Aktifit AM

in AEM seals and gaskets

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1 Introduction

AEM is a specialty polymer which is for example used for "under the hood" applications in the automotive industry, as it offers outstanding resistance against heat and oils.

Until recently, carbon black which offers a well balanced property profile was the filler of choice in this area; but in numerous cases the results obtained did not give full satisfaction.

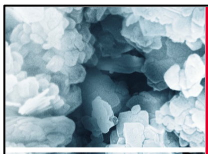
To date it has not been possible to avoid the problems generated by carbon black with a mineral filler without negatively affecting the strong points of the system.

The present study introduces the Calcined Neuburg Silicoeus Earth grade Aktifit AM as a proposed solution to replace carbon black N 550 in a diamine-crosslinked AEM compound.

In order to evaluate the results close to industrial conditions, the PSA standard S22 5106 Class 165 is used as base reference.

2 Experimental

2.1 Fillers and compound preparation



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Fillers and Characteristics

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	Particle Size d_{50} [µm]	Particle Size d_{97} [µm]	Oil absorption [g/100g]	Specific Surface Area BET [m²/g]	Functionalization
Carbon Black N 550	n.s.	n.s.	121	39	×
Aktifit AM	2	10	55	7	amino

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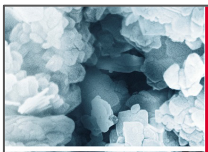
The low figures for the oil absorption and the specific surface area indicate that the product Aktifit AM, modified with an amino functional group, compared with carbon black N 550, is a filler of rather low activity.

Therefore, it is advisable to add a markedly higher amount to the compound, as shown in the present study.



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Preparation and Curing of the Compound

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- **Mixing**
  Open mill Ø 150 x 300 mm
  Batch volume: approx. 1000 g
  Temperature: 50 °C
  Mixing time: approx. 15 min.
- **Curing**
  Press, 10 min. / 180 °C
- **Post-cure**
  4 h / 185 °C
- **Hot air aging**
  168 h / 165 °C
- **Immersion in oil**
  Engine oil, type OS 206 304,
  168 h / 165 °C

All mechanical properties given in this report are related to the post-cured specimens.

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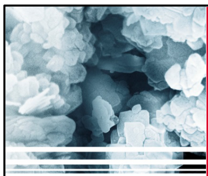
The compounding was done on a laboratory mill (Schwabenthan Polymix 150 L), with the rubber added at 50 °C, milled and pulled out to a uniform sheet. Subsequently, the filler was added along with the plasticizer and the processing aids before introducing the cross-linking system.

Both the sample sheets of 2 mm thickness and the compression set samples of 6 mm thickness were press-cured for 10 minutes at 180 °C and the following day post-cured 4 hours at 185 °C in a hot air oven. Following PSA S22 5106 Class 165, both hot air aging and immersion in engine oil were carried out for 168 hours at 165 °C. All mechanical results reported in the following refer to the post-cured samples.

2.2 Formulation



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Formulations
Target 60 – 65 Shore A

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in phr	Carbon Black N 550			Aktifit AM		
	10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Vamac Ultra IP	100	100	70	100	100	100
Vamac VMX-3110	-	-	30	-	-	-
Luvomaxx CDPA	2	2	2	2	2	2
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5
Vanfre VAM	1.5	1.5	1.5	1.5	1.5	1.5
Crodamide ER	1	1	1	1	1	1
N 550	50	50	50	-	-	-
Aktifit AM	-	-	-	137.5	137.5	137.5
Rhenosin W 759	10	20	10	25	30	-
Edenol T810T	-	-	-	-	-	30
Diak No 1	1.5	1.5	1.5	1.25	1.5	1.5
Luv. Safecure CA	3	3	3	3.5	3	3

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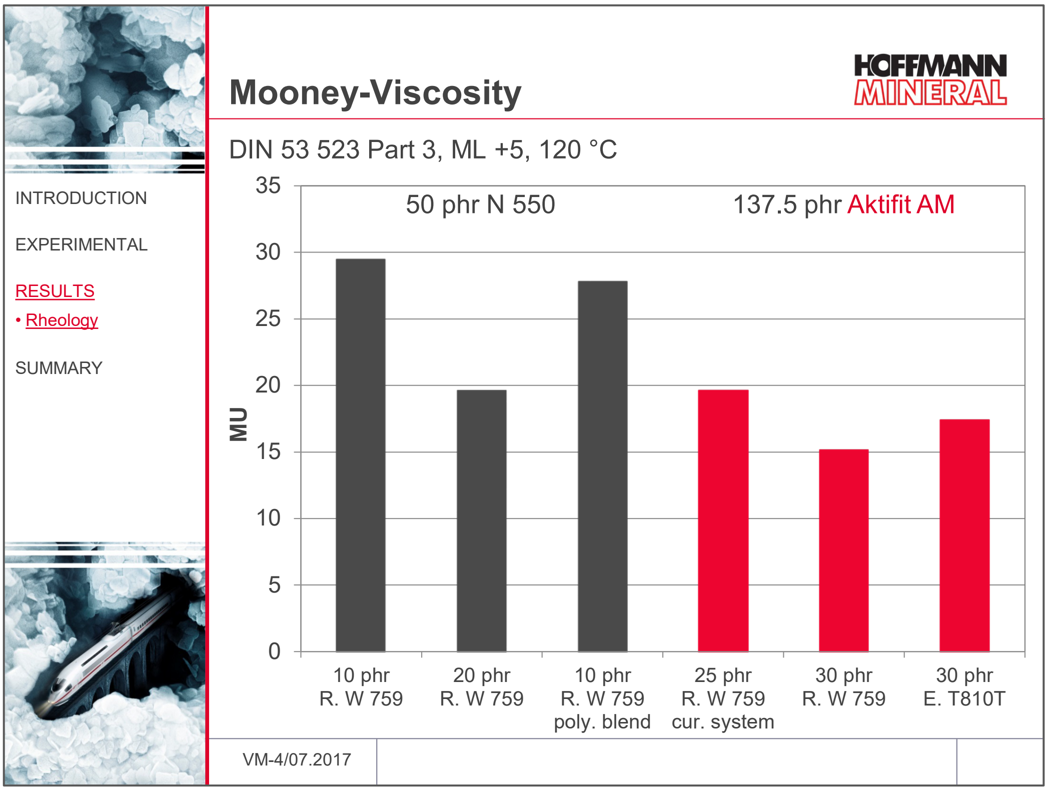
Vamac Ultra IP	AEM (ML 1+4, 100 °C: 29 ± 4 MU)
Vamac VMX-3110	AEM (ML 1+4, 100 °C: 30 ± 8 MU), higher oil resistance
Luvomaxx CDPA	antioxidant, diphenylamine
Stearic acid	processing aid
Vanfre VAM	processing aid
Crodamide ER	processing aid
Rhenosin W 759	ester/ether plasticizer
Edenol T810T	ester plasticizer
Diak No. 1	diamine
Luvomaxx SafeCure CA	activator

The reference was the compound with 50 phr N 550 and 10 phr Rhenosin W 759. In order to meet the specification PSA S22 5106 Class 165 with carbon black, different variants were evaluated. For one, the addition of Rhenosin W 759 was increased to 20 phr. Furthermore, a blend of Vamac Ultra IP and Vamac VMX-3110 was tested; this aimed at increasing the oil resistance along with adjusting a good compression set.

As already mentioned, it is possible to markedly increase the loading of Aktifit AM due to its lower activity. If hardness equal to the reference compound is to be obtained, this is even absolutely necessary. The present study, therefore, shows results with 137.5 phr Aktifit AM. Apart from the slightly modified curing system and the moderately increased plasticizer loading, a compound containing 30 phr Rhenosin W 759 was also included, as well as a replacement of this plasticizer with an equal amount of Edenol T810T.

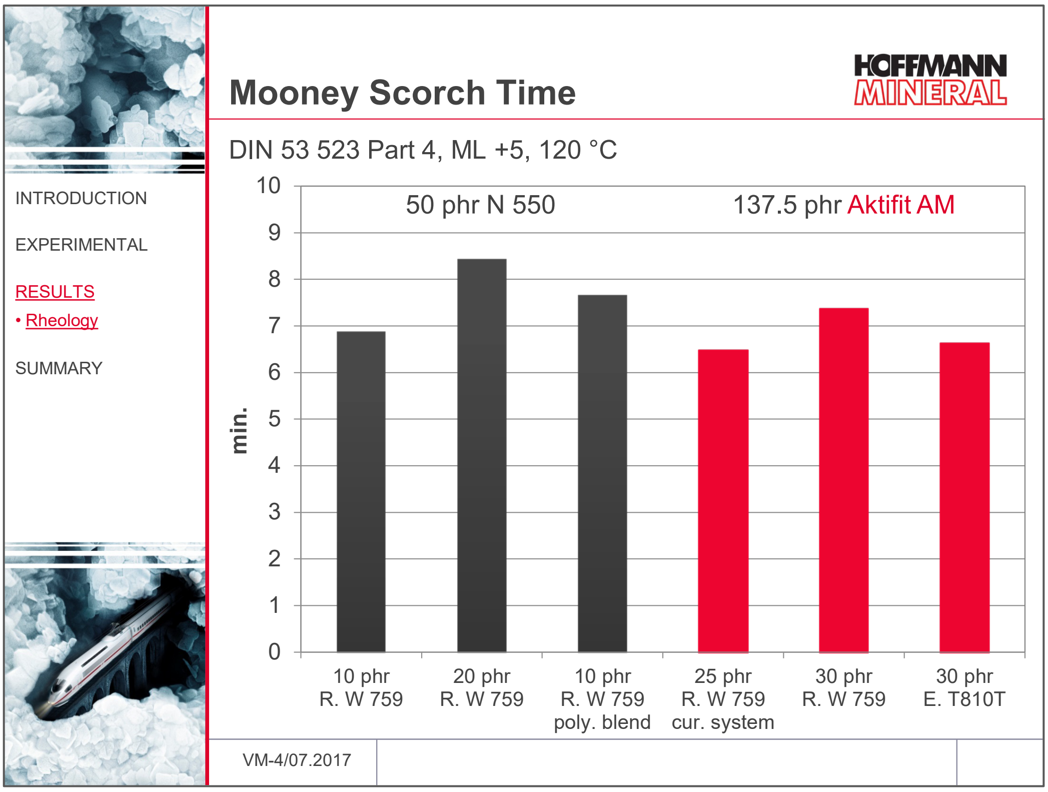
3 Results

3.1 Rheological properties



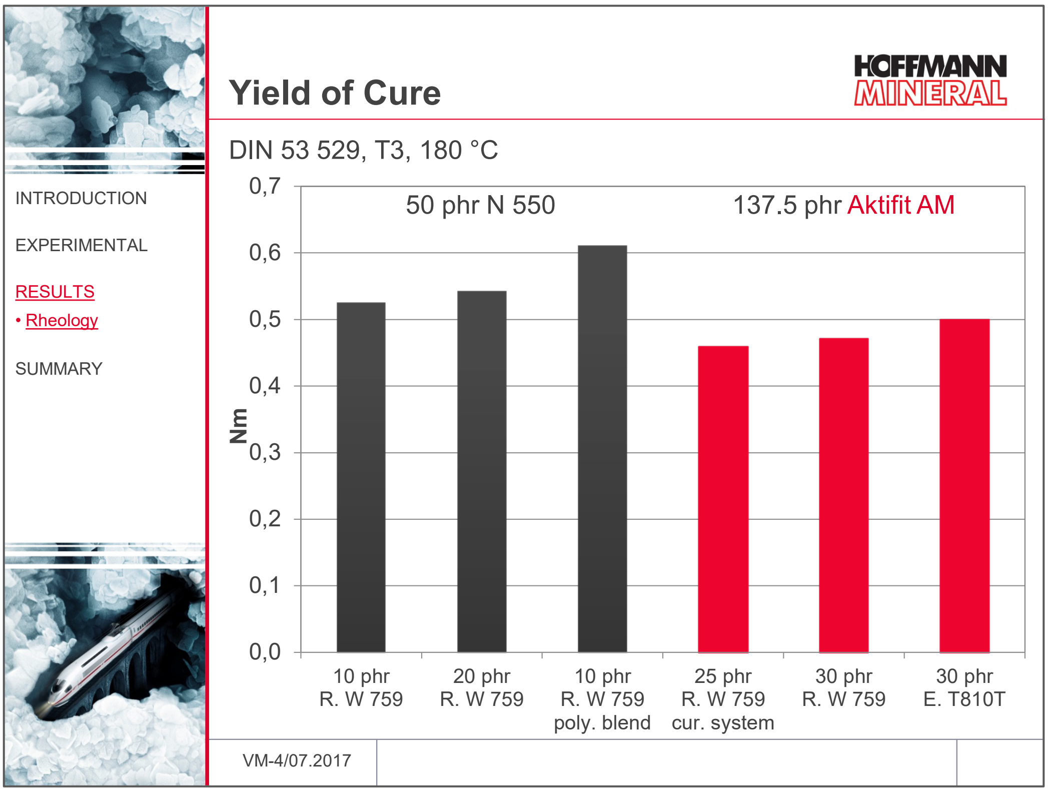
Upon increasing the plasticizer loading from 10 to 20 phr, according to expectations the Mooney viscosity will come out reduced, while blending Vamac Ultra IP with Vamac VMX-3110 practically does not affect the viscosity at all.

The compounds containing Aktifit AM offer viscosities practically on level with the carbon black compound with 20 phr plasticizer Rhenosin W 759. The replacement of Rhenosin W 759 with Edenol T810T remains practically without any significant effect.



The scorch time of all of compounds comes out above 6 minutes. Basically one can say that it becomes somewhat longer with increasing plasticizer content, i.e. the result is a somewhat improved processing safety when using a high amount of plasticizer. This is also true for the blend of Vamac Ultra IP and Vamac VMX-3110. The replacement of Rhenosin W 759 with Edenol T810T does not give any significant effect.

Between carbon black N 550 and Aktifit AM no clear differences with respect to the scorch behavior can be observed.



The yield of cure does practically not change with an increased plasticizer addition, while there is a certain increase when blending Vamac Ultra IP with Vamac VMX-3110, as evident in the carbon black containing compounds.

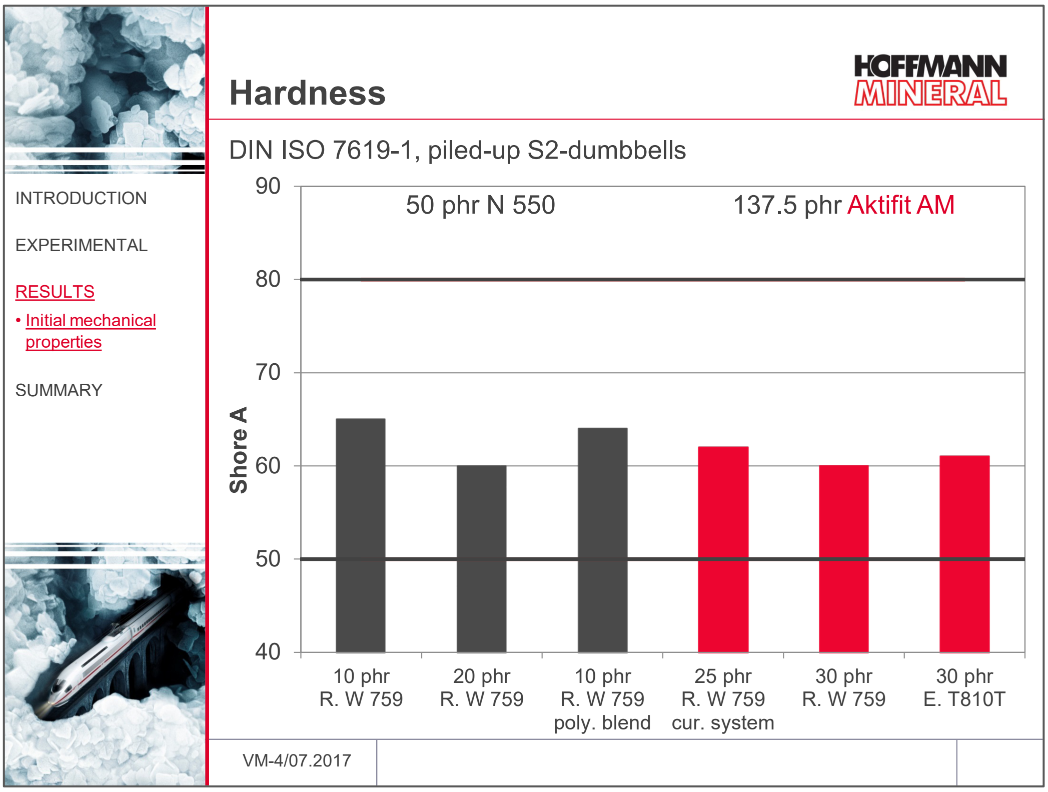
Within the compounds loaded with Aktifit AM, there is no difference in the crosslinking yield between the formulations with somewhat less Rhenosin W 759 in combination with the adjusted crosslinking system vs. the compound with 30 phr plasticizer. If here one works with Edenol T810T at equal dosage, the result is a somewhat higher yield of cure.

3.2 Mechanical properties

The mechanical properties given in the following, as already indicated, refer to the post-cured samples. Post-cure was carried out 4 hours at 185 °C.

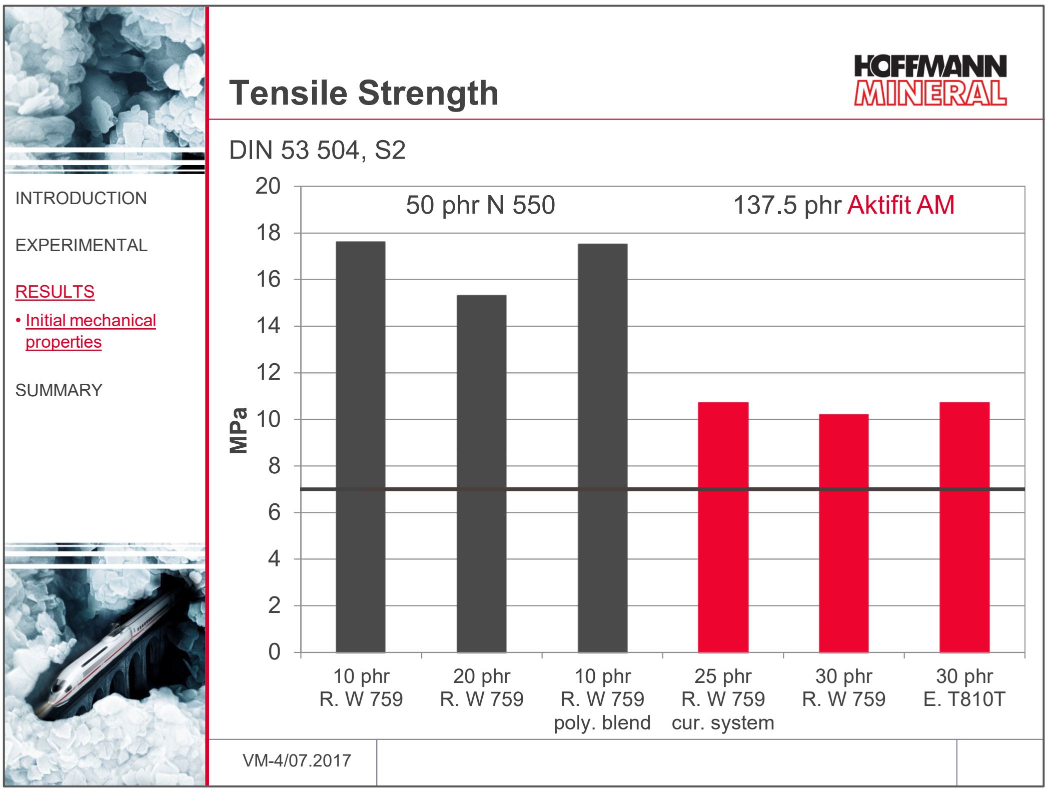
The results obtained are compared with the standard PSA S22 5106 Class 165 in order to arrive at an evaluation close to industrial conditions.

The specification limits of this standard are marked in the following graphs by red lines.

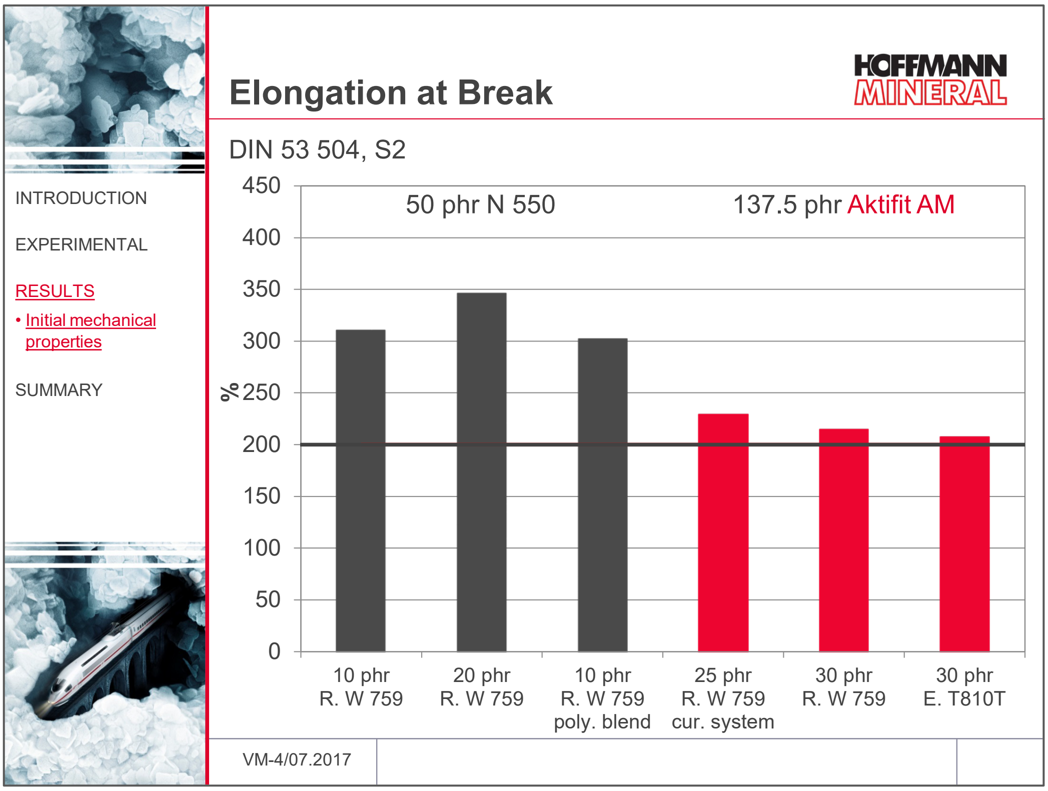


The high loading of Aktifit AM in combination with the increased plasticizer content leads to hardness values on a comparable level with the carbon black compounds which, however, contain a lower amount of filler.

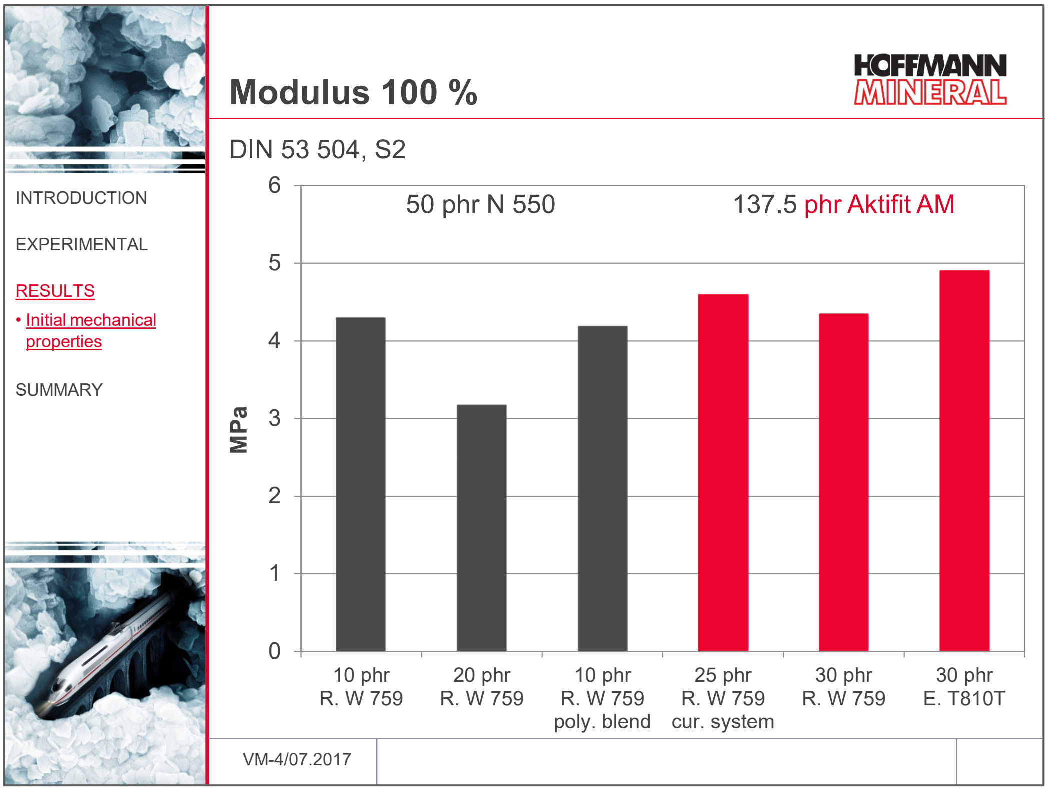
The change of the plasticizer or also the blend of Vamac Ultra IP with Vamac VMX-3110 do not affect the hardness which in all formulation variants comes out well inside the limits of the PSA S22 5106 Class 165 specification.



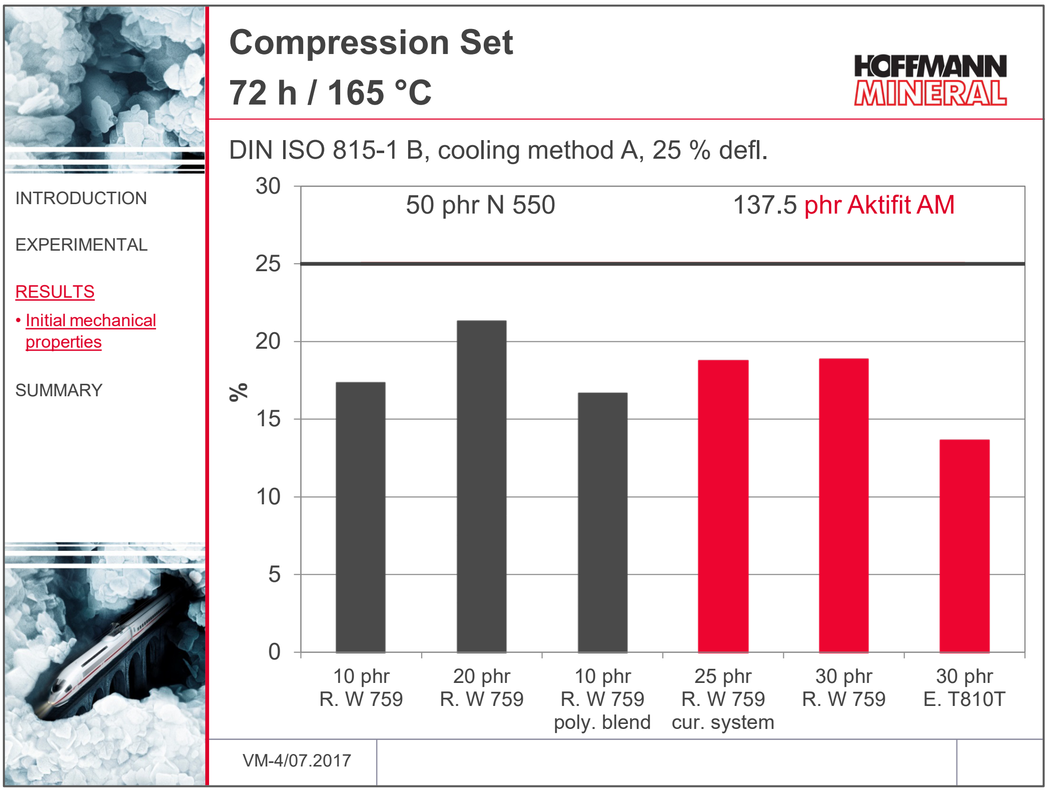
With respect to the tensile strength, the lower activity of Aktifit AM compared to the carbon black results in lower figures which, however, well meet the requirements of the PSA specification. Overall it does not make any difference if Rhenosin W 759 is replaced by Edenol T810T, or if the dosage is somewhat reduced and the crosslinking system slightly modified.



In the course of the study it proved difficult to reach the required oil resistance with Aktifit AM and simultaneously arrive at a sufficient initial elongation at break. By increasing the plasticizer content the elongation can now be moved upwards to go above the specification limit. The modified crosslinking system here comes out with the best result, while Edenol T810T only very marginally manages to meet the specification. As repeated tests on new compounds in our laboratories have confirmed, these results are well reproducible.



The modulus at 100 % elongation is not included in the PSA S22 5106 Class 165; it can, however, be used as an evaluation criterion for the handling of the cured articles on demolding after the injection molding process. The carbon black compounds with 10 phr Rhenosin W 759 represent industrial products which can be processed without any problems, i.e. demolded after cure in the mold. As the moduli of the compounds loaded with Aktifit AM are fully comparable with those of the carbon black mixes, a disadvantage on demolding does not appear very probable.

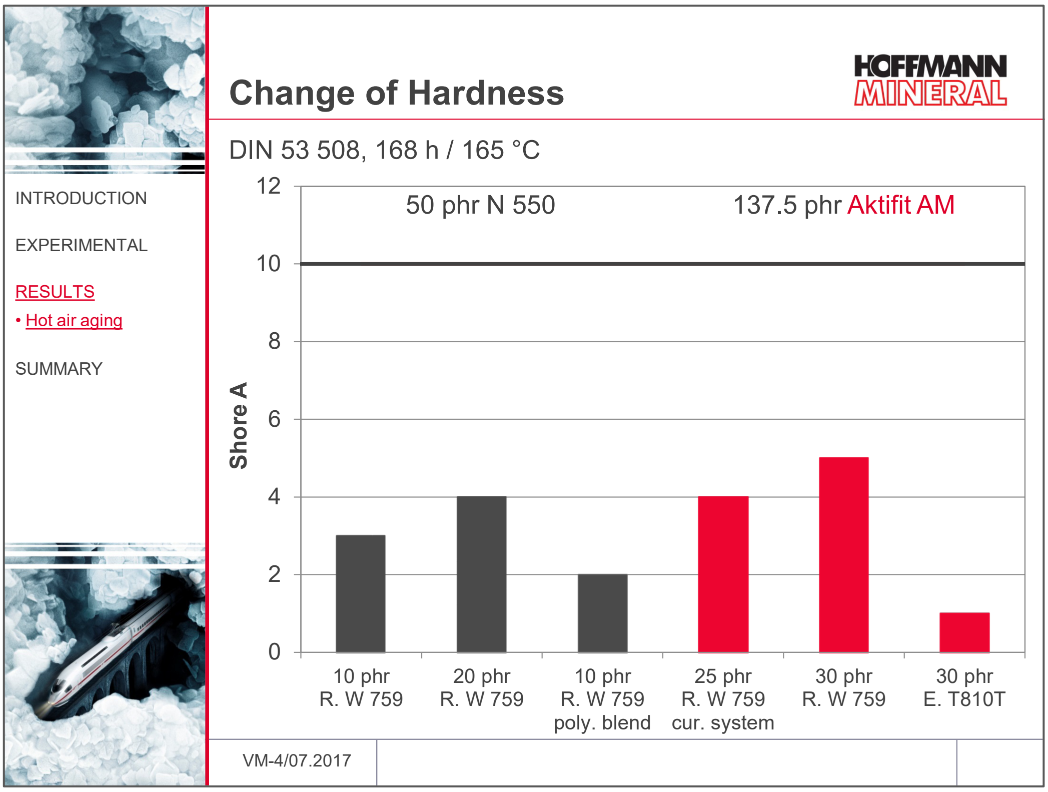


The higher Rhenosin W 759 addition leads to an increase of the compression set in the carbon black compounds, whereas no change in the results can be observed in the compounds based on the blend of Vamac Ultra IP and Vamac VMX-3110.

The higher loading of Aktifit AM in combination with an increased plasticizer addition results in compression set levels which are largely comparable with the reference compound. Here it does not make any difference if the combination of a somewhat reduced Rhenosin W 759 content with a modified curing system is used, or the plasticizer is moved up to 30 phr. In comparison it looks well possible to reduce the compression set by replacing Rhenosin W 759 with Edenol T810T.

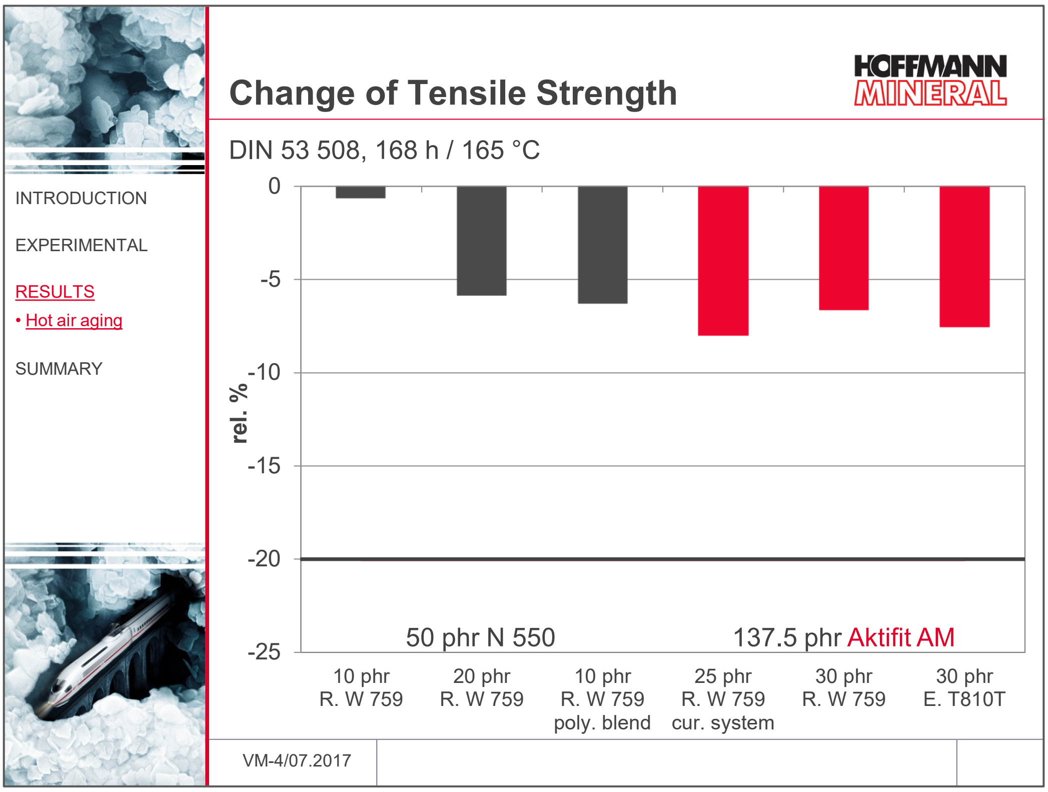
3.3 Heat resistance

The heat resistance, according to the PSA S22 5106 Class 165 specification was tested on post-cured S2-dumbbell specimens for 168 hours at 165 °C.

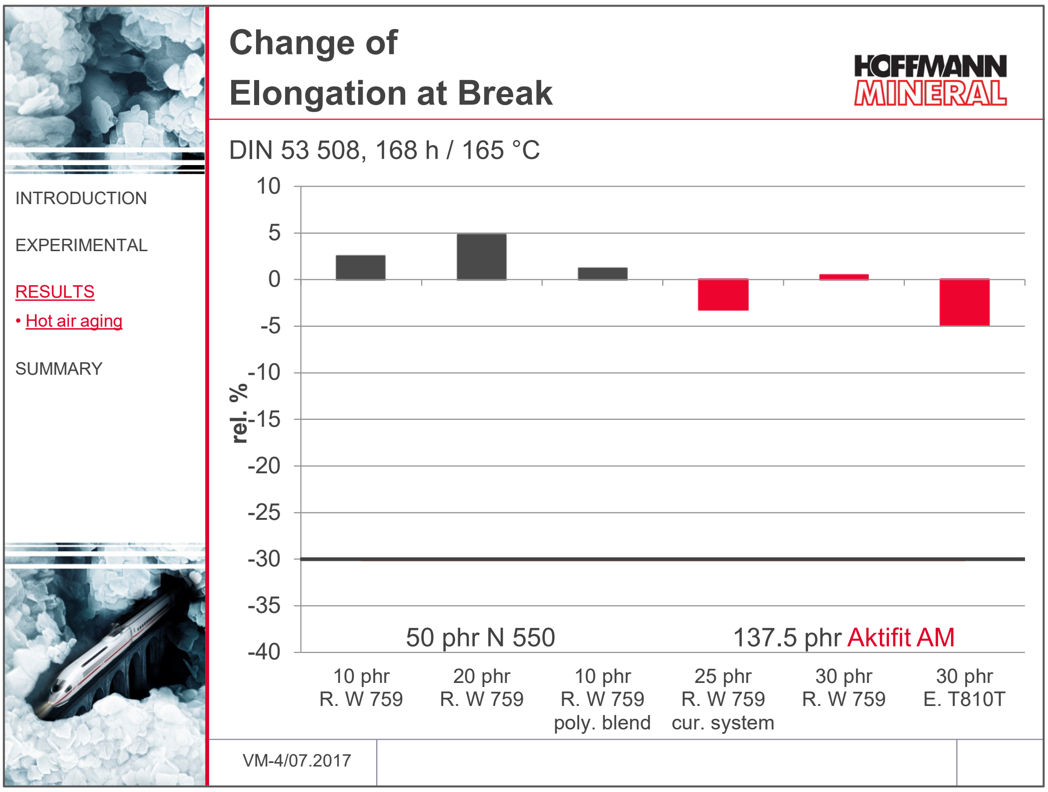


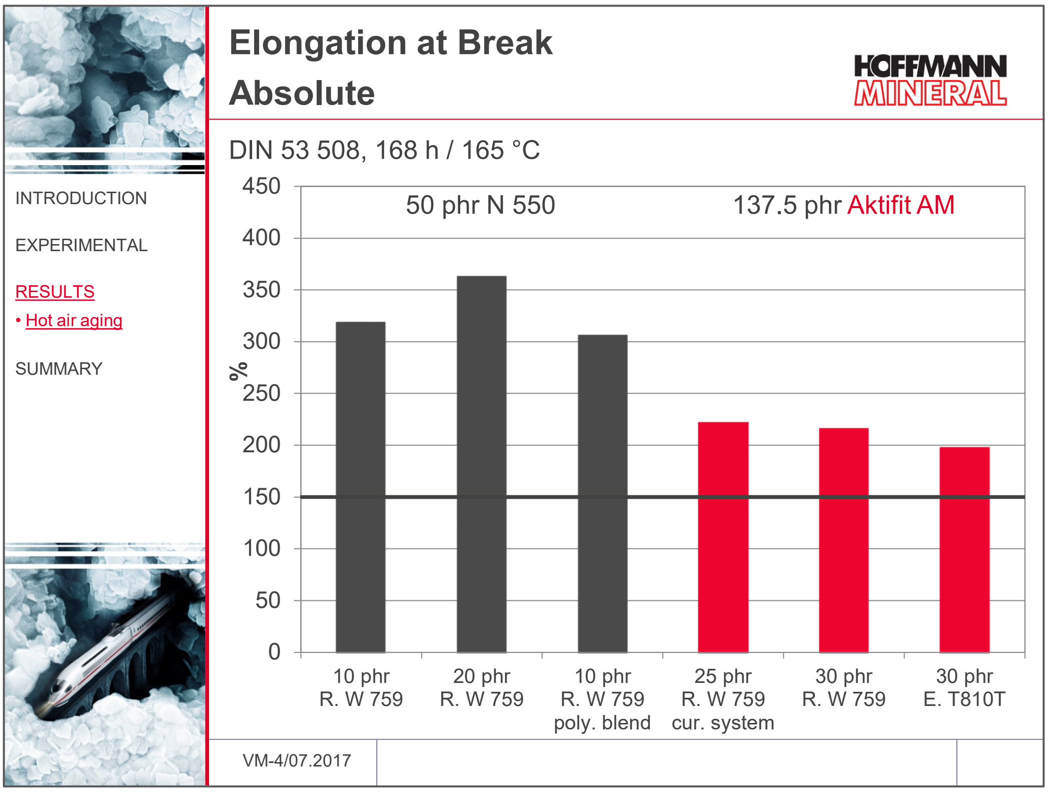
The change of hardness after hot air aging of the carbon black loaded compounds comes out similar to the Aktifit AM mixes, with both meeting the PSA specification without any problems.

The Vamac Ultra IP / Vamac VMX-3110 blend seems to offer some improvement and the replacement of Rhenosin W 759 with Edenol T810T leads to a further drop of the hardness increase.



The tensile strength as well as the elongation at break (lower graph) of the carbon black loaded or equally the Aktifit AM containing compounds do not significantly react to aging in hot air.



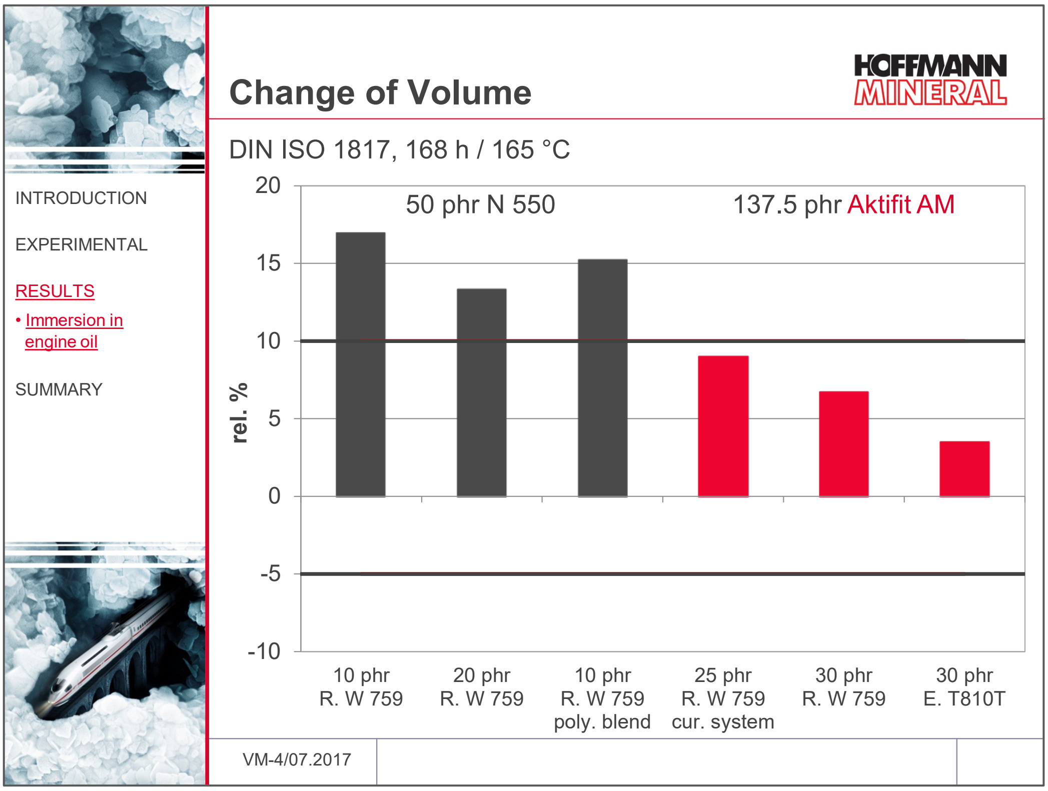


Elongation at break only is subject to minimal changes which means the results largely remain at the initial level.

As the results indicate, neither with the carbon black loaded compounds nor with the Aktifit AM containing compounds heat resistance represents a problem with respect to meeting the PSA S22 5106 Class 165.

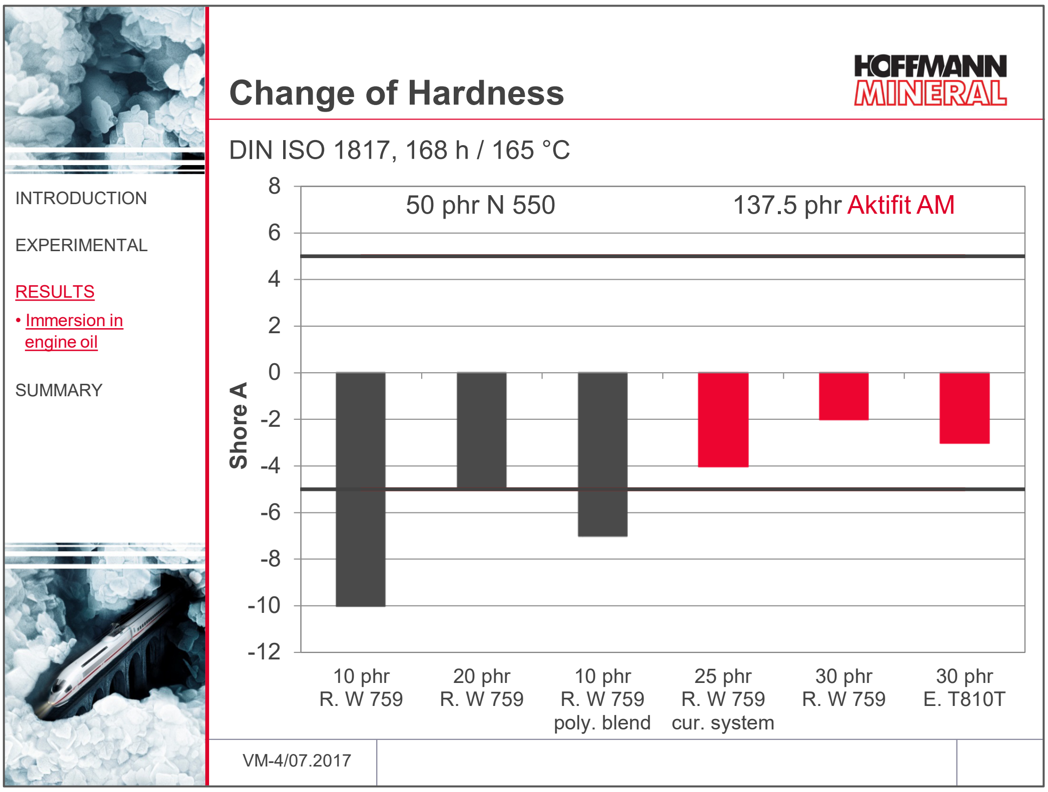
3.4 Oil resistance

The oil resistance according to the PSA S22 5106 Class 165 was tested on post-cured S2-dumbbell specimens immersed in reference oil OS206304 for 168 hours at 165 °C.

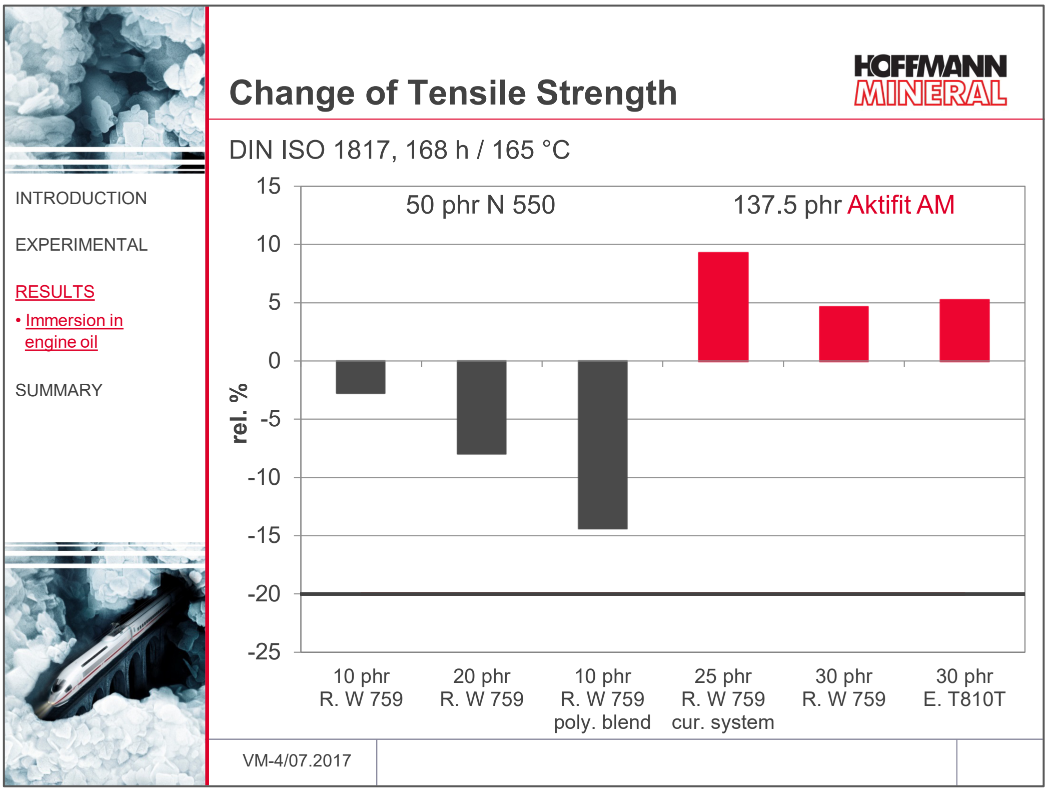


The reference compound loaded with 50 phr carbon black N 550 for the first time is not able to meet a requirement of the PSA specification. The volume change comes out too high and cannot be reduced sufficiently by increasing the plasticizer content nor by using the blend of Vamac Ultra IP with Vamac VMX-3110.

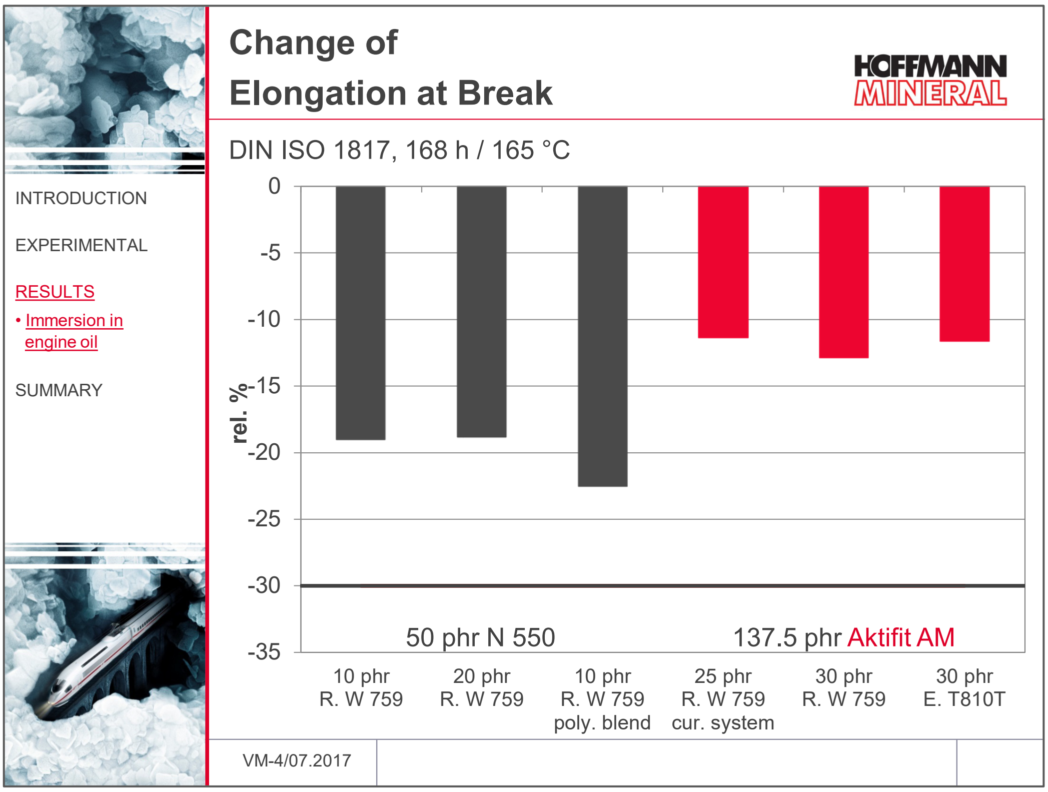
By working with Aktifit AM and this way increasing the filler loading, however, the PSA standard can already be met with 25 phr Rhenosin W 759 and the modified crosslinking system; when adding 30 phr of the plasticizer, the volume increase will come out somewhat further decreased. In addition, another advantage of Edenol T810T becomes evident: its use allows to decrease the volume change to below 5 %.



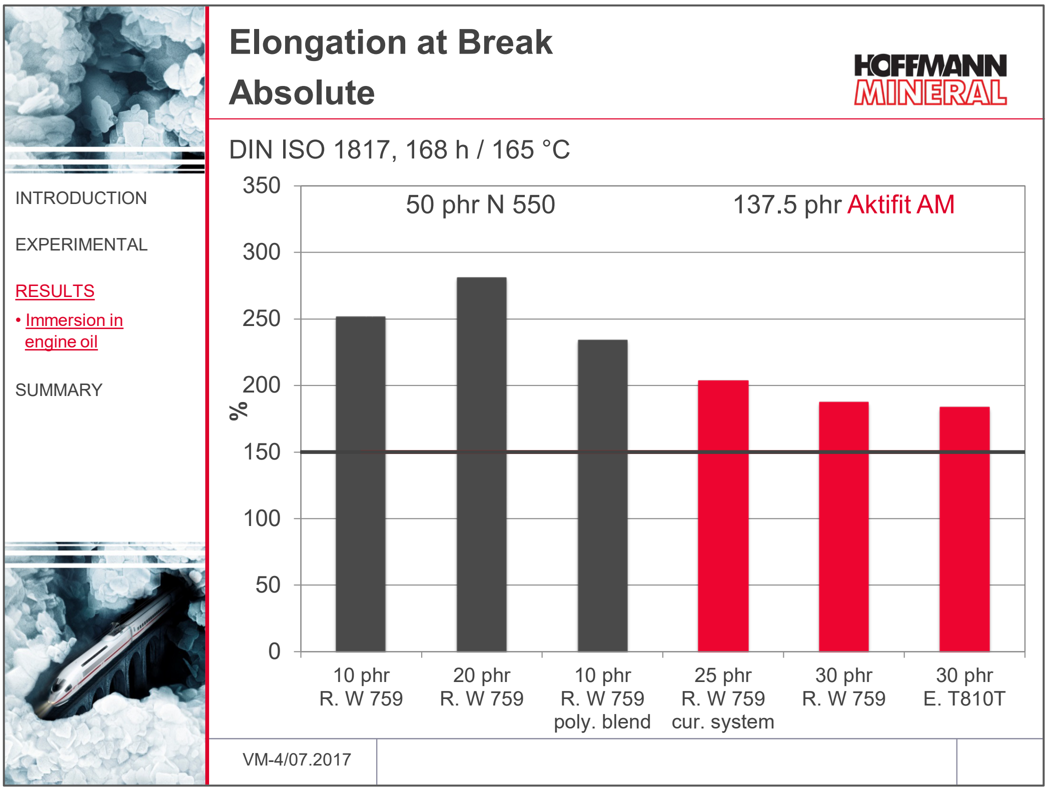
Similarly, meeting the requirement concerning the hardness change upon oil immersion is not possible with carbon black and just barely when adding 20 phr Rhenosin W 759, while all compounds loaded with Aktifit AM are able to come out within the limits of the PSA specification S 22 5106 Class 165.



It is true that the change of tensile strength for all compounds results relatively low, but still a marked trend towards generally higher figures is evident for the compounds loaded with Aktifit AM while the tensile strength of the carbon black loaded rubbers rather points to a lower level.



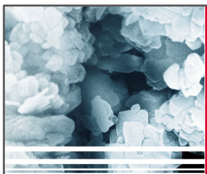
Likewise, the elongation at break of the compounds with Aktifit AM during oil immersion go down less than with the carbon black mixes which is why their absolute figures manage to level up a bit, as shown in the lower graph.



Overall, the oil resistance tests of the compounds reveal a clear advantage for the use of Aktifit AM in place of carbon black. In particular, the critical parameters volume increase and hardness decrease can be positively influenced with Aktifit AM.

4 Summary

For a comparative assessment of the compounds tested, a point system was developed which encompassed the critical requirements of the PSA S 22 5106 Class 165.
The following graph illustrates the points assigned to the initial quality level:



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SUMMARY 550

Rating
Initial Mech. Properties

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	Carbon Black N 550			Aktifit AM		
	10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Hardness	++	++	++	++	++	++
Tensile strength	++	++	++	+	+	+
Elongation at break	++	++	++	+	+	+
Modulus 100 %	++	+	++	++	++	++
Compression set	++	+	++	++	++	+++
Sum of +	**10**	**8**	**10**	**8**	**8**	**9**

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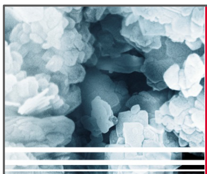
For the initial results, the evaluation of the carbon black compounds comes out somewhat better, as they impress with higher tensile strength and elongation at break figures. Only the variant with increased plasticizer content comes out at the same level of assessment as the Aktifit AM containing compounds because their compression set and modulus at 100 % elongation are somewhat lower. As expected, the compounds loaded with Aktifit AM give rise to lower tensile strength and elongation figures, but they still meet the PSA specification.



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Rating
Heat Resistance

HOFFMANN MINERAL

	Carbon Black N 550			Aktifit AM		
	10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Δ Hardness	++	++	++	++	++	++
Δ Tensile strength	++	++	++	++	++	++
Δ Elongation at b.	++	++	++	++	++	++
Elongation at b. abs.	++	++	++	+	+	+
Sum of +	**8**	**8**	**8**	**7**	**7**	**7**

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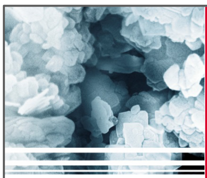
The heat resistance of all compound variations can be judged as equivalent. As the compounds containing Aktifit AM already start from a lower initial elongation, the carbon black is able to offer a slight advantage vs. the Calcined Neuburg Siliceous Earth.



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Rating
Resistance to Engine Oil

	Carbon Black N 550			Aktifit AM		
	10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Δ Hardness	- -	+	-	++	++	++
Δ Tensile strength	+	+	+	++	++	++
Δ Elongation at b.	+	+	+	++	++	++
Elongation at b. abs.	++	++	++	+	+	+
Δ Volume	- -	- -	- -	+	+	++
Sum of +	**0**	**3**	**1**	**8**	**8**	**9**

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When comparing the individual parameters concerning oil resistance, however, the benefits of Aktifit AM come out clearly. Here too the absolute figures for the elongation at break are lower than for the carbon black but the requirements with respect to decrease of hardness and volume change cannot be met neither with an increased plasticizer addition nor by blending Vamac Ultra IP with Vamac VMX-3110.

As in view of the lower filler activity of Aktifit AM it is possible or even necessary to work with higher loadings in order to arrive at the hardness of the reference compound, the changes of hardness and volume during oil immersion can be adjusted to fall within the PSA specification S22 5106 Class 165.

This can be achieved with 25 phr Rhensoin W 759 in combination with a modified curing system or alternatively by increasing the plasticizer content to 30 phr. The volume change can be further improved by replacing Rhenosin W 759 with an equal amount of Edenol T810T.

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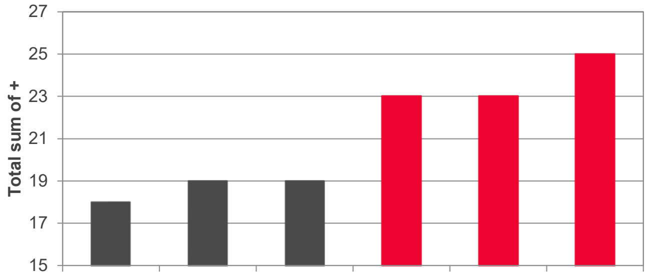
Total Sum of +

HOFFMANN MINERAL

	Carbon Black N 550			Aktifit AM		
	10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Initial mech. properties	10	8	10	8	8	9
Heat resistance	8	8	8	7	7	7
Engine oil resistance	0	3	1	8	8	9
Total sum of +	**18**	**19**	**19**	**23**	**23**	**25**



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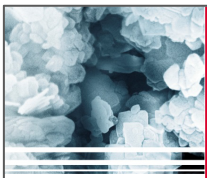
If the assigned points of each category are summed up, it becomes again clear that the use of Aktifit AM results in compounds which in particular with respect to oil resistance offer advantages over carbon black compounds and which all in all enjoy a well balanced property profile.

An aspect which has not been considered in this study but which potentially could be of importance is the improved low temperature resistance that can be expected in view of the high plasticizer additions.

Another advantage when using Aktifit AM in place of carbon black refers to color. The color-neutral mineral filler makes it possible to arrive at compounds which can individually be combined with components differentiated by various colors.

Furthermore, carbon black is a mineral oil based raw material whose price has gone up steadily over recent years which of course negatively affects the compound costs. By contrast, Aktifit AM is a filler which is not subject to price changes along with mineral oils and which further via higher loading (in combination with the increased plasticizer addition) is able to reduce the compound costs compared with carbon black compounds.

Conclusion:
Calcined Neuburg Siliceous Earth Aktifit AM has been confirmed as a mineral filler which can replace carbon black N 550 with positive effects in AEM based seals and gaskets for applications "under the hood" in the automotive industry.



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		Carbon Black N 550			Aktifit AM		
		10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Rheology							
Mooney viscosity, ML 1+4, 120 °C	MU	29	20	28	20	15	17
Mooney scorch time, ML +5, 120 °C	min.	6.9	8.4	7.7	6.5	7.4	6.6
Rotorless curemeter M_{max} 180 °C	Nm	0.55	0.56	0.64	0.47	0.48	0.51
Rotorless curemeter t_{90} 180 °C	min.	6.5	7.8	4.4	2.6	3.7	3.7
Mechanical Properties – curing conditions 10 min. / 180 °C, post-cure 4 h / 185 °C							
Hardness	Sh. A	61	60	64	62	60	61
Tensile strength	MPa	18	15	18	11	10	11
Elongation at break	%	310	346	302	229	215	207
Modulus 50 %	MPa	1.8	1.4	1.7	1.8	1.7	1.8
Modulus 100 %	MPa	4.3	3.2	4.2	4.6	4.3	4.9
Compression Set 72 h / 165 °C, 25 % defl.	%	17	21	17	19	19	14

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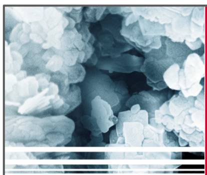

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		Carbon Black N 550			Aktifit AM		
		10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Mechanical Properties – after hot air aging, 168 h / 165 °C (post-cured)							
Hardness	Sh. A	65	64	66	66	65	62
Tensile strength	MPa	17	14	16	9.8	9.5	9.9
Elongation at break	%	318	362	306	222	216	197
Modulus 50 %	MPa	2.0	1.6	1.8	1.8	1.8	1.8
Modulus 100 %	MPa	4.6	3.4	4.1	4.5	4.5	4.9
Δ Hardness	Sh. A	+3	+4	+2	+4	+5	+1
Δ Tensile strength	%	-1	-6	-6	-8	-7	-7
Δ Elongation at break	rel. %	+3	+5	+1	-3	0	-5
Δ Modulus 50 %	%	+11	+11	+2	-1	+8	-1
Δ Modulus 100 %	%	+7	+7	-1	-3	+3	-1

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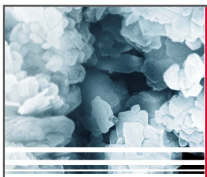

Table of Results

HOFFMANN MINERAL

		Carbon Black N 550			Aktifit AM		
		10 phr W 759	20 phr W 759	10 phr W 759 pol.blend	25 phr W 759 cur.syst.	30 phr W 759	30 phr T810T
Mechanical Properties – after immersion in engine oil OS 206 304, 168 h / 165 °C (post-cured)							
Hardness	Sh. A	55	55	57	58	58	58
Tensile strength	MPa	17	14	15	12	11	11
Elongation at break	%	251	281	234	203	187	183
Modulus 50 %	MPa	1.8	1.5	1.7	1.8	1.8	2.0
Modulus 100 %	MPa	5.1	3.9	4.9	5.0	5.0	5.6
Δ Hardness	Sh. A	-10	-5	-7	-4	-2	-3
Δ Tensile strength	%	-3	-8	-14	+9	+5	+5
Δ Elongation at break	rel. %	-19	-19	-22	-11	-13	-12
Δ Modulus 50 %	%	+5	+9	0	+3	+9	+10
Δ Modulus 100 %	%	+18	+23	+17	+10	+14	+13
Δ Volume	%	+17	+13	+15	+9	+7	+4

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Supplement September 2016

Luvomaxx is no longer available, the recommended replacement is Luvomaxx DBU DL 70 (2.57 phr ≘ 3 phr Luvomaxx Safecure CA).

This leads to a strong decrease of the elongation at break of compounds filled with Aktifit AM.

The solution is a partial replacement of Aktifit AM by Silfit Z 91 in a ratio of 75:25. The benefits of the Calcined Neuburg Siliceous Earth over carbon black remain.

Furthermore, injection molding tests did not show any significant differences between carbon black filled compounds and those filled with Calcined Neuburg Siliceous Earth.