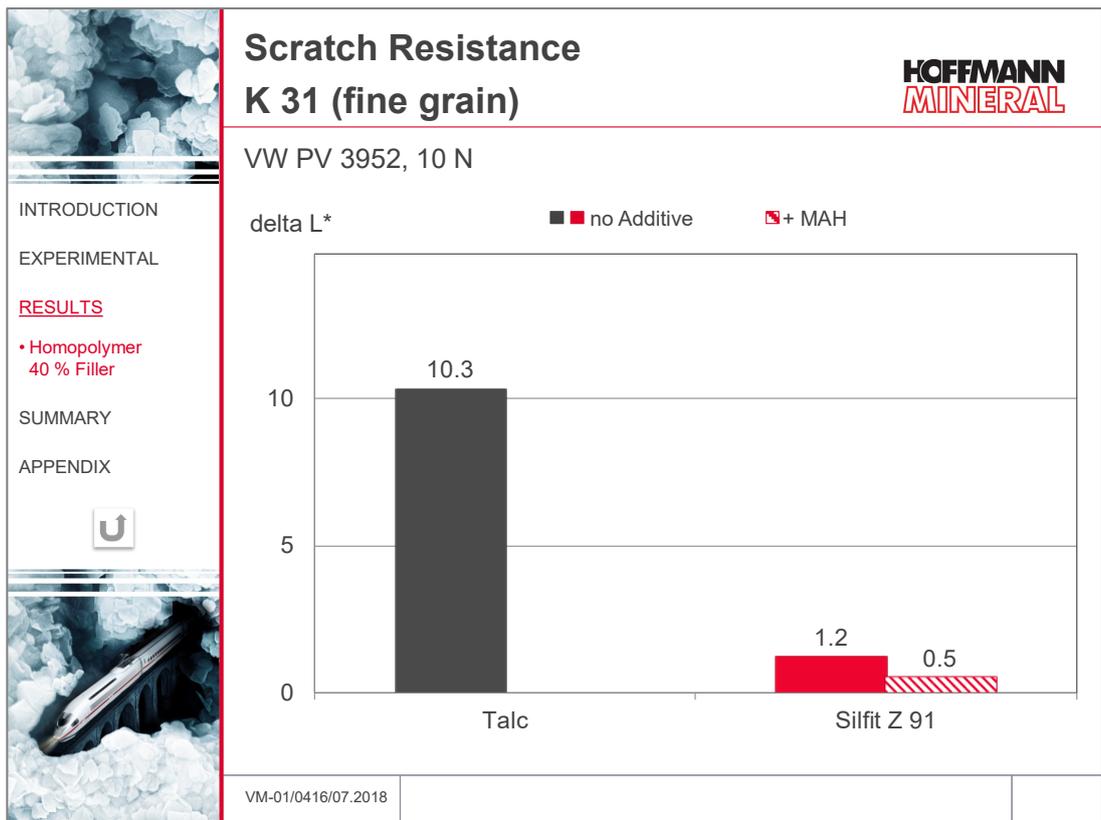
Vicat softening point



A different picture is obtained when the evaluation is made according to the Vicat method. In this case no differences exist any longer between the two fillers tested.

5.5 Scratch resistance

Fine-grained surface (K 31)



The greatest benefit, however, remains in the markedly improved scratch resistance on fine-grained substrate with Silfit Z 91, which can be somewhat further optimized by working in combination with MAH.

6 Summary

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

- Similar melt flow rate
- Somewhat lower heat deflection
- Same softening temperature
- Lower stiffness
- Somewhat lower tensile 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

Silfit Z 91 and especially Aktifit AM improve impact strength and scratch resistance in PP Compounds.

The addition of MAH-modified PP results in a further improvement of the scratch resistance, especially with Aktifit AM. Further effects are: improvement of tensile strength/yield stress and strain properties as well as of HDT and impact strength.

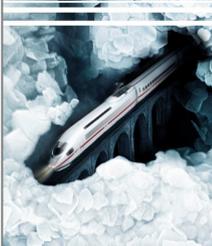
In PP homopolymer Silfit Z 91 already performs well.

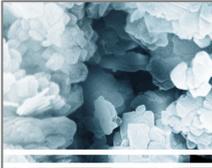
Comparing 40 % CSNE to 20 % talc, Silfit Z 91 and Aktifit AM outperform talc regarding scratch resistance and strain properties (Aktifit AM + MAH) despite the higher filler content.

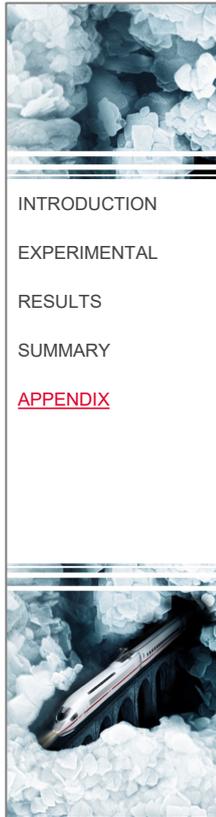
As a result of the high impact resistance with Silfit and Aktifit AM, in compounds designed for very high impact strength, special impact modifiers can be reduced, which also should help to alleviate differences in stiffness.

Our technical service suggestions and the information contained in this report are based on experience and are made to the best of our knowledge and belief, but must nevertheless be regarded as non-binding advice subject to no guarantee. Working and employment conditions over which we have no control exclude any damage claims arising from the use of our data and recommendations. Furthermore, we cannot assume any responsibility for any patent infringements which might result from the use of our information.

7 Appendix: Summary tables

		Talc			Silfit Z 91		Aktifit AM		
		Additive	---	MAH	Anti-scratch	---	MAH	---	MAH
 INTRODUCTION EXPERIMENTAL RESULTS SUMMARY APPENDIX 	PP Copo 20 % Filler								
	Summary table (1)								
	Density	g/cm ³	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	cm ³ /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	
VM-01/0416/07.2018									

		Talc			Silfit Z 91		Aktifit AM		
		Additive	---	MAH	Anti-scratch	---	MAH	---	MAH
 INTRODUCTION EXPERIMENTAL RESULTS SUMMARY APPENDIX 	PP Copo 20 % Filler								
	Summary table (2)								
	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/m ²	5.6	3.6	4.8	6.1	6.0	6.1	5.7	
Charpy 1eA -30 °C	kJ/m ²	1.8	1.3	1.7	1.1	1.5	1.6	2.1	
Impact strength									
Charpy 1eU (4 J) 23 °C	kJ/m ²	54	32	45	NB	70	NB	NB	
Charpy 1eU (7.5 J) 23 °C	kJ/m ²	---	---	---	145	---	115	126	
Charpy 1eU -30 °C	kJ/m ²	19	15	16	32	28	34	48	
VM-01/0416/07.2018									



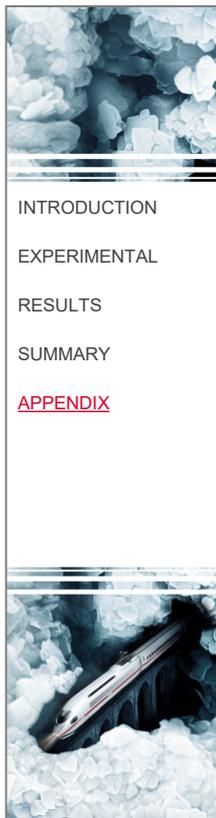
PP Copo 40 % Filler Summary table (1)

**HOFFMANN
MINERAL**

INTRODUCTION
EXPERIMENTAL
RESULTS
SUMMARY
[APPENDIX](#)

	Additive	Talc			Silfit Z 91		Aktifit AM	
		---	MAH	Anti-scratch	---	MAH	---	MAH
Density	g/cm ³	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	cm ³ /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

VM-01/0416/07.2018



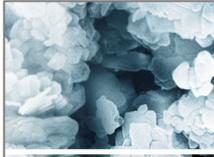
PP Copo 40 % Filler Summary table (2)

**HOFFMANN
MINERAL**

INTRODUCTION
EXPERIMENTAL
RESULTS
SUMMARY
[APPENDIX](#)

	Additive	Talc			Silfit Z 91		Aktifit AM	
		---	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
Yield 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/m ²	2.1	3.0	2.2	3.8	2.5	4.5	5.7
Charpy 1eA -30 °C	kJ/m ²	1.0	1.2	0.8	0.9	1.6	1.6	2.2
Impact strength								
Charpy 1eU 23 °C	kJ/m ²	15	20	15	78	49	69	93
Charpy 1eU -30 °C	kJ/m ²	9	12	8	17	21	18	38

VM-01/0416/07.2018



PP Homo 40 % Filler Summary table

**HOFFMANN
MINERAL**

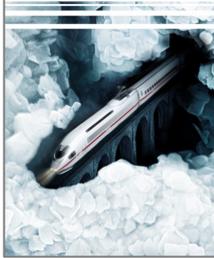
INTRODUCTION

EXPERIMENTAL

RESULTS

SUMMARY

APPENDIX



	Additive	Talc	Silfit Z 91	
		---	---	MAH
Density	g/cm ³	1.22	1.23	1.23
Filler content	%	38.5	39.6	39.7
Melt volume-flow rate	cm ³ /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/m ²	1.8	2.2	2.3
Charpy 1eA -30 °C	kJ/m ²	1.6	1.1	1.5
Impact strength				
Charpy 1eU 23 °C	kJ/m ²	15	37	30
Charpy 1eU -30 °C	kJ/m ²	8.2	10.7	11.6
Scratch resistance delta brightness L*				
K 31 (fine grain)	DL	10.3	1.2	0.5

VM-01/0416/07.2018