HOFFMANN MINERAL
Unternehmensgruppe Hoffmann

Neuburg Siliceous Earth as an acid resistant and tintable alternative to carbon black N990 in peroxide cured FKM

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1 Introduction

Fluoropolymers are generally known for their high temperature and media resistance. Therefore, they are commonly used in sealing and media routing applications where the properties of other polymers are insufficient.

The property profile of fluoropolymers can be improved by using fillers.

Either carbon black N990 or mineral fillers, such as wollastonite or barium sulfate have been used up to now.

The latter turn out to be critical when it comes to acid resistance, as the following figure shows:

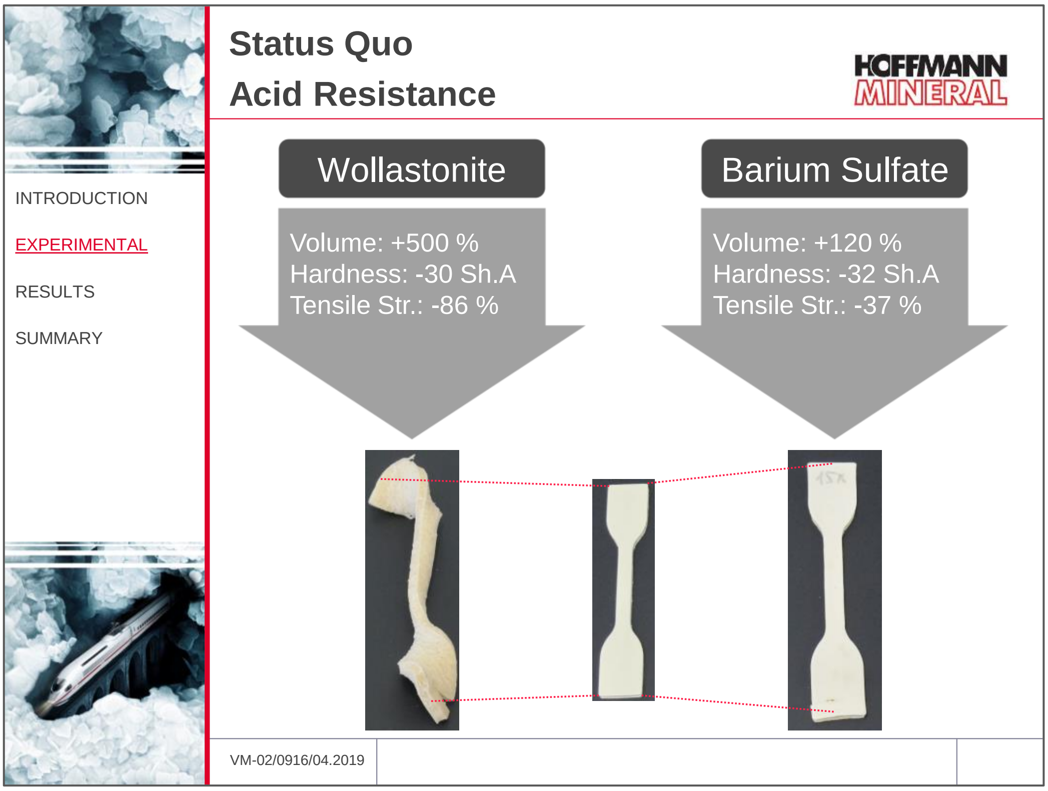
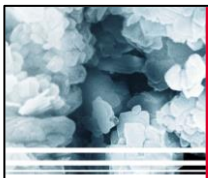


Fig. 1

This paper presents different grades of Neuburg Siliceous Earth (NSE) as alternatives to carbon black N990 in a peroxide cured FKM formulation in order to show some examples of acid resistant and simultaneously tintable vulcanized rubbers.

2 Experimental

2.1 Filler, formulation and compound preparation



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Fillers, Characteristics

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Filler	Description	Functionalization
N990	Carbon black, MT	-
Aktifit VM	Calcined Neuburg Siliceous Earth, d_{50}: 2 µm	Vinyl
Aktifit PF 111	Calcined Neuburg Siliceous Earth, d_{50}: 2 µm	Alkyl
Aktifit AM	Calcined Neuburg Siliceous Earth, d_{50}: 2 µm	Amino
Aktifit PF 115	Calcined Neuburg Siliceous Earth, d_{50}: 2 µm	Special Amino
Aktisil AM	Neuburg Siliceous Earth, d_{50}: 2 µm	Amino
Aktisil Q	Neuburg Siliceous Earth, d_{50}: 4 µm	Methacrylic

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Fig. 2

The grades Aktifit VM, Aktifit PF 111, Aktifit AM and Aktifit PF 115 are based on Calcined Neuburg Siliceous Earth and surface-modified with functional groups, described in Fig. 2.

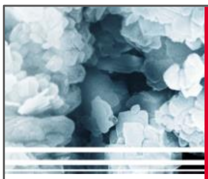
Aktisil AM and Aktisil Q are derived from conventional NSE grades, although the latter product is based on a somewhat coarser Neuburg Siliceous Earth grade.

The employed formulation (Fig. 3) shows the typical setup of a peroxide cured FKM compound with zinc oxide, co-activator and peroxide.

Carbon black N990 and the NSE grades were used in identical loadings of 30 phr. This resulted in a comparable hardness range of 65 to 70 Shore A.

The compounds were prepared on a laboratory mill (Schwabenthan Polymix 150 L). The rubber was given onto the mill at 50 °C and milled to a uniform sheet. The filler was added after the zinc oxide had been worked in. The compound was removed, rolled up and placed on the mill 10 times to ensure good distribution of the components. In order to be able to remove the sheet off the mill, the mill temperature was reduced to 30 °C. The total mixing time was 15 minutes.

Total press-cure was 7 minutes at 177 °C, post-cure was carried out for 2 hours at 232 °C.



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Formulation

		N 990	NSE
Viton GAL-200S	66 % fluorine, 25 MU (ML1+10, 121 °C) terpolymer (HFP+VFD+TFE)	**100**	**100**
Zinkoxyd aktiv	ZnO	**3**	**3**
Diak No. 7	co-activator TAIC	**3**	**3**
Varox DBPH-50	2,5-dimethyl-2,5-di(tertbutylperoxy)-hexane	**2**	**2**
N990	Carbon black	**30**	**-**
NSE	Neuburg Siliceous Earth	**-**	**30**

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Fig. 3

2.2 Tests

The results shown here refer to post-cured specimens.

Tensile tests were carried out and compression set was tested in addition to measuring the hardness.

Media resistance was tested as follows:

• Hot air	94 hours / 230 °C
	504 hours / 210 °C
• Engine oil OS206304	168 hours / 150 °C
• Fuel FAM B (DIN 51604)	70 hours / 23 °C
• Acetic acid (1M, pH 3)	168 hours / 100 °C

The results of all tested compounds and all tests are listed at the end of the report in tabular form.

3 Results

3.1 Rheological properties

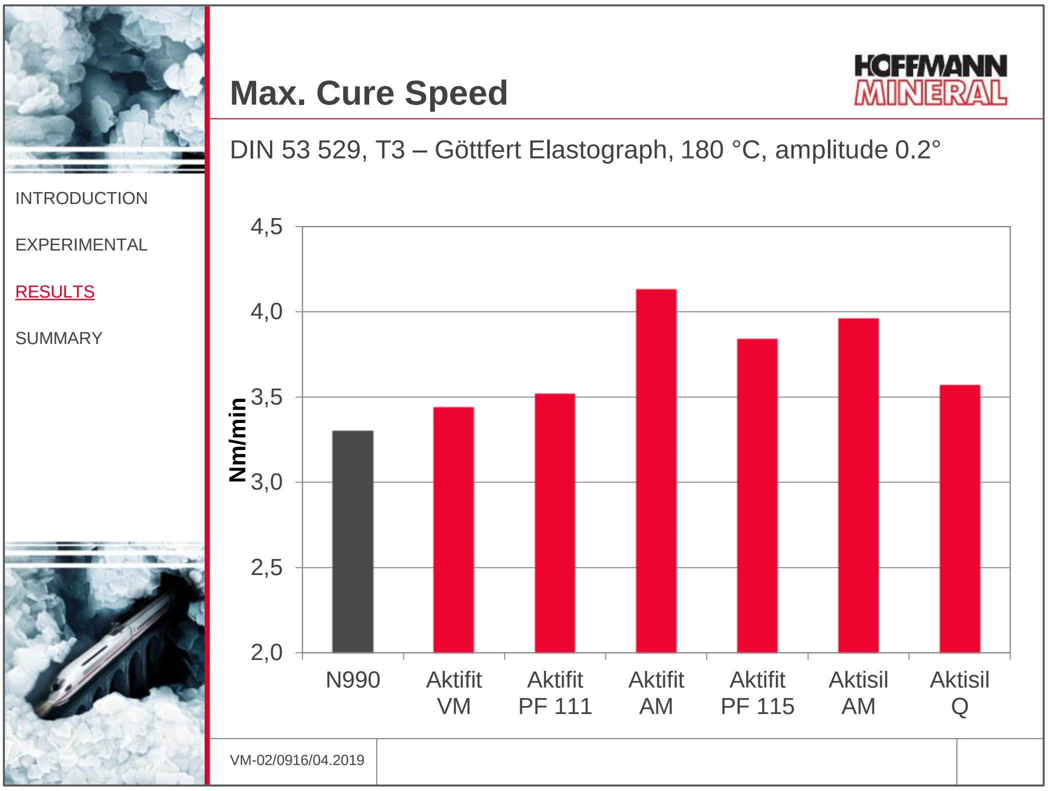


Fig. 4

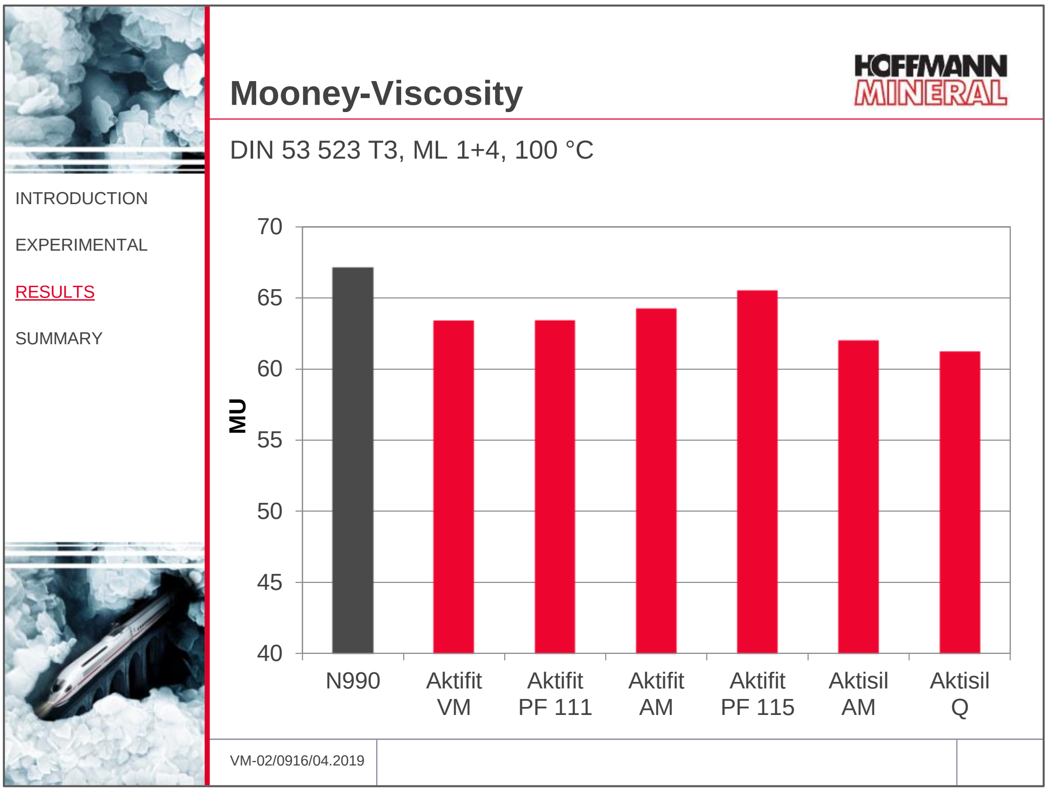


Fig. 5

With all NSE grades the maximum cure speed can be increased compared to N990 (Fig. 4). Te fastest curing can be reached with Aktifit AM, Aktifit PF 115 and Aktisil AM, all treated with amino functional groups.

Viscosity decreases when carbon black is replaced by Neuburg Siliceous Earth (Fig. 5).

3.2 Mechanical properties

As previously mentioned, the hardness of the cured rubbers ranges from 65 to 70 Shore A.

Generally, the modulus at 100 % elongation is increased with Neuburg Siliceous Earth compared to N990 (Fig. 6).

Aktisil AM yields the highest modulus and also a higher tensile strength (Fig. 7) than N990, while the elongation at break (Fig. 8) does not change.

An even higher tensile strength can be achieved with Aktifit VM, with only minor losses in elongation at break.

The other NSE grades are comparable to carbon black in terms of tensile strength and elongation at break.

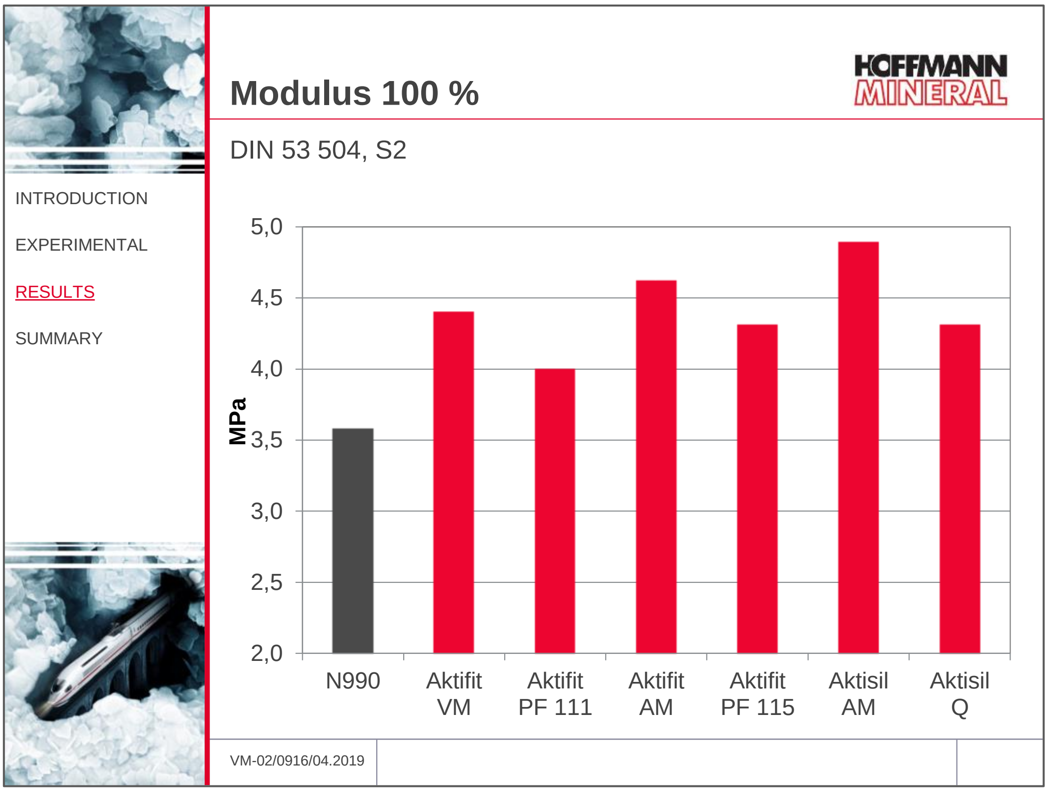
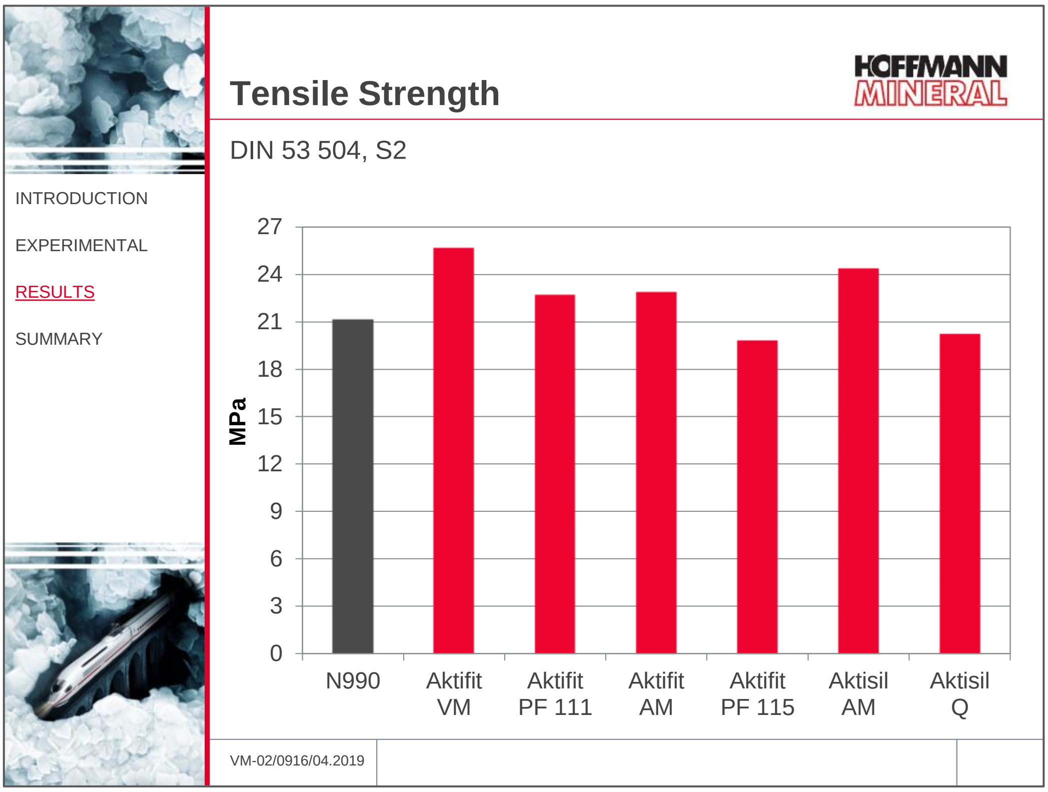


Fig. 6


Tensile Strength
HOFFMANN
MINERAL
DIN 53 504, S2
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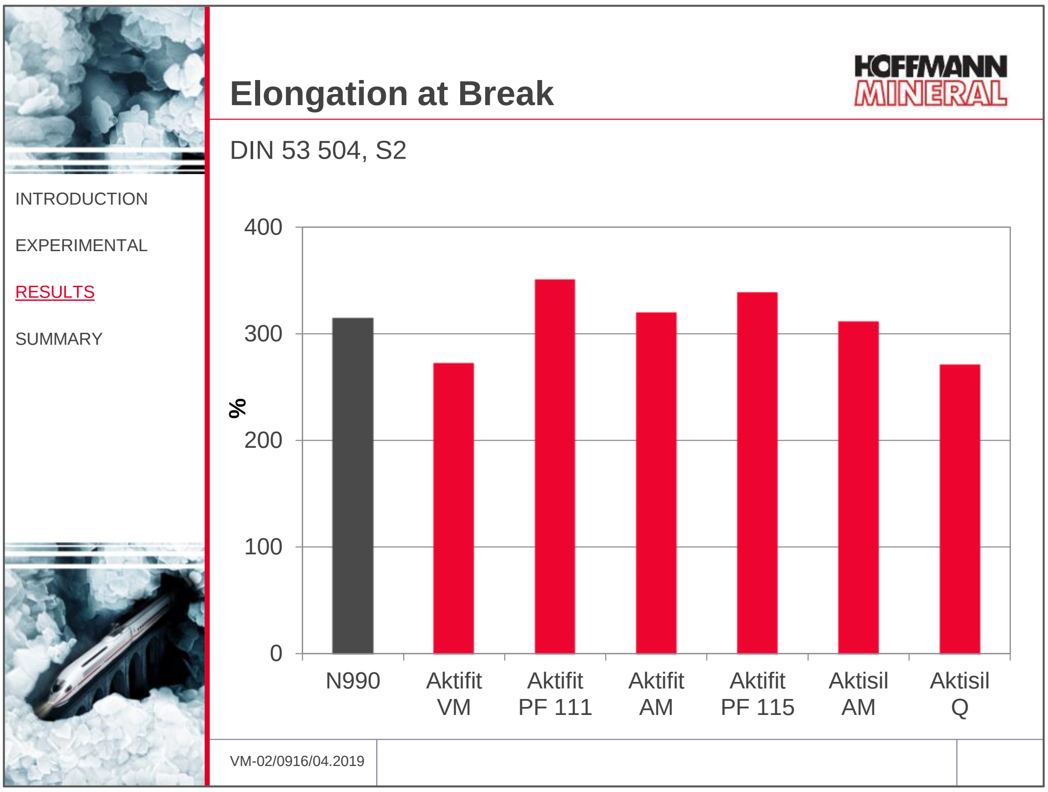
MPa
27
24
21
18
15
12
9
6
3
0
N990
Aktifit VM
Aktifit PF 111
Aktifit AM
Aktifit PF 115
Aktisil AM
Aktisil Q
VM-02/0916/04.2019

Fig. 7


Elongation at Break
HOFFMANN
MINERAL
DIN 53 504, S2
INTRODUCTION
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%
400
300
200
100
0
N990
Aktifit VM
Aktifit PF 111
Aktifit AM
Aktifit PF 115
Aktisil AM
Aktisil Q
VM-02/0916/04.2019

Fig. 8

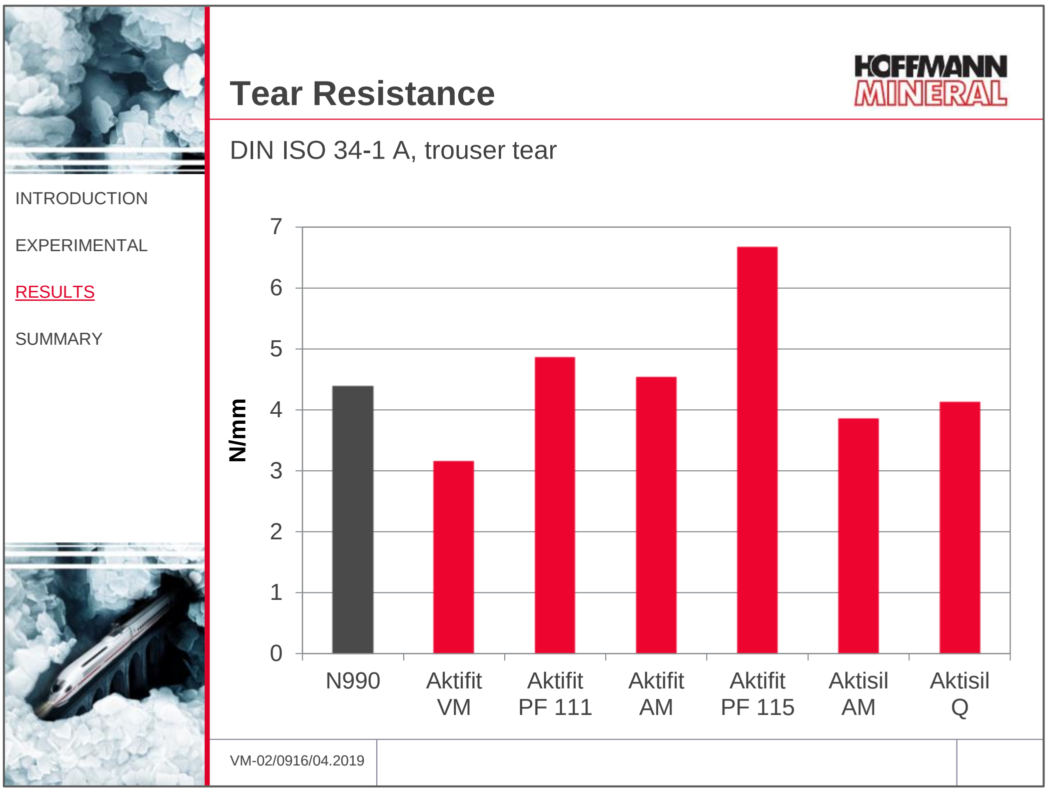


Fig. 9

The highest tear resistance can be achieved with Aktifit PF 115 (Fig. 9).

The other NSE grades (except for Aktifit VM) yield values comparable to N990.

The compression set (ISO, Fig. 10) does not vary in the NSE grades and N990 at a test temperature of 200 °C. When the test temperature is increased to 232 °C, Aktifit VM, Aktifit PF 111 and Aktisil Q remain comparable with carbon black.

A look at the compression set of the specimens that have not been post-cured (Fig. 11) reveals the benefit of Aktisil Q known from other studies, which results in an improvement compared to N990 at 100 °C as well as at 232 °C.

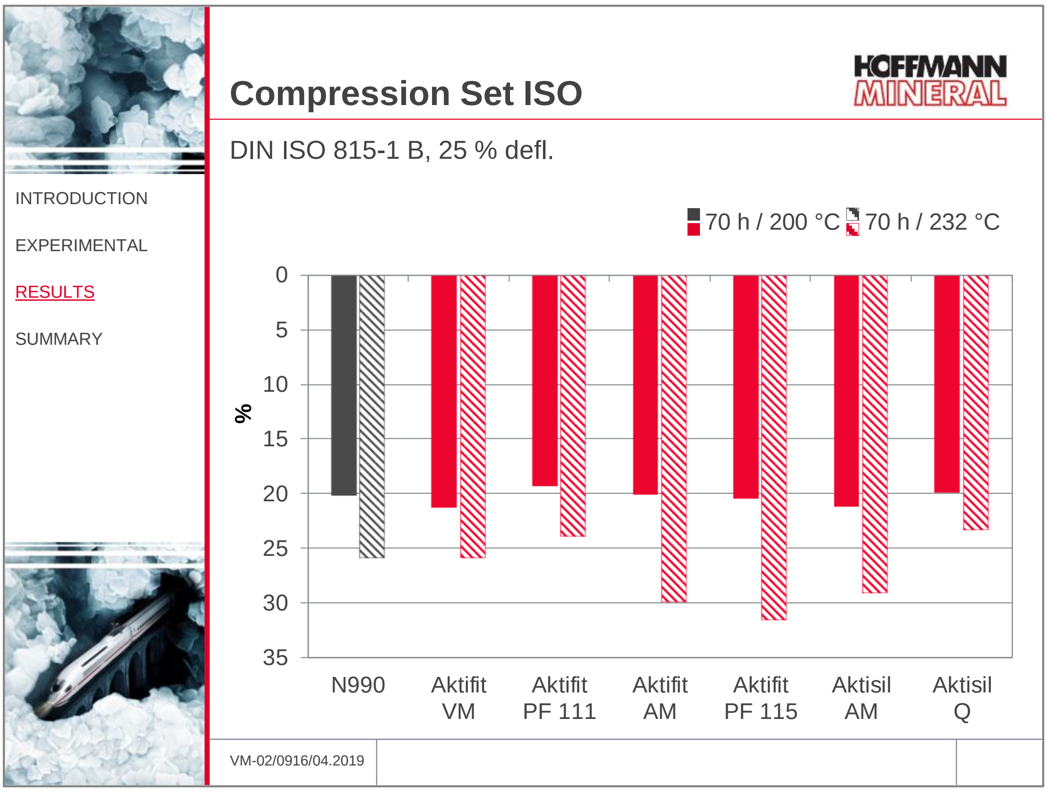


Fig. 10

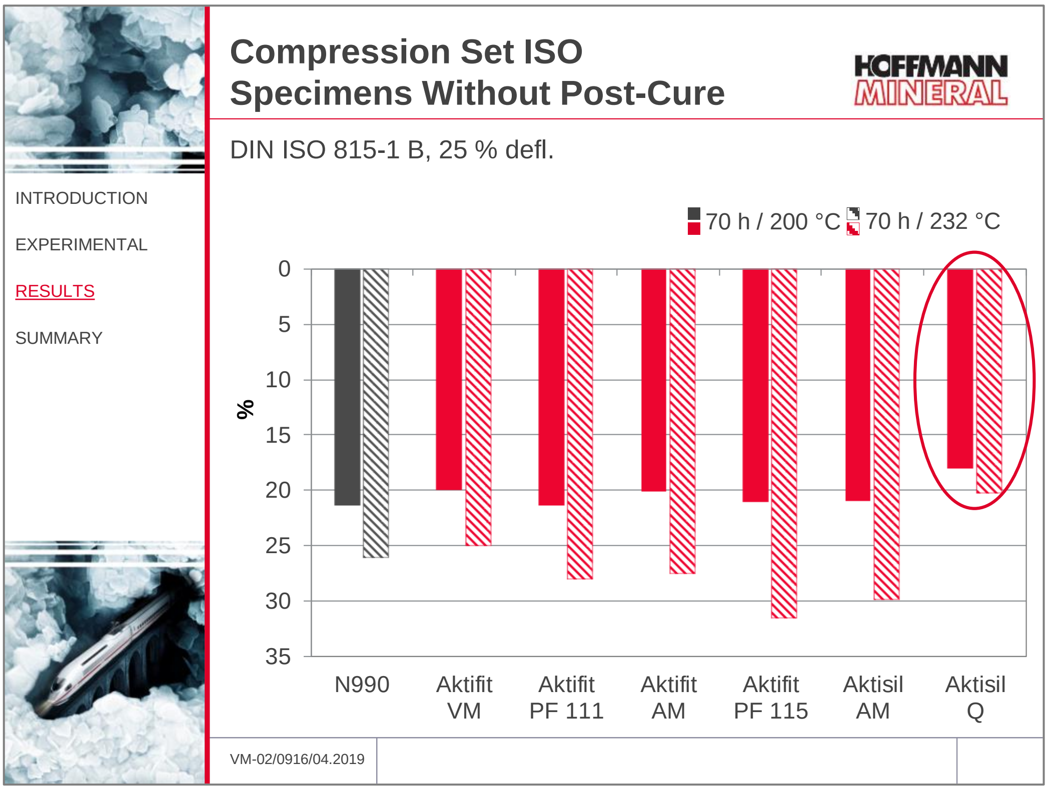


Fig. 11

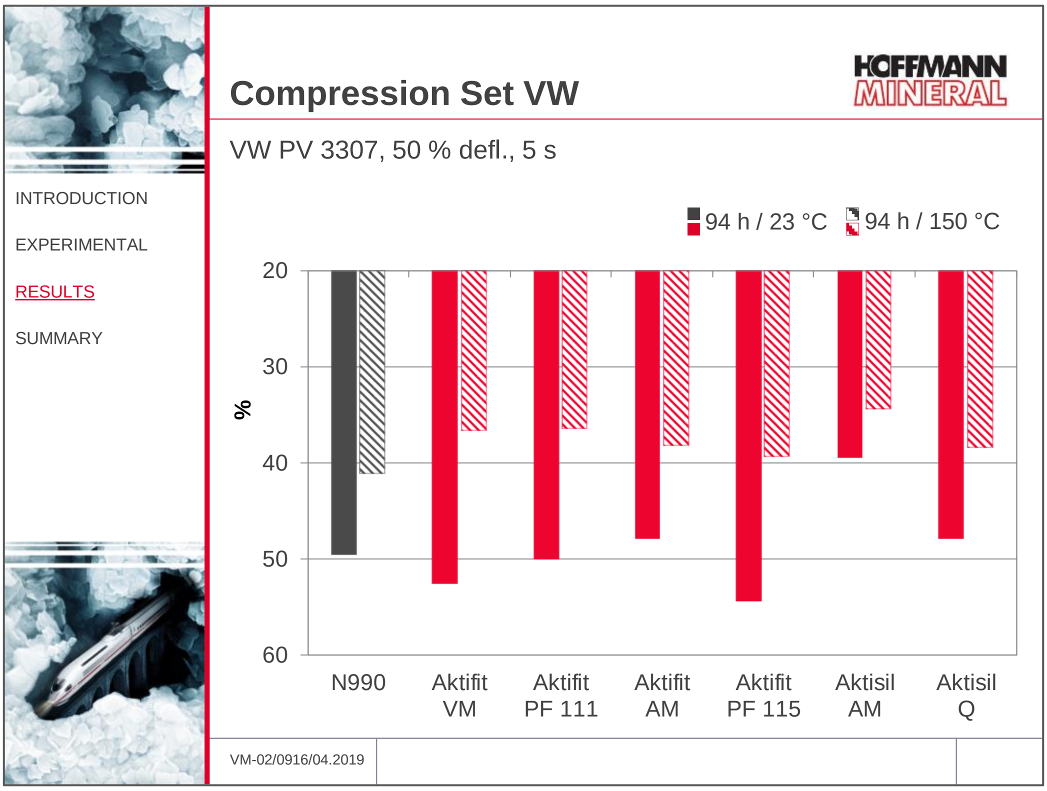


Fig. 12

To improve the compression set according to the VW test standard (Fig. 12) it is advisable to use Aktisil AM, which achieves significant reduction of the value at room temperature as well as at increased temperature compared to carbon black.

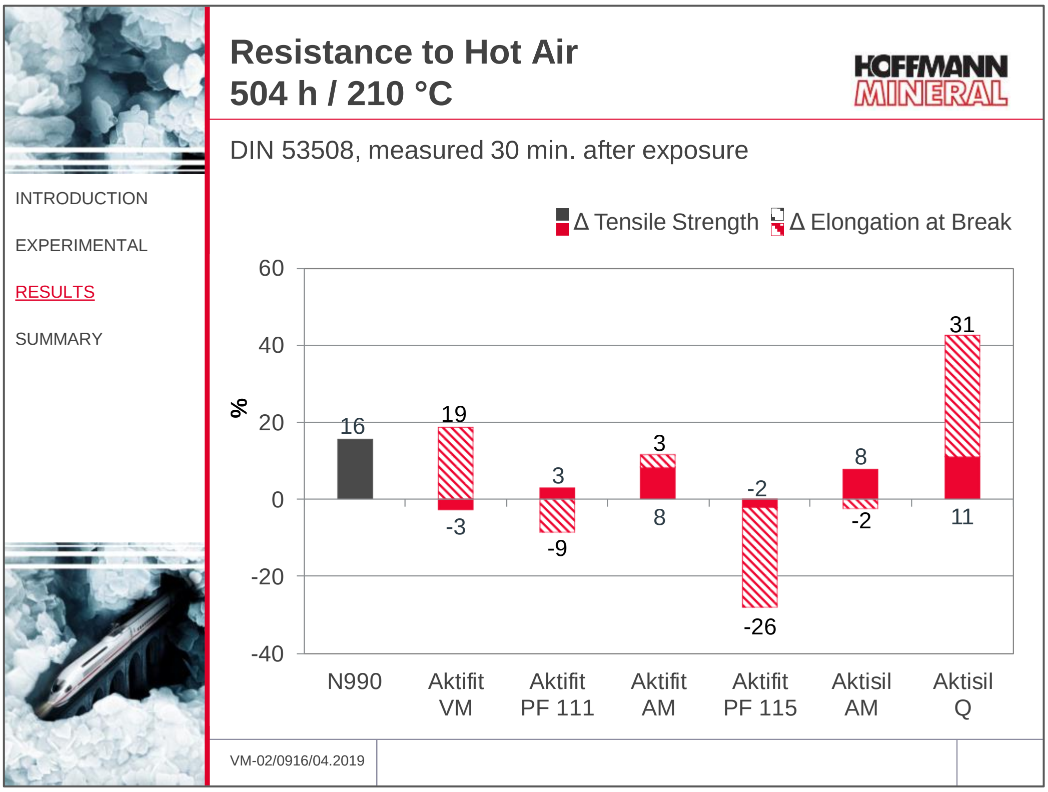
3.3 Hot air resistance

Resistance to hot air is assessed on the basis of changes in tensile strength and elongation at break.

The long-term aging test (504 h) shows that the resistance to hot air with Aktisil Q is higher than with carbon black at 210 °C, while Aktifit VM, Aktifit PF 111, Aktifit AM and Aktisil AM are on a comparable level (Fig. 13).

The short-term aging (94 h) at increased temperature (230 °C) – Fig. 14 – shows that not only Aktisil Q but also Aktifit VM helps to improve resistance to hot air.

The changes in the modulus at 100 % elongation after hot air aging (Fig. 15) also confirm these results.


Resistance to Hot Air
504 h / 210 °C
HOFFMANN MINERAL
DIN 53508, measured 30 min. after exposure
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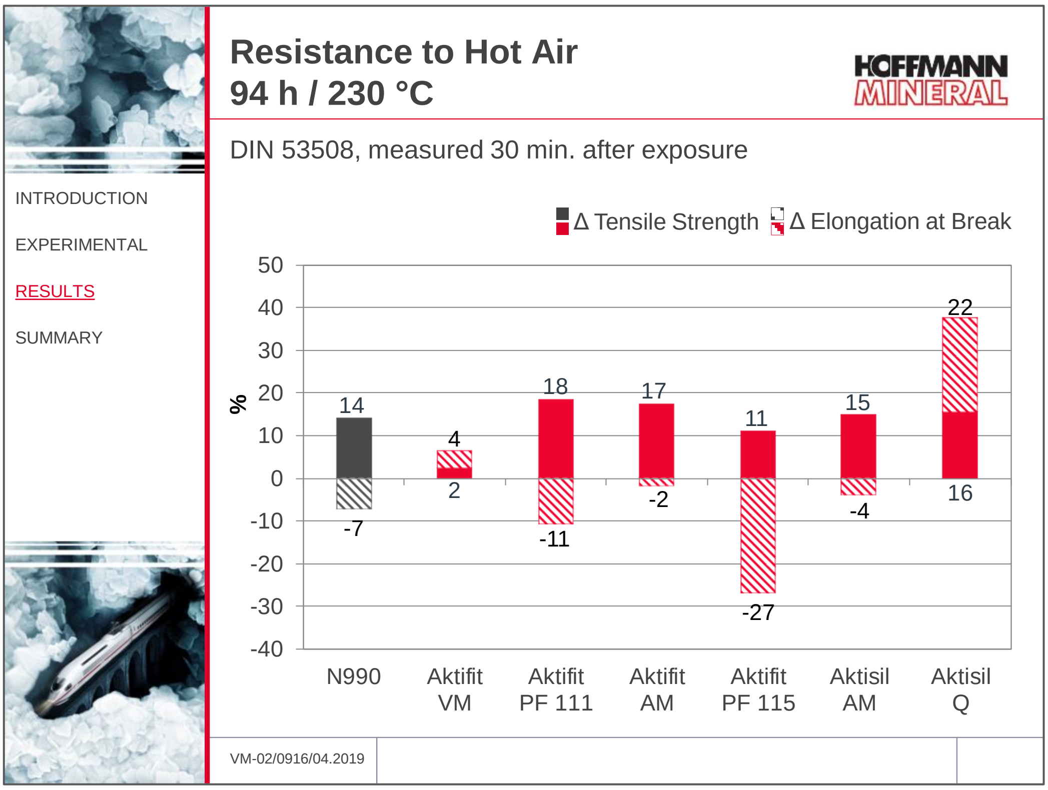
Δ Tensile Strength
Δ Elongation at Break
%
60
40
20
0
-20
-40
16
19
-3
3
-9
3
8
-2
-26
8
-2
31
11
N990
Aktifit VM
Aktifit PF 111
Aktifit AM
Aktifit PF 115
Aktisil AM
Aktisil Q
VM-02/0916/04.2019
Fig. 13


Resistance to Hot Air
94 h / 230 °C
HOFFMANN MINERAL
DIN 53508, measured 30 min. after exposure
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SUMMARY
Δ Tensile Strength
Δ Elongation at Break
%
50
40
30
20
10
0
-10
-20
-30
-40
14
-7
4
2
18
-11
17
-2
11
-27
15
-4
22
16
N990
Aktifit VM
Aktifit PF 111
Aktifit AM
Aktifit PF 115
Aktisil AM
Aktisil Q
VM-02/0916/04.2019
Fig. 14

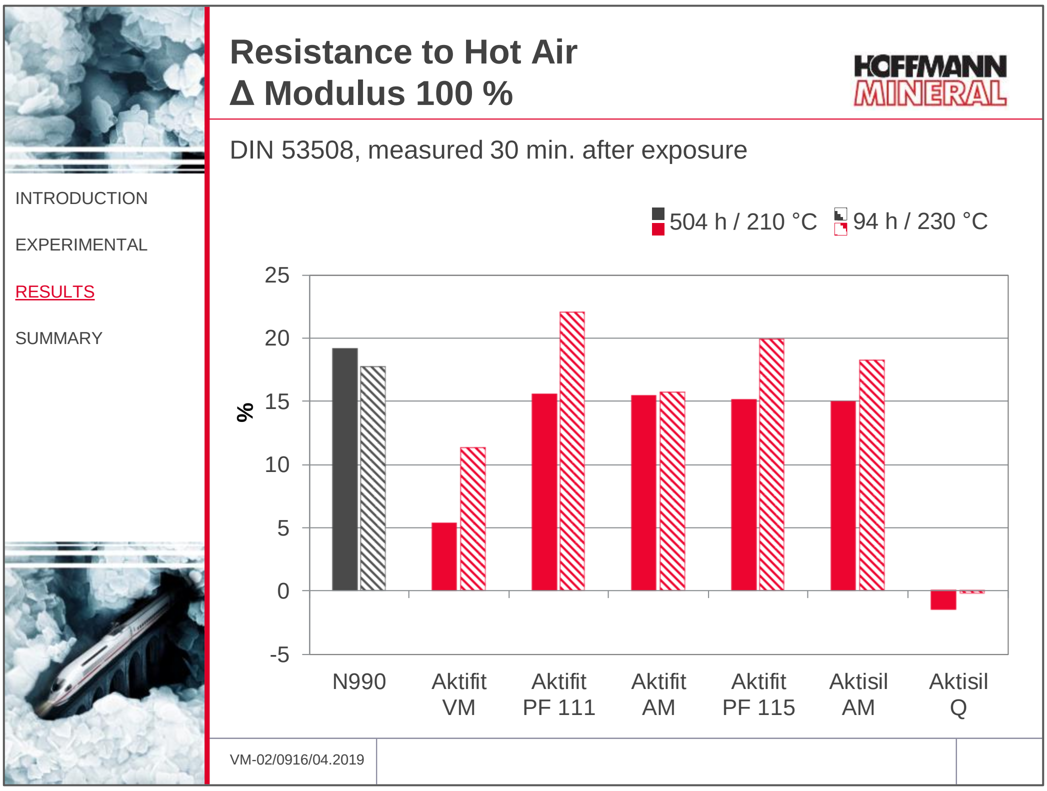
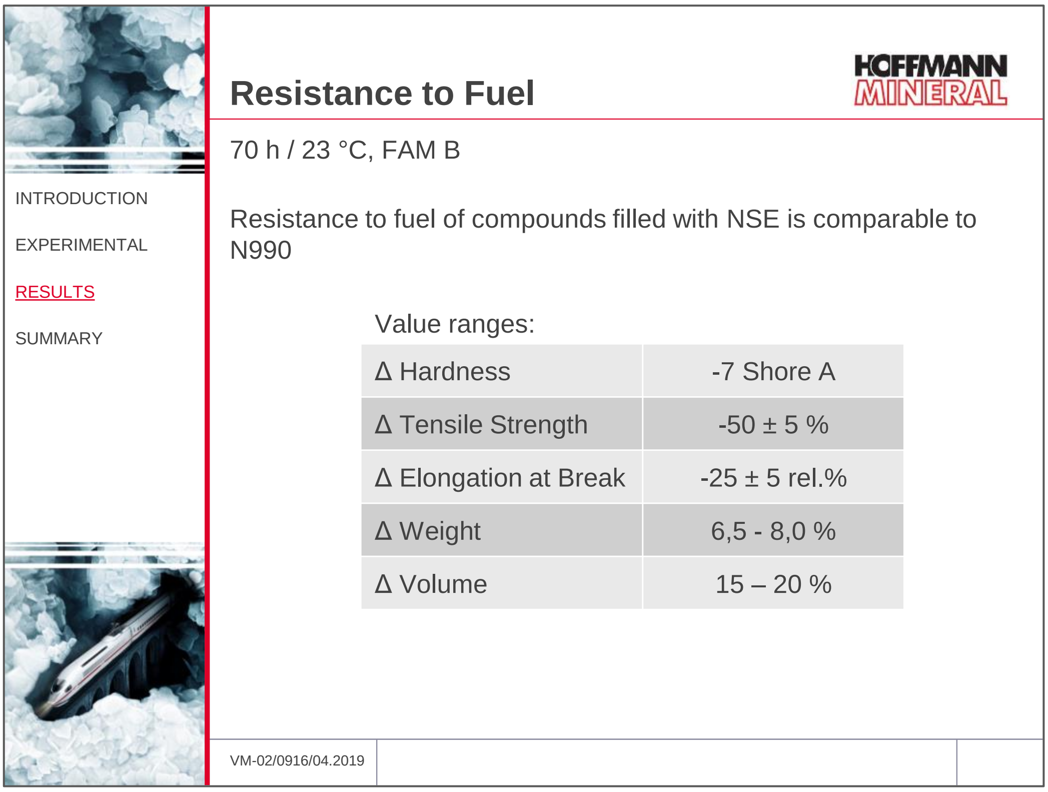


Fig. 15

3.4 Fuel resistance

Since the vulcanized rubbers filled with NSE have a resistance to methanolic fuel (FAM B, 70 h / 23 °C) comparable to N990, the single values are not shown here, but only the values' ranges are listed.



Δ Hardness	-7 Shore A
Δ Tensile Strength	-50 ± 5 %
Δ Elongation at Break	-25 ± 5 rel.%
Δ Weight	6,5 - 8,0 %
Δ Volume	15 – 20 %

Fig. 16

3.5 Oil resistance

Oil resistance was tested with engine oil OS206304 at 150 °C for 168 hours.
As Fig. 17 shows, Aktifit PF 111, Aktifit AM, Aktisil AM and Aktisil Q result in improved oil resistance compared to carbon black. The reduced changes in the mechanical properties of elongation at break and tensile strength result in increased value levels for e.g. Aktisil Q after storage in oil compared to N990 (Fig. 18).

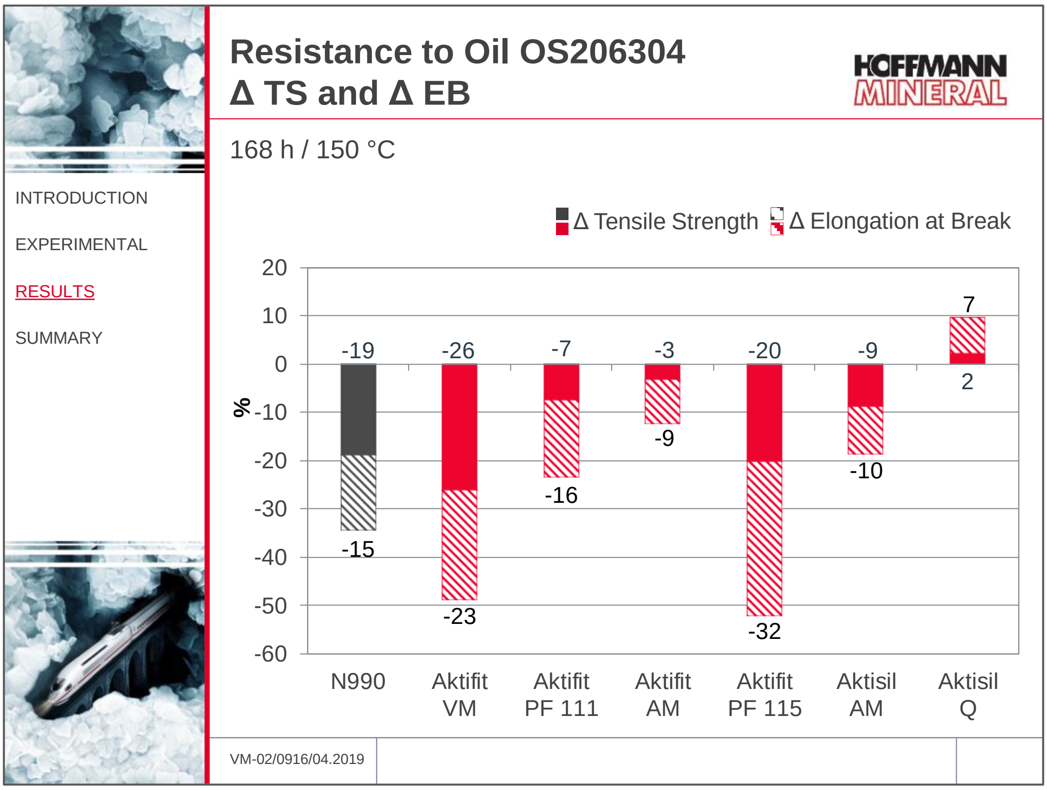


Fig. 17

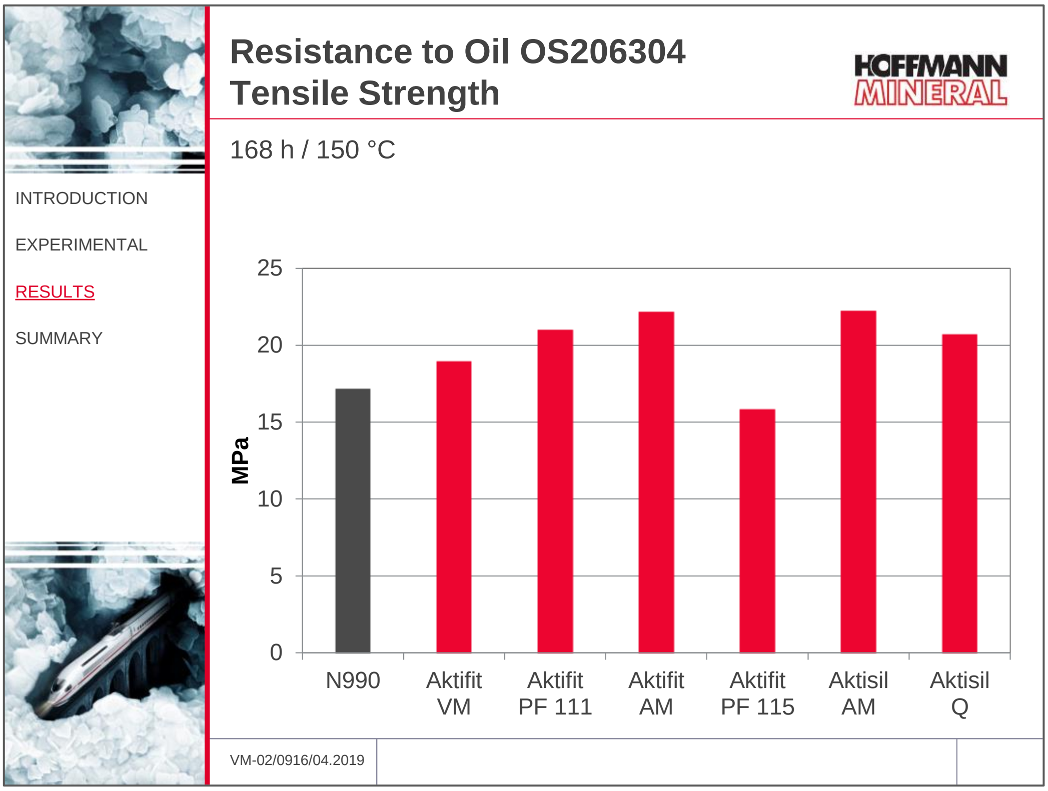


Fig. 18

3.6 Acid resistance

The acid resistance test was carried out with acetic acid (1M, pH3) at 100 °C for 168 hours. The purpose of this test is to simulate blow-by resistance.

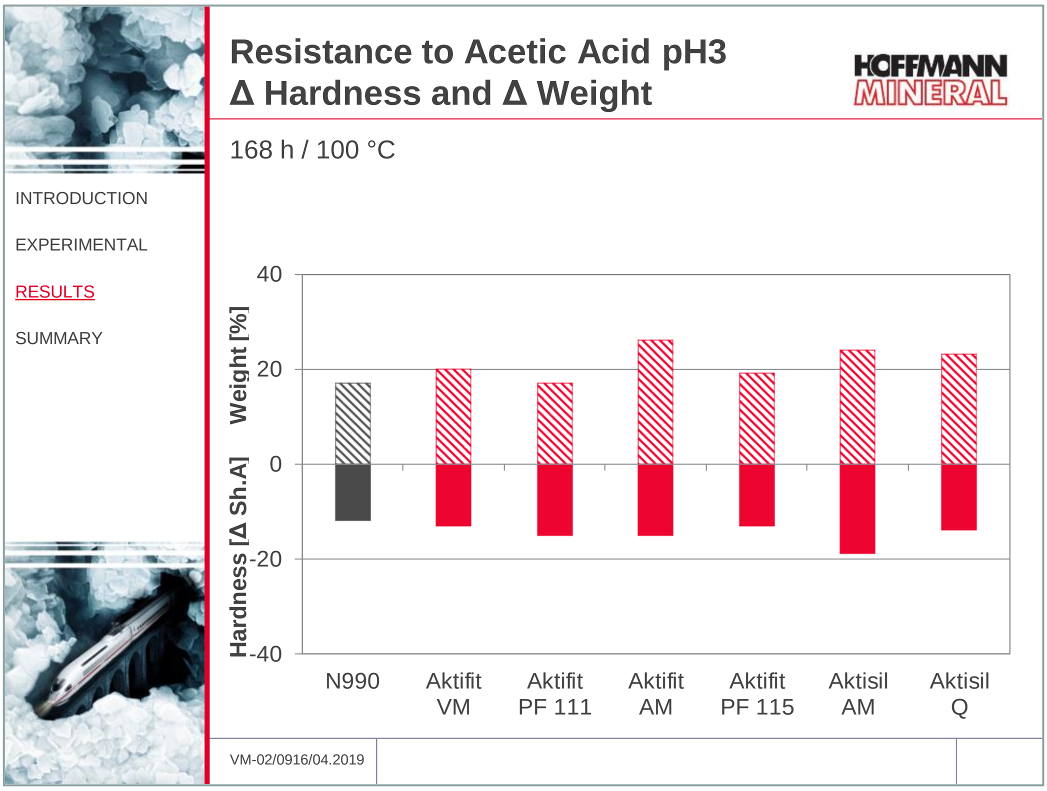


Fig. 19

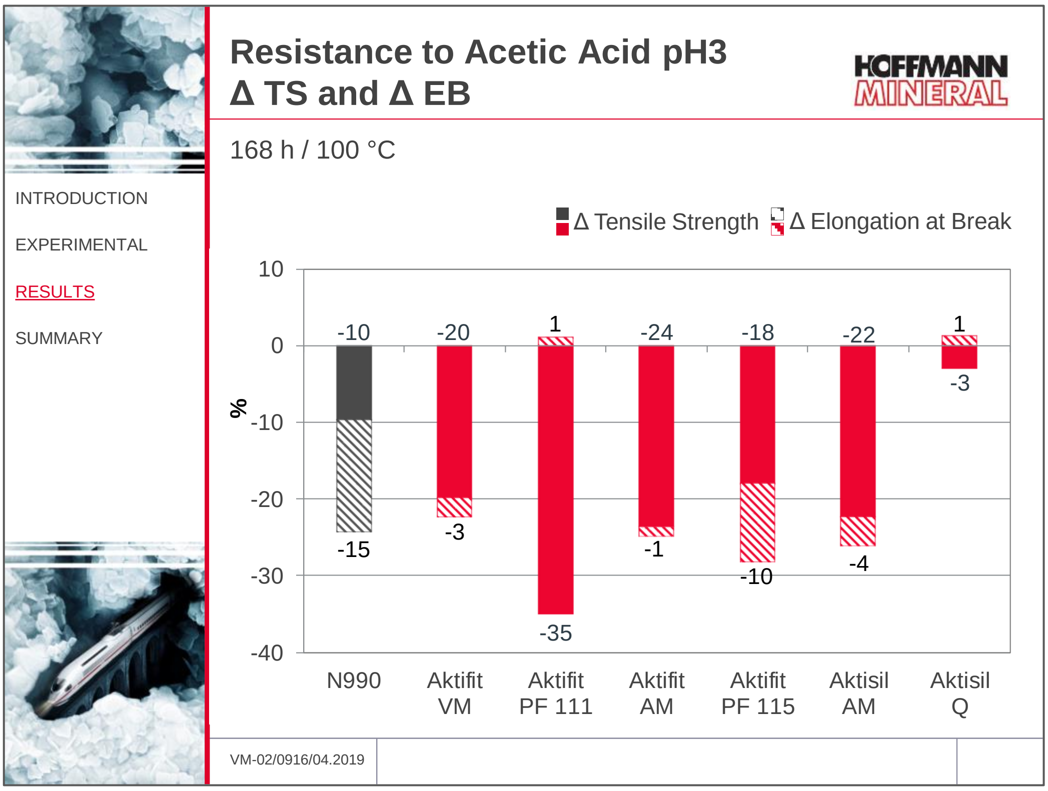


Fig. 20

While the weight and hardness changes after storage in acetic acid do not show any significant differences between the NSE grades and N990, particularly Aktisil Q does not result in any change in tensile strength or elongation at break. The other NSE grades result in a resistance to acetic acid comparable with carbon black. The absolute values shown in Fig. 21 result from the changes in tensile strength.

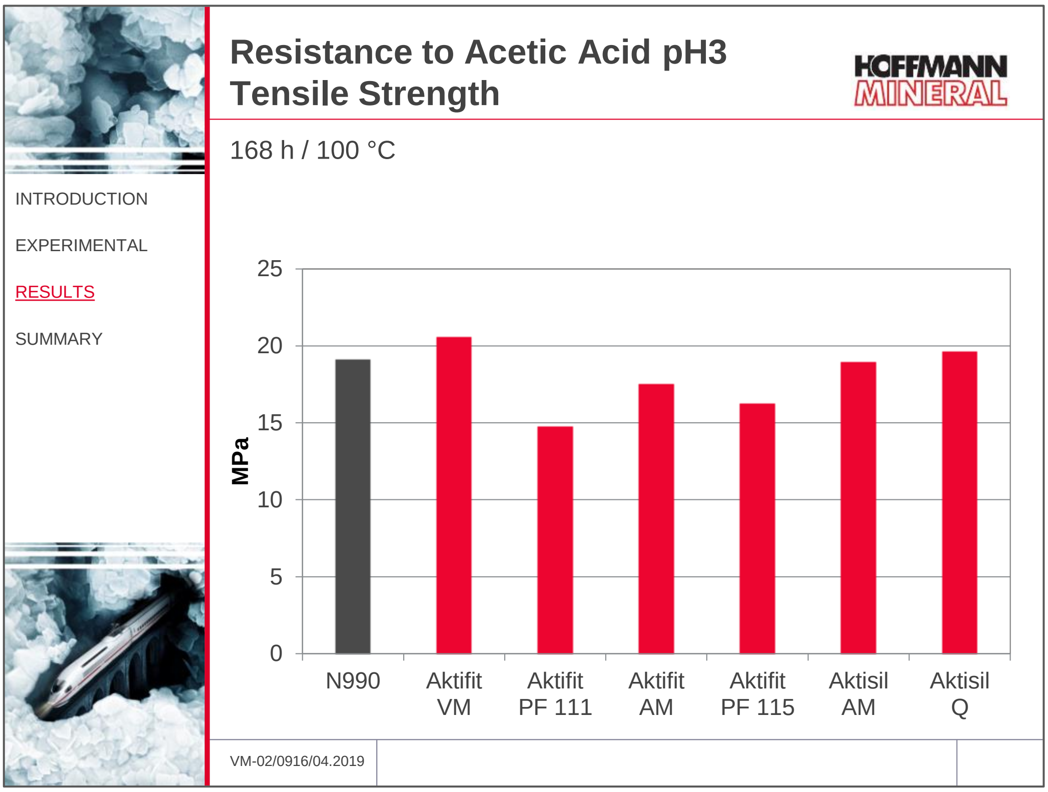


Fig. 21

As mentioned in the beginning, the typically used non-black fillers wollastonite or barium sulfate result in an inferior resistance to acetic acid compared to carbon black. On the one hand, they distinctly swell. On the other hand, there is a severe decrease in tensile strengths, for example, as can be seen in Fig. 22.

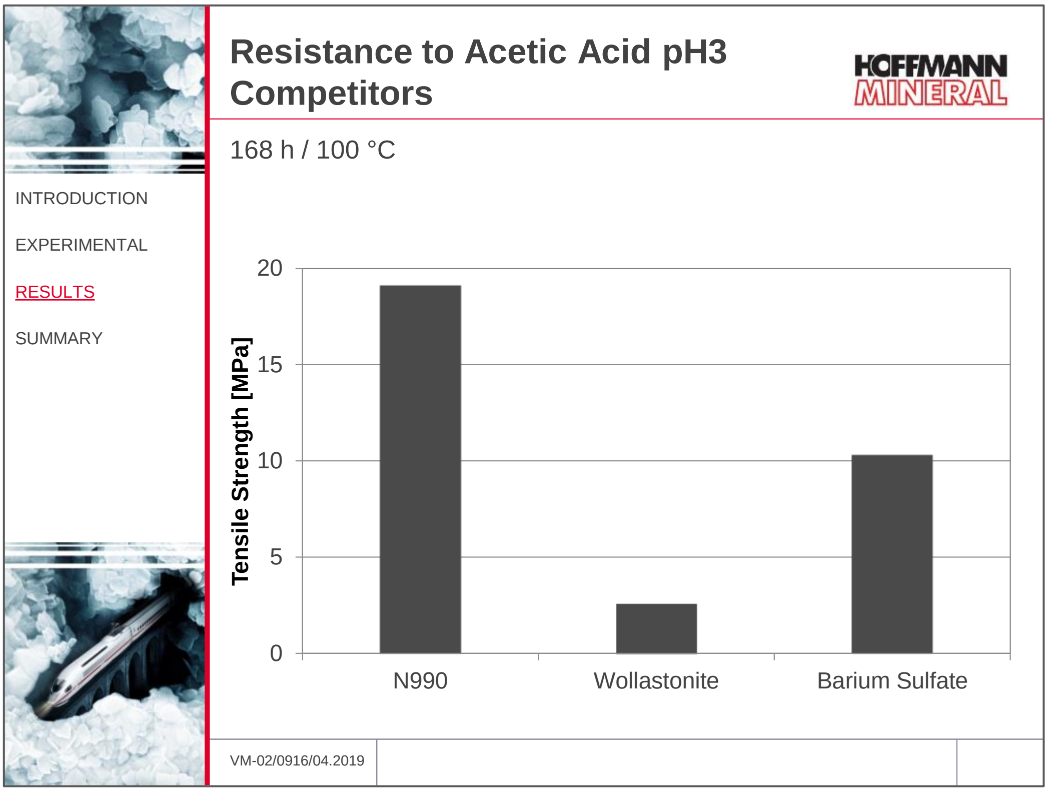


Fig. 22

4 Summary

This study shows that it is possible to obtain peroxide cured FKM compounds with Neuburg Siliceous Earth which are comparable with ones filled with carbon black or even improve some properties. Thus the vulcanized rubbers are tintable without sacrificing good acid resistance.

The following table provides an overview of the property profiles of the tested NSE grades to enable a selection of the most suitable product for the respective application.

Evaluation

NSE vs. N990

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N 990 65 - 70 Shore A	Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q
Cure speed	=	=	+	+	+	=
Viscosity	+	+	+	+	+	+
Tensile strength	+	=	=	=	+	=
Elongation at break		+	=	=	=	
Modulus100 %	+	+	+	+	+	+
Tear resistance		=	=	+	=	=
CS ISO 200 °C	=	=	=	=	=	=
CS ISO 200 °C, no post-cure	=	=	=	=	=	+
CS ISO 232 °C	=	=				=
CS ISO 232 °C, no post-cure	=					+
CS VW 23 °C	=	=	=		+	=
CS VW 150 °C	+	+	=	=	+	=
Hot air resistance 210 °C	=	=	=		=	+
Hot air resistance 230 °C	+	=	=		=	+
Fuel resistance	=	=	=	=	=	=
Oil resistance		+	+		+	+
Resistance to acetic acid	=	=	=	=	=	+

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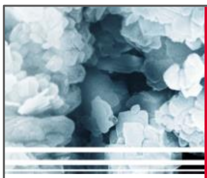
Fig. 23

Fig. 23 shows which NSE grade either improves (+) the respective property compared to N990 or equals (=) it. The + signs highlighted in red mean that this product achieves the best value among the tested NSE grades for the corresponding property.

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.



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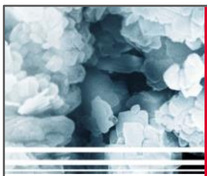
		Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q	N 990
Rheology								
Mooney Viscosity, ML Min., 100 °C	MU	63	63	64	66	62	61	67
Rotorless Curemeter M_{min} 177 °C	Nm	0.04	0.04	0.04	0.05	0.04	0.04	0.04
Rotorless Curemeter V_{max} 177 °C	Nm/min.	3.4	3.5	4.1	3.8	4.0	3.6	3.3
Rotorless Curemeter t_{90} 177 °C	min.	0.8	0.9	0.8	0.8	0.8	0.8	0.8
Mechanical properties – Cure conditions 7 min. / 177 °C, no post-cure								
Hardness	Sh. A	64	65	65	65	64	63	65
Tensile Strength	MPa	22	16	18	17	19	16	17
Modulus 50 %	MPa	1.70	1.68	1.68	1.72	1.83	1.60	1.58
Modulus100 %	MPa	3.9	3.5	3.7	3.6	4.2	3.6	3.2
Elongation at Break	%	278	336	364	395	312	257	330
Tear Resistance	N/mm	3.1	5.4	4.7	6.0	4.5	4.0	4.6
CS ISO 70 h / 200 °C, 25 % defl.	%	20	21	20	21	21	18	21
CS ISO 70 h / 232 °C, 25 % defl.	%	25	28	28	32	30	20	26



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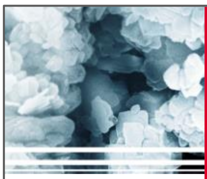
		Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q	N990
Mechanical properties – Cure conditions 7 min. / 177 °C, post-cure 2 h / 232 °C								
Hardness	Sh. A	65	66	66	65	66	65	66
Tensile Strength	MPa	26	23	23	20	24	20	21
Modulus 50 %	MPa	1.75	1.76	1.83	1.84	1.92	1.72	1.66
Modulus100 %	MPa	4.4	4.0	4.6	4.3	4.9	4.3	3.6
Elongation at Break	%	272	351	320	339	311	271	314
Tear Resistance	N/mm	3.2	4.9	4.5	6.7	3.9	4.1	4.4
CS ISO 70 h / 200 °C, 25 % defl.	%	21	19	20	20	21	20	20
CS ISO 70 h / 232 °C, 25 % defl.	%	26	24	30	32	29	23	26
CS VW PV3307 94 h / 23 °C, 50 % defl.	%	53	50	48	54	39	48	50
CS VW PV3307 94 h / 150 °C, 50 % defl.	%	37	36	38	39	34	38	41
Abrasion Loss	mm³	60	72	67	74	71	73	53



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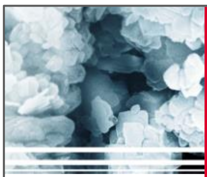
		Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q	N 990
Hot air aging, 504 h / 210 °C, measured 30 min. after exposure								
Hardness	Sh. A	68	68	68	69	69	67	69
Tensile Strength	MPa	25	23	25	19	26	22	24
Elongation at Break	%	323	320	330	252	304	356	314
Δ Hardness	Sh. A	+3	+2	+2	+4	+3	+2	+3
Δ Tensile Strength	%	-3	+3	+8	-2	+8	+11	+16
Δ Elongation at Break	rel.%	+19	-9	+3	-26	-2	+31	0
Hot air aging, 94 h / 230 °C, measured 30 min. after exposure								
Hardness	Sh. A	67	68	67	68	68	65	69
Tensile Strength	MPa	26	27	27	22	28	23	24
Elongation at Break	%	283	313	314	247	299	331	292
Δ Hardness	Sh. A	+2	+2	+1	+3	+2	0	+3
Δ Tensile Strength	%	+2	+18	+17	+11	+15	+16	+14
Δ Elongation at Break	rel.%	+4	-11	-2	-27	-4	+22	-7



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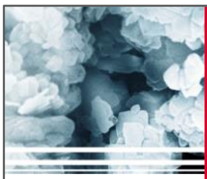
		Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q	N 990
Exposure to fuel FAM B, 70 h / 23 °C								
Hardness	Sh. A	58	59	58	58	58	58	59
Tensile Strength	MPa	14	11	12	10	11	10	12
Elongation at Break	%	220	255	246	268	238	206	231
Δ Hardness	Sh. A	-7	-7	-8	-7	-8	-7	-7
Δ Tensile Strength	%	-46	-51	-48	-50	-53	-49	-44
Δ Elongation at Break	rel.%	-19	-27	-23	-21	-24	-24	-26
Δ Weight	%	+7.3	+7.8	+8.0	+8.0	+6.8	+8.0	+6.7
Δ Volume	%	+18	+19	+19	+19	+17	+19	+15
Exposure to engine oil OS206304, 168 h / 150 °C								
Hardness	Sh. A	65	65	65	66	65	64	65
Tensile Strength	MPa	19	21	22	16	22	21	17
Elongation at Break	%	210	295	291	231	281	291	266
Δ Hardness	Sh. A	0	-1	-1	+1	-1	-1	-1
Δ Tensile Strength	%	-26	-7	-3	-20	-9	+2	-19
Δ Elongation at Break	rel.%	-23	-16	-9	-32	-10	+7	-15
Δ Weight	%	+0.8	+0.7	+0.6	+0.6	+0.7	+0.6	+0.8
Δ Volume	%	+1.4	+1.2	+0.9	+1.4	+1.2	+0.7	+1.4



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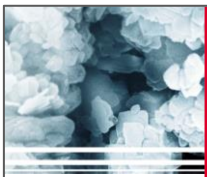
		Aktifit VM	Aktifit PF 111	Aktifit AM	Aktifit PF 115	Aktisil AM	Aktisil Q	N 990
Exposure to acetic acid pH3, 168 h / 100 °C								
Hardness	Sh. A	52	51	51	52	47	51	54
Tensile Strength	MPa	21	15	17	16	19	20	19
Elongation at Break	%	265	355	316	304	300	274	268
Δ Hardness	Sh. A	-13	-15	-15	-13	-19	-14	-12
Δ Tensile Strength	%	-20	-35	-24	-18	-22	-3	-10
Δ Elongation at Break	rel.%	-3	+1	-1	-10	-4	+1	-15
Δ Weight	%	+20	+17	+26	+19	+24	+23	+17
Δ Volume	%	+37	+34	+50	+37	+47	+43	+30

VM-02/0916/04.2019



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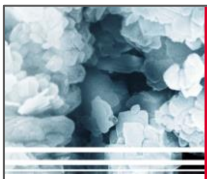
Loading 30 phr (49 phr for $BaSO_4$)		Wollastonite AST	Wollastonite EST	Barium Sulfate	N 990
Rheology					
Mooney Viscosity, ML Min., 100 °C	MU	63	59	66	67
Rotorless Curemeter M_{min} 177 °C	Nm	0.03	0.03	0.04	0.04
Rotorless Curemeter V_{max} 177 °C	Nm/min.	3.4	3.1	3.4	3.3
Rotorless Curemeter t_{90} 177 °C	min.	0.9	0.9	0.9	0.8
Mechanical properties – Cure conditions 7 min. / 177 °C, no post-cure					
Hardness	Sh. A	61	63	61	65
Tensile Strength	MPa	18	16	15	17
Modulus 50 %	MPa	1.9	1.6	1.4	1.58
Modulus100 %	MPa	4.2	3.2	2.1	3.2
Elongation at Break	%	397	393	421	330
Tear Resistance	N/mm	6.0	5.9	4.7	4.6
CS ISO 70 h / 200 °C, 25 % defl.	%	24	21	22	21
CS ISO 70 h / 232 °C, 25 % defl.	%	35	28	31	26

VM-02/0916/04.2019



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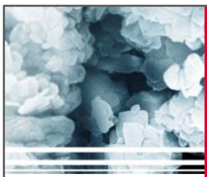
Loading 30 phr (49 phr for $BaSO_4$)		Wollastonite AST	Wollastonite EST	Barium Sulfate	N 990
Mechanical properties – Cure conditions 7 min. / 177 °C, post-cure 2 h / 232 °C					
Hardness	Sh. A	67	64	67	66
Tensile Strength	MPa	19	20	16	21
Modulus 50 %	MPa	1.7	1.7	1.4	1.7
Modulus100 %	MPa	3.9	3.5	2.4	3.6
Elongation at Break	%	337	399	407	314
Tear Resistance	N/mm	6.2	6.9	6.1	4.4
CS ISO 70 h / 200 °C, 25 % defl.	%	22	18	22	20
CS ISO 70 h / 232 °C, 25 % defl.	%	29	24	28	26
CS VW PV3307 94 h / 23 °C, 50 % defl.	%	51	51	49	50
CS VW PV3307 94 h / 150 °C, 50 % defl.	%	35	36	36	41
Abrasion Loss	mm³	104	114	124	53

VM-02/0916/04.2019



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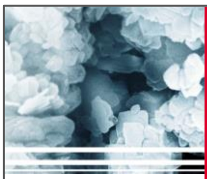
Loading 30 phr (49 phr for $BaSO_4$)		Wollastonite AST	Wollastonite EST	Barium Sulfate	N 990
Hot air aging, 504 h / 210 °C, measured 30 min. after exposure					
Hardness	Sh. A	64	64	65	69
Tensile Strength	MPa	20	21	24	24
Elongation at Break	%	331	390	372	314
Δ Hardness	Sh. A	-3	0	-2	+3
Δ Tensile Strength	%	+6	+3	+43	+16
Δ Elongation at Break	rel.%	-2	-2	-9	0
Hot air aging, 94 h / 230 °C, measured 30 min. after exposure					
Hardness	Sh. A	65	65	65	69
Tensile Strength	MPa	22	22	25	24
Elongation at Break	%	325	335	353	292
Δ Hardness	Sh. A	-2	1	-2	+3
Δ Tensile Strength	%	+18	+8	+51	+14
Δ Elongation at Break	rel.%	-4	-16	-13	-7

VM-02/0916/04.2019



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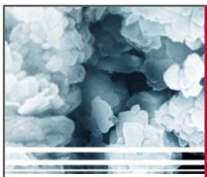
Loading 30 phr (49 phr for $BaSO_4$)		Wollastonite AST	Wollastonite EST	Barium Sulfate	N 990
Exposure to fuel FAM B, 70 h / 23 °C					
Hardness	Sh. A	56	55	52	59
Tensile Strength	MPa	8.6	7.0	7.7	12
Elongation at Break	%	241	261	329	231
Δ Hardness	Sh. A	-11	-9	-15	-7
Δ Tensile Strength	%	-53	-66	-53	-44
Δ Elongation at Break	rel.%	-28	-35	-19	-26
Δ Weight	%	+7,6	+7,2	+7,4	+6,7
Δ Volume	%	+19	+18	+20	+15
Exposure to engine oil OS206304, 168 h / 150 °C					
Hardness	Sh. A	62	62	62	65
Tensile Strength	MPa	17	13	11	17
Elongation at Break	%	286	297	340	266
Δ Hardness	Sh. A	-5	-2	-5	-1
Δ Tensile Strength	%	-7	-35	-34	-19
Δ Elongation at Break	rel.%	-15	-26	-17	-15
Δ Weight	%	+0.6	+0.6	+0.5	+0.8
Δ Volume	%	+1.2	+1.1	+1.3	+1.4



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Loading 30 phr (49 phr for $BaSO_4$)		Wollastonite AST	Wollastonite EST	Barium Sulfate	N 990
Exposure to acetic acid pH3, 168 h / 100 °C					
Hardness	Sh. A	37	non-determinable	35	54
Tensile Strength	MPa	2.6	2.8	10	19
Elongation at Break	%	54	84	252	268
Δ Hardness	Sh. A	-30	non-determinable	-32	-12
Δ Tensile Strength	%	-86	-86	-37	-10
Δ Elongation at Break	rel.%	-84	-79	-38	-15
Δ Weight	%	+288	+227	+55	+17
Δ Volume	%	+593	+499	+121	+30