

# Calcined Neuburg Siliceous Earth in white building profiles

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# 1 Introduction

Until recently, in white building profiles calcined clays are the predominant fillers, as they give good extrusion and mechanical properties. They are also very suitable for this application because they impart high brightness and color neutrality.

Neuburg Siliceous Earth represents an excellent alternative to calcined clay with regard to mechanical properties and extrusion performance. In view of its yellowish tint, however, it could only be used in grey or black profiles, but rather presented problems for color neutrality in very light-colored or white sections.

As Hoffmann Mineral constantly endeavours to enlarge its product portfolio in order to offer customers an increased variety of solutions, there have been launched now also calcined versions of Neuburg Siliceous Earth - Silfit Z 91 and Aktifit VM.

Silfit Z 91 is a naturally occurring conglomerate of amorphous and cryptocrystalline silica with lamellar kaolinite, which has been subjected to a heat treatment. The components and the thermal process lead to a product that offers special performance benefits as a functional filler.

Aktifit VM is an activated Silfit Z 91, produced by modifying the surface with a special vinyl functional group.

In the present study, the effects of the Calcined Neuburg Siliceous Earth grades were evaluated in a grey-white building profile compound with respect to mechanical properties as well as color and extrusion performance. The assessment of the results was done relative to the standard for extruded sealing profiles RAL GZ 716/1, elastomer class B II and RAL 9002 for the grey-white color.

In the subsection "Comparison of fillers" the above-mentioned properties are compared with standard Neuburg Siliceous Earth grades and a calcined clay. In addition, the influence of the base polymer of the formulation is followed.

The polymer used, along with the peroxide concentration, plays a role also in section 4 "Optimized formulation - performance potential of Aktifit VM". Furthermore, the loading of Aktifit VM was gradually increased, as this will affect the extrusion properties as well as the compound costs.

## 2 Experimental

### 2.1 Mineral fillers and compound preparation

| INTRODUCTION<br><u>EXPERIMENTAL</u><br>RESULTS<br>SUMMARY | Particle Size           |                         | Oil absorption<br>[g/100g] | Specific surface area BET<br>[m <sup>2</sup> /g] | Calcination       |            |
|---|-------------------------|-------------------------|----------------------------|--|-------------------|------------|
|   | d <sub>50</sub><br>[μm] | d <sub>97</sub><br>[μm] |                            |  | Surface treatment |            |
|   |                         | Polestar 200 R          | 3.6                        | 19   | 60                | 6.5        |
|   | Sillitin Z 89           | 1.6                     | 6.1                        | 51   | 11                | none none  |
|   | Silfit Z 91             | 1.9                     | 8.6                        | 52   | 6.5               | with none  |
|   | Aktisil VM 56/89        | 1.8                     | 7.4                        | 45   | 7.3               | none vinyl |
|   | Aktifit VM              | 1.9                     | 8.7                        | 49   | 6.2               | with vinyl |

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Along with the two Calcined Neuburg Siliceous Earth grades Silfit Z 91 and Aktifit VM, the two standard grades Sillitin Z 89 and - in analogy to the surface treatment for Aktifit VM with vinyl functional group - Aktisil VM 56/89 and the non-treated calcined clay Polestar 200 R were included in the comparative tests.

The particle size results indicate the coarser nature of the calcined clay in comparison with the Hoffmann Mineral products. Sillitin Z 89 offers the smallest particle size, Aktisil VM 56/89 slightly higher. The two calcined versions are somewhat coarser than the two standard Siliceous Earth grades.

The oil absorption of the Neuburg Siliceous Earth grades are on a comparable level slightly below Polestar 200 R.

The table further shows that Sillitin Z 89 offers the highest specific surface area, while the other fillers can be judged as similar within each other.

The compounds were prepared on a laboratory mill Ø 150 x 300 mm at a roll temperature of 50 °C. The batch size was about 750 cm<sup>3</sup>, the mixing process time on average 15 minutes.

Samples were cured for 5 minutes in a press and a temperature of 180 °C.

### 3 Comparison of fillers

In this section, all above-mentioned fillers will be compared. The assessment follows RAL GZ 716/1, elastomer class B II and RAL 9002.

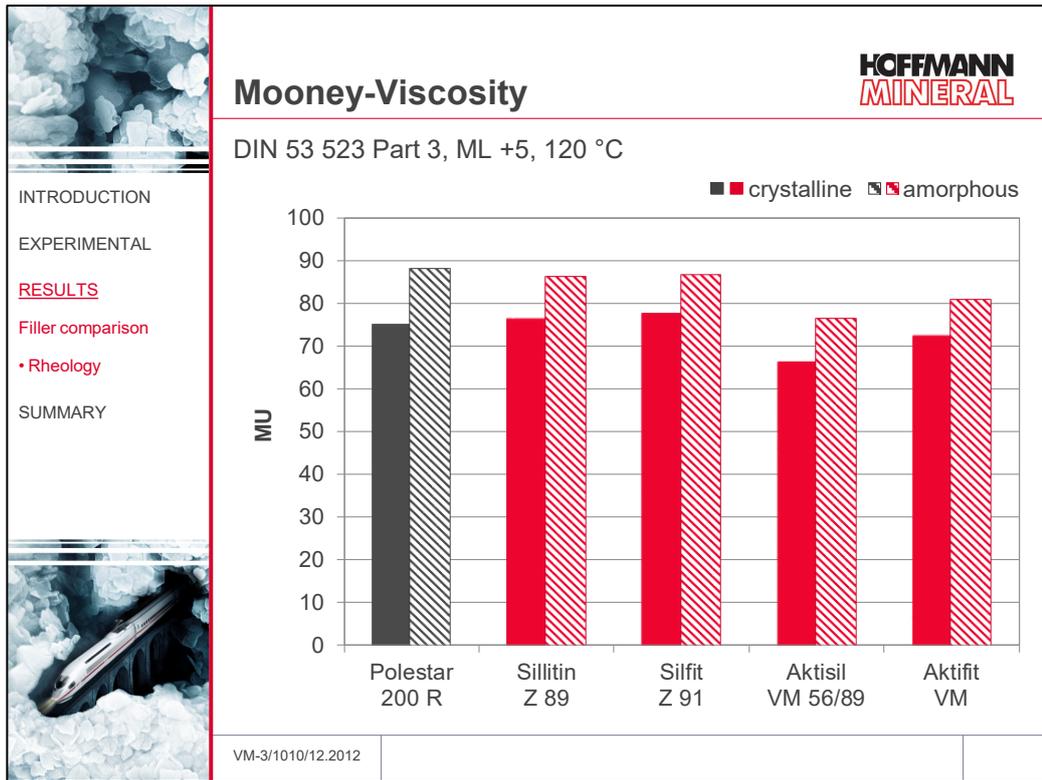
#### 3.1 Formulation

| <b>HOFFMANN<br/>MINERAL</b> |  |               |
|-----------------------------|--|---------------|
| <b>Formulation</b>          |  |               |
|                             | <b>in phr</b>  |               |
| INTRODUCTION                | Keltan 778 Z – crystalline or<br>Keltan 8340 A – amorphous | 100.00        |
| <b>EXPERIMENTAL</b>         | Zinkoxyd aktiv   | 5.00          |
| Filler comparison           | Stearic acid   | 0.50          |
| RESULTS                     | Vulkanox HS/LG   | 0.75          |
| SUMMARY                     | Lipoxol 3000   | 2.00          |
|                             | Kronos 2222  | 15.00         |
|                             | Kezadol GR   | 10.00         |
|                             | Aflux 42   | 2.00          |
|                             | Trigonox 29/40 B pd  | 3.00          |
|                             | Perkadox 14/40 B pd  | 3.00          |
|                             | Rhenofit TRIM/S  | 1.00          |
|                             | Primol 352   | 45.00         |
|                             | Mineral filler   | 200.00        |
|                             | <b>Total</b>   | <b>387.25</b> |
|                             | VM-3/1010/12.2012  |               |

|                      |   |
|----------------------|---|
| Keltan 778 Z:        | EPDM, crystalline   |
| Keltan 8340 A:       | EPDM, amorphous   |
| Zinkoxyd aktiv:      | zinc oxide  |
| Stearinsäure:        | processing aid  |
| Vulkanox HS/LG:      | antioxidant, TMQ  |
| Lipoxol 3000:        | polyethylene glycol                                       |
| Kronos 2222:         | titanium dioxide  |
| Kezadol GR:          | calcium oxide, 80 %                                       |
| Aflux 42:            | dispersing and lubricating aid                            |
| Trigonox 29/40 B pd: | 1,1-Di(tert-butylperoxy)-3,3,5-trimethylcyclohexane, 40 % |
| Perkadox 14/40 B pd: | Di(tert-butylperoxyisopropyl)benzene, 40 %                |
| Rhenofit TRIM/S:     | Trimethylolpropane trimethacrylate, 70 %                  |
| Primol 352:          | white oil, medical quality                                |

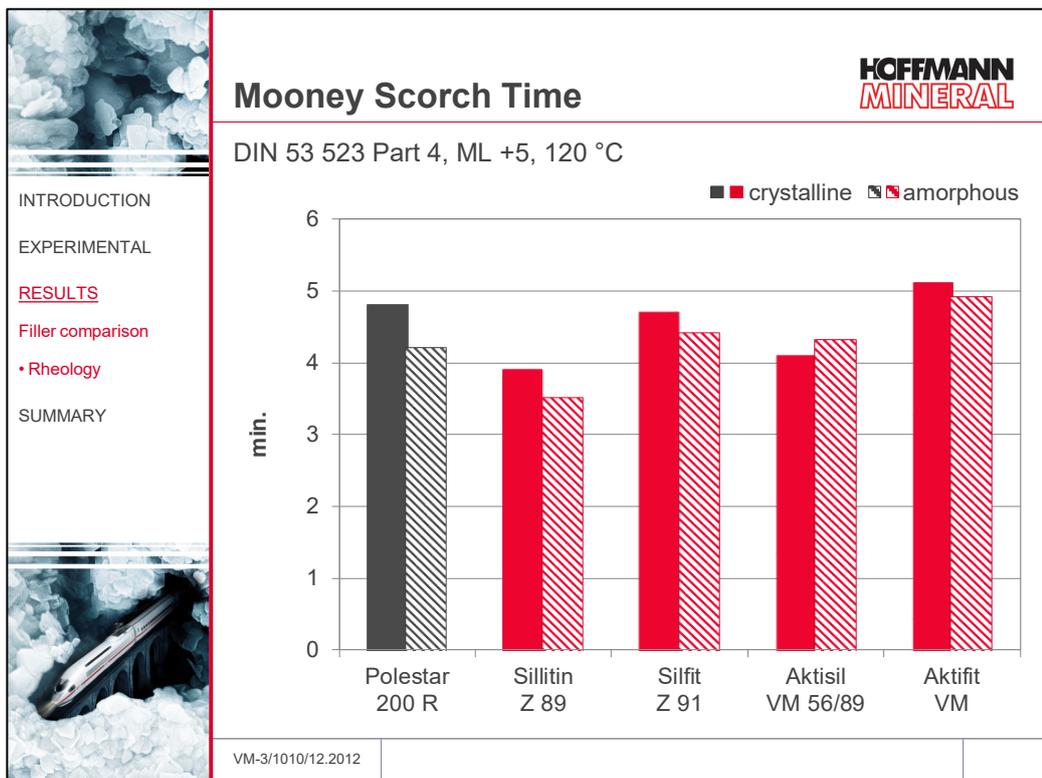
It is true, with a crystalline EPDM the requirements of RAL GZ 716/1, elastomer class B II can be met, but it is all the same recommended to use an amorphous grade, in case the conditions regarding the low temperature properties (hardness increase, compression set) are becoming more stringent.

### 3.2 Viscosity, scorch and curing characteristics



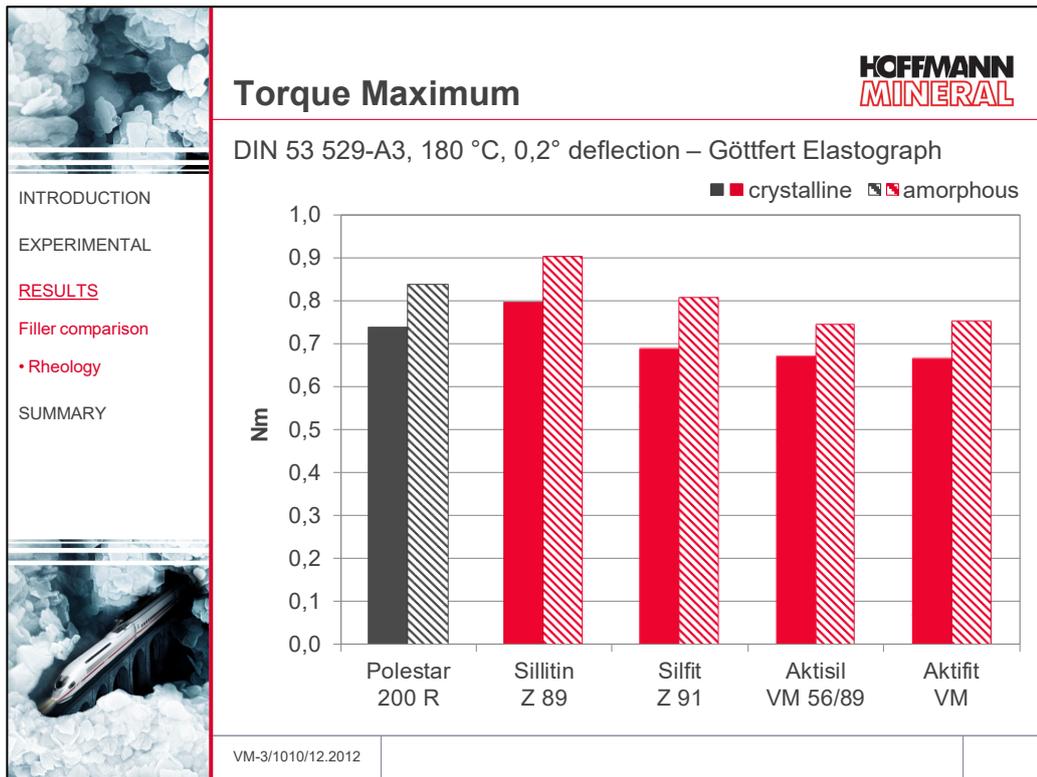
The graph shows that the results for the non-treated fillers come out at a comparable level. The viscosity with Aktifit VM is somewhat lower, and the lowest results are obtained with Aktisil VM 56/89.

These trends are evident with both polymers. The results with the amorphous grade are higher than those for the crystalline rubber.

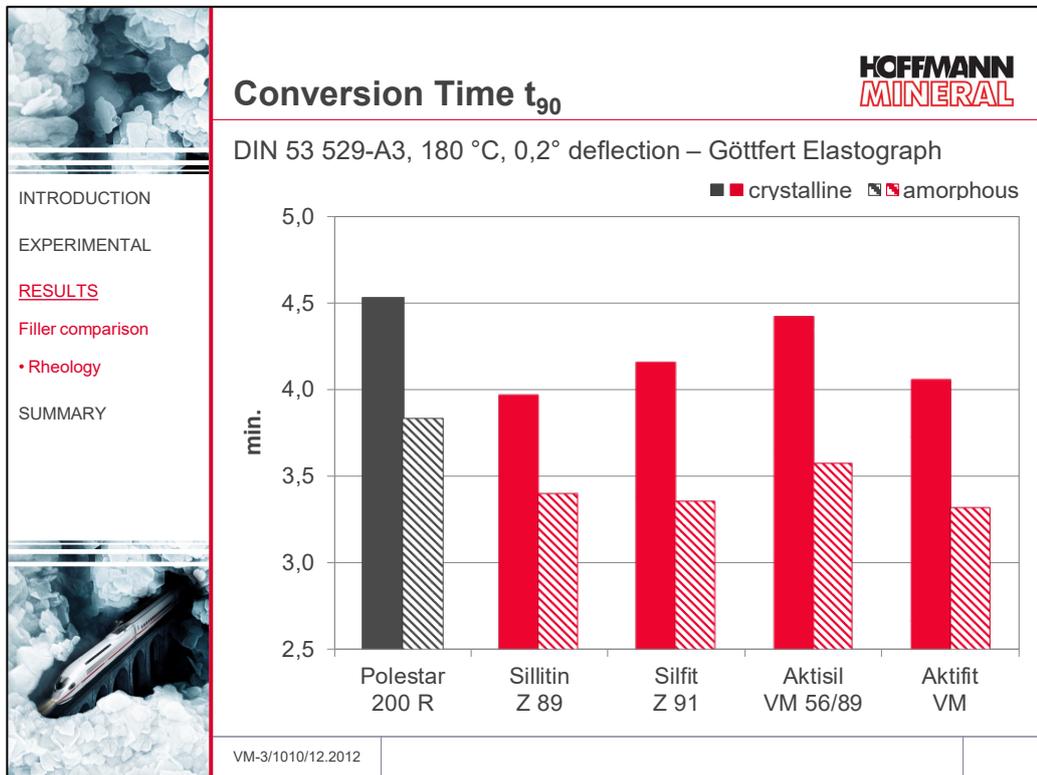


The shortest scorch time is imparted by Sillitin Z 89. The two calcined non-treated fillers Polestar 200 R and Silfit Z 91 come out somewhat longer. As a result of the surface treatment, the scorch times with Aktisil VM 56/89 and Aktifit VM are marginally longer in comparison with their base products Sillitin Z 89 and Silfit Z 91.

These trends are evident with both polymers. The scorch times with the crystalline type are directionally somewhat longer in comparison with the amorphous rubber.



In cure meter testing the highest torque maximum is obtained with Sillitin Z 89. In the crystalline EPDM, Polestar 200 R and Silfit Z 91 come out slightly lower. The torque maxima with Aktisil VM 56/89 and Aktifit VM are comparable with each other and somewhat lower than with the two above-mentioned fillers. In the amorphous EPDM, no difference is seen between Silfit Z 91, Aktisil VM 56/89 and Aktifit VM. Their results come out just below Polestar 200 R. Again in the amorphous rubber grade, Sillitin Z 89 ranks highest.



The conversion time  $t_{90}$  with Aktisil VM 56/89 is similar to the result with Polestar 200 R. Sillitin Z 89, Silfit Z 91 and Aktifit VM give somewhat shorter conversion times, which all are found at a comparable level. This shorter time requirement should represent a favour-

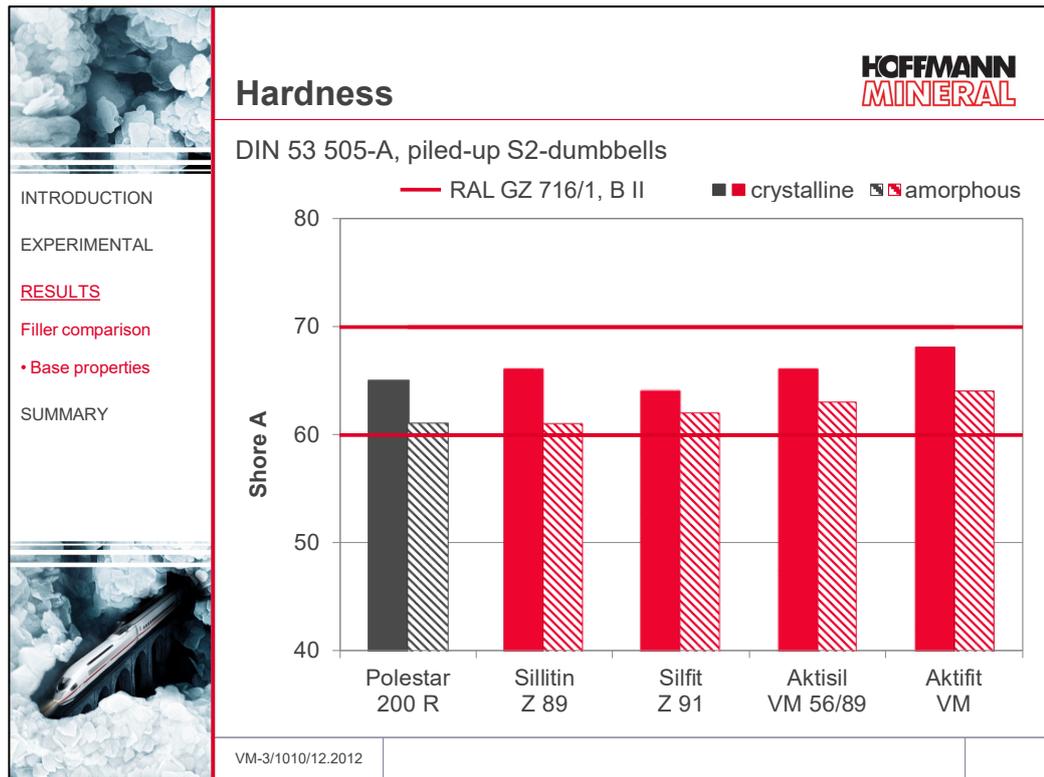
able effect for instance for Aktifit VM, as with a given salt bath length and a given curing temperature a higher haul-off speed can be operated than with the calcined clay Polestar 200 R.

These results are true for both the crystalline and the amorphous EPDM grade, with the conversion times with the amorphous rubber coming up shorter than with the crystalline rubber.

### 3.3 Mechanical properties

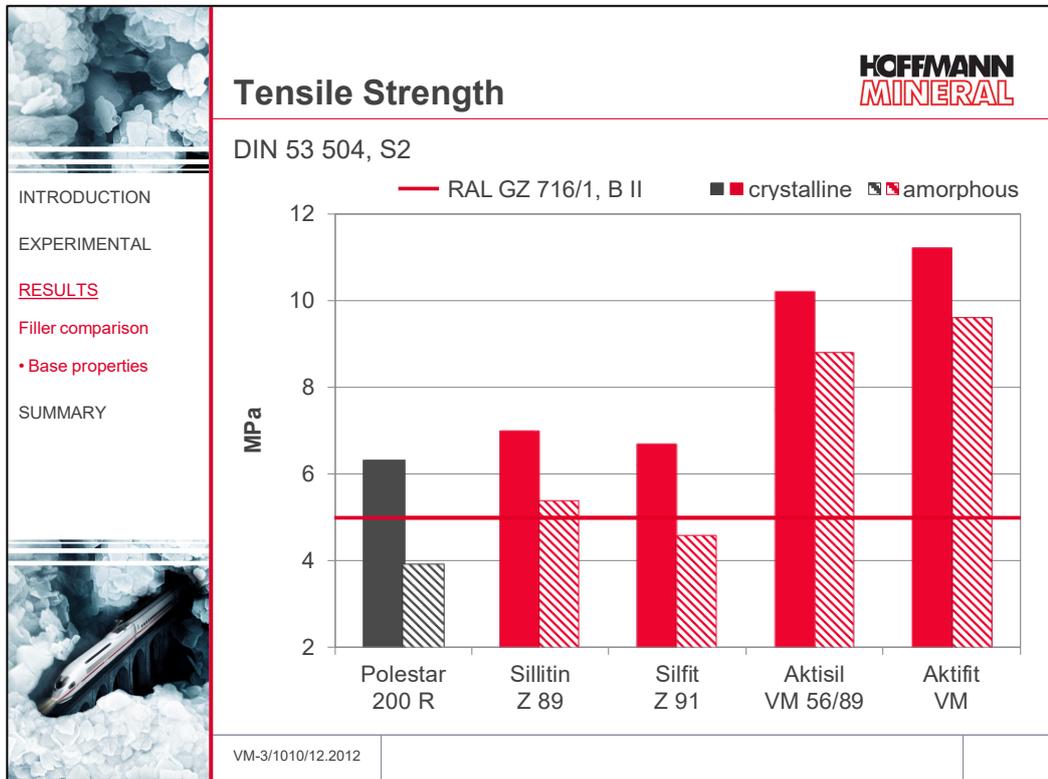
The compounds were press-cured at 180 °C.

As the conversion time  $t_{90}$  was below 5 minutes throughout, all compounds and all the different sample geometries were cured for a fixed time of 5 minutes.



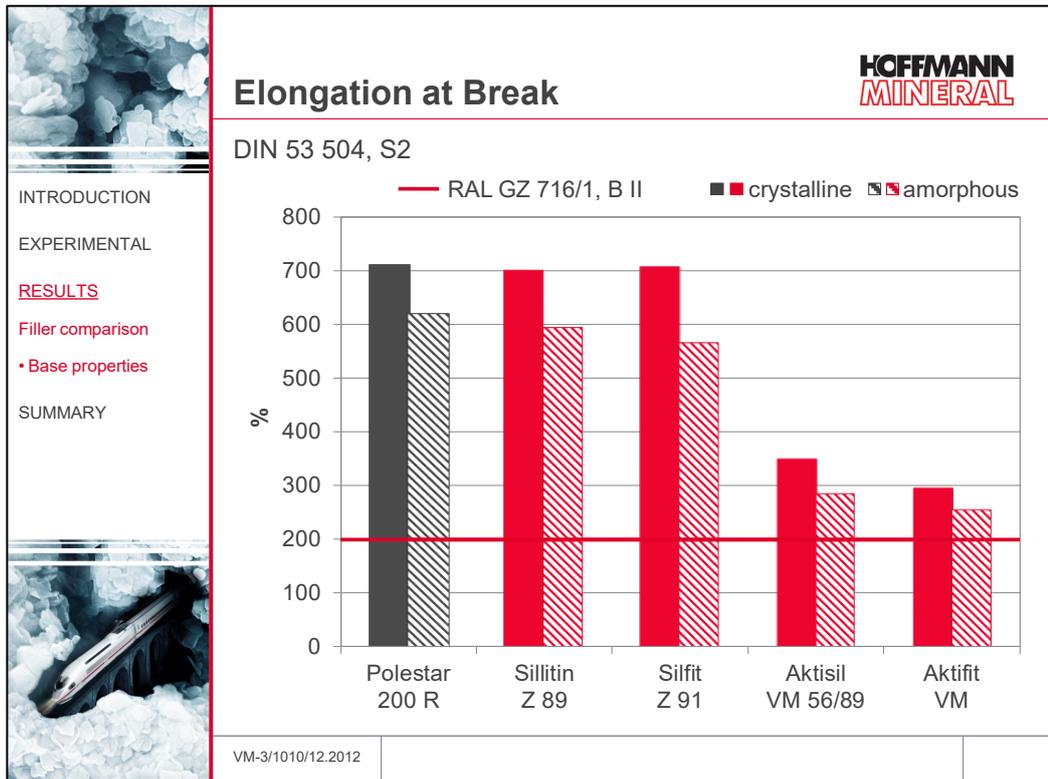
Hardness was determined on three piled-up S2-dumbbells.

As the graph shows, the individual fillers in both polymers are found at a comparable level which fulfils the requirements of RAL GZ 716/1, B II. The hardness level with the amorphous EPDM comes out lower than with the crystalline elastomer.

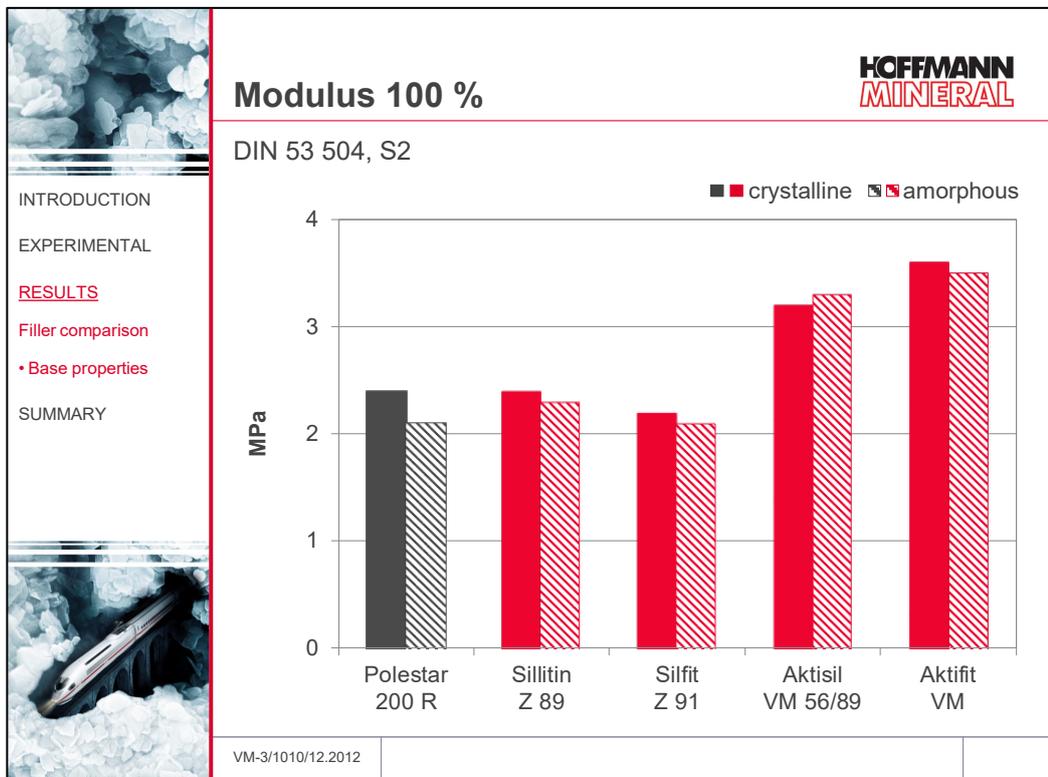


The crystalline EPDM allows to comply with the tensile strength specified in RAL GZ 716/1, B II with all fillers tested. The non-treated grades Polestar 200 R, Sillitin Z 89 and Silfit Z 91 are found at a similar level. The surface treatment of Aktisil VM 56/89 and Aktifit VM leads to a marked increase of the tensile strength for both fillers.

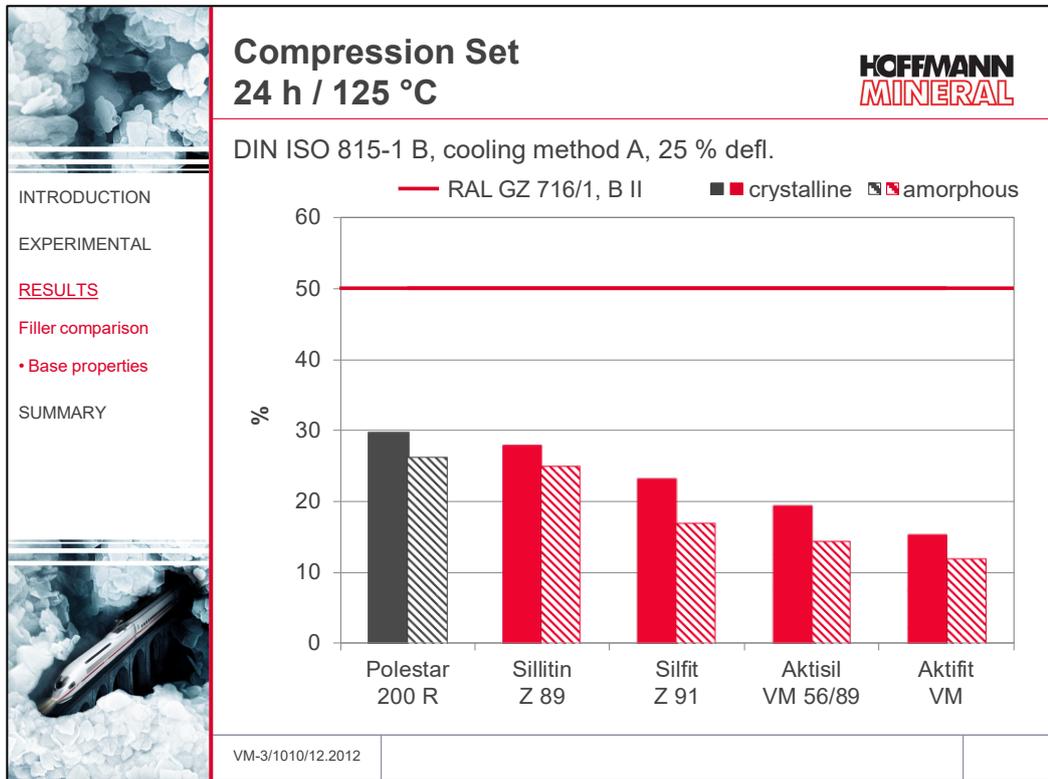
Sillitin Z 89 is the only non-treated filler that just meets the requirement of the standard when used with the amorphous EPDM grade, while Silfit Z 91 and Polestar 200 R come out slightly below the specified level of 5 MPa. Silfit Z 91 arrives here at a somewhat higher level than Polestar 200 R. With Aktisil VM 56/89 and Aktifit VM the tensile strength requirements of the standard can be met without problems also in the amorphous EPDM elastomer. In both rubbers, Aktifit VM marks the highest results.



The non-treated fillers impart comparable elongations which, as expected, are higher than for the treated grades. The results with the amorphous EPDM are somewhat below those with the crystalline grade. The requirements of the standard are satisfied in all cases.

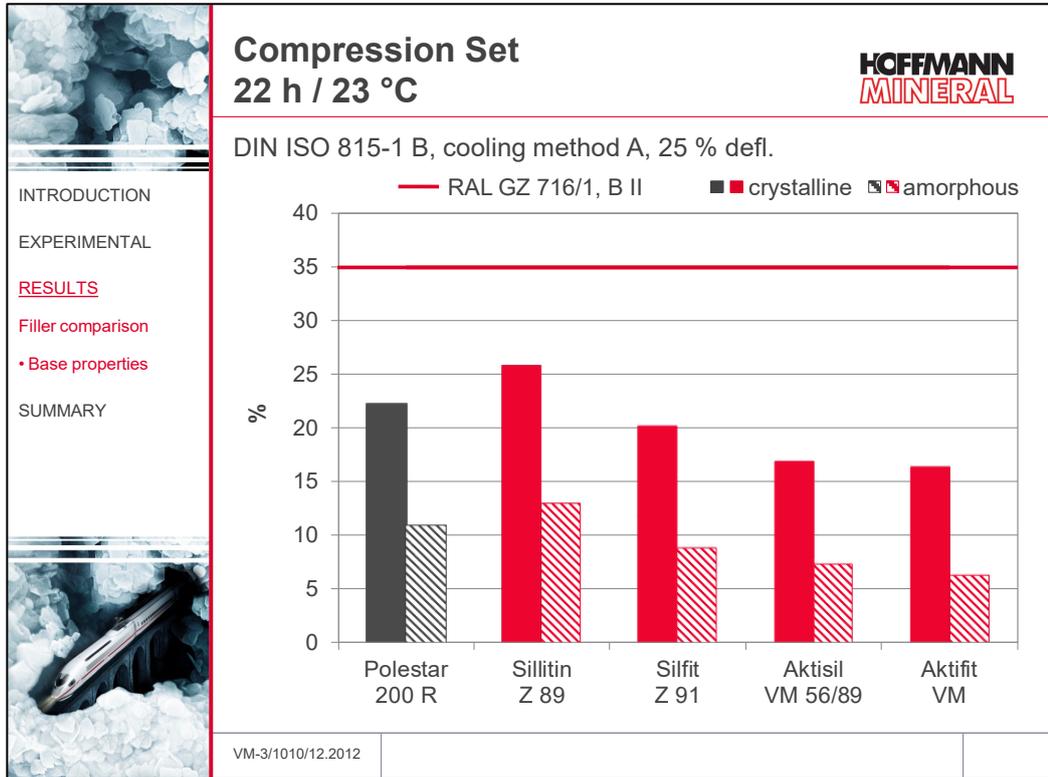


The modulus results at 100 % elongation of the non-treated fillers come out at a similar level, and this below those of the treated grades. Aktifit VM gives the highest moduli. No significant difference is seen here between the two elastomer grades.



The compression set was determined under the more critical condition of 125 °C, and here it is compared with the requirement of RAL GZ 716/1, B II for 22 hours at 70 °C. Quite obviously, results largely below 50 % are obtained even at the markedly higher test temperature. This means, the major function of "sealing over long periods of time" will be further improved.

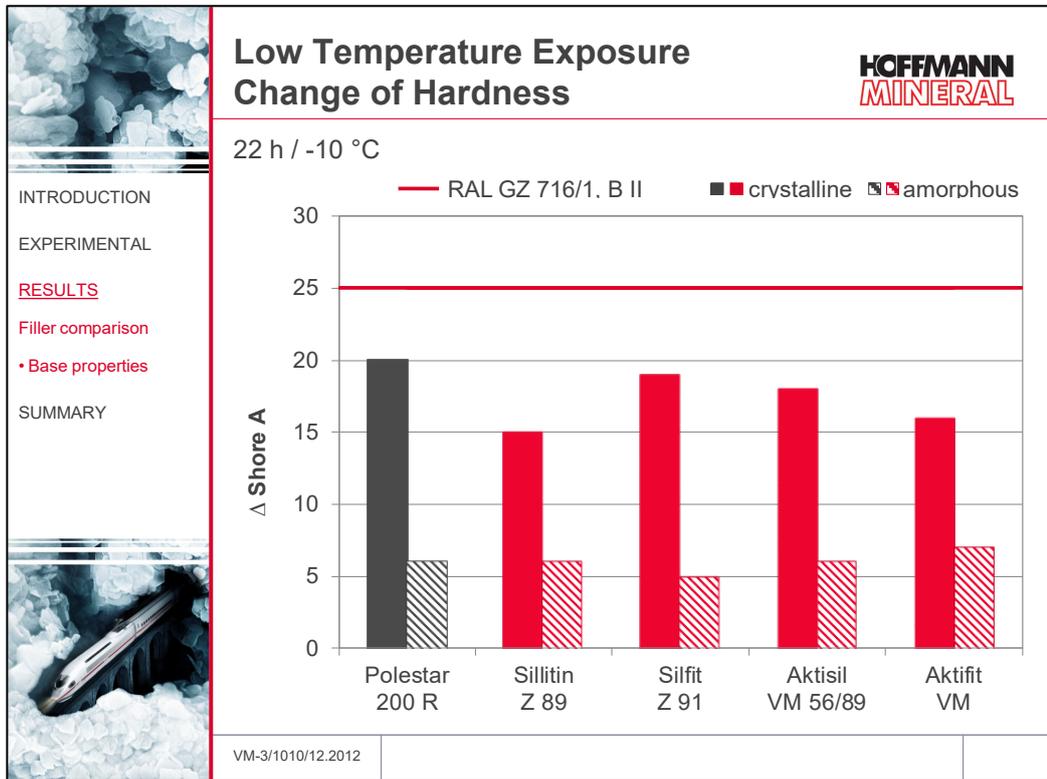
The graph further shows that the compression set with Sillitin Z 89 looks similar to Polestar 200 R. Among the non-treated fillers, Silfit Z 91 imparts the lowest set. The results with Aktisil VM 56/89 are still somewhat lower and is outperformed positively by Aktifit VM. These statements are true equally for the crystalline as well as for the amorphous EPDM grade, with the latter on a distinctly lower level.



Equally, when tested 22 hours at 23 °C, the specification of the standard is satisfied with all fillers.

At 23 °C, Sillitin Z 89 arrives at a somewhat higher compression set compared with Polestar 200 R. Here again, Silfit Z 91 comes out with the lowest result among the non-treated fillers. No difference is observed between Aktisil VM 56/89 and Aktifit VM.

These findings are equally true for the crystalline and the amorphous EPDM type, with the latter offering markedly lower results. As already mentioned, this means that a better low temperature performance can be expected with the amorphous EPDM grade.

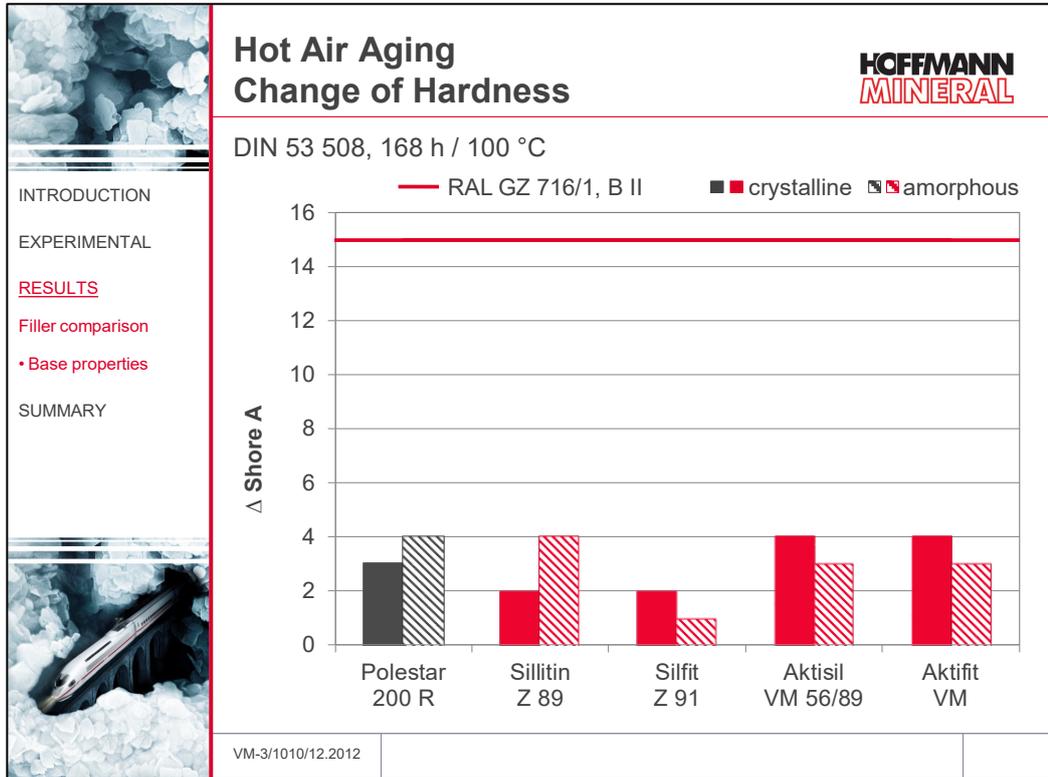


The biggest change in hardness after 22 hours of exposure to -10 °C is registered with the crystalline EPDM loaded with Polestar 200 R. Silfit Z 91 and Aktisil VM 56/89 come off slightly lower. Sillitin Z 89 and Aktifit VM in the crystalline elastomer show comparably the lowest hardness changes.

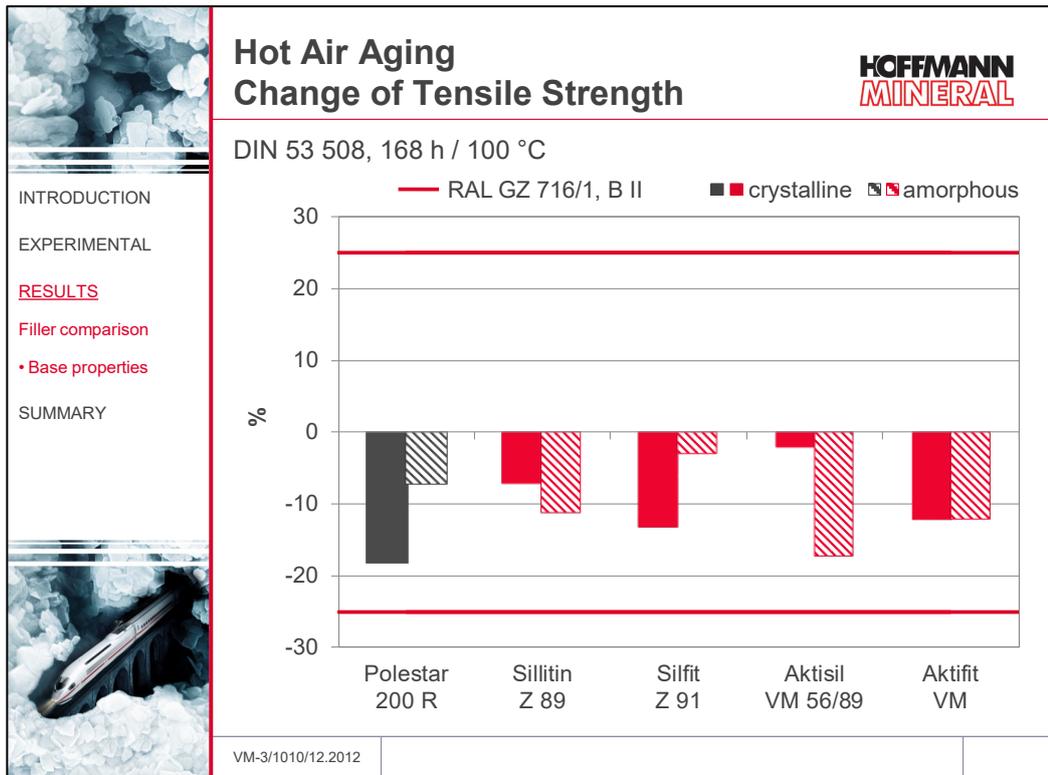
The graph clearly shows the advantage of the amorphous polymer. The hardness changes are reduced so significantly that no differences can be found any longer between the individual fillers.

Working with the amorphous EPDM can be of interest, for instance, with regard to the specifications of DIN 7863, where for black building profiles a maximum hardness increase of 10 Shore A after storage at -10 °C is defined.

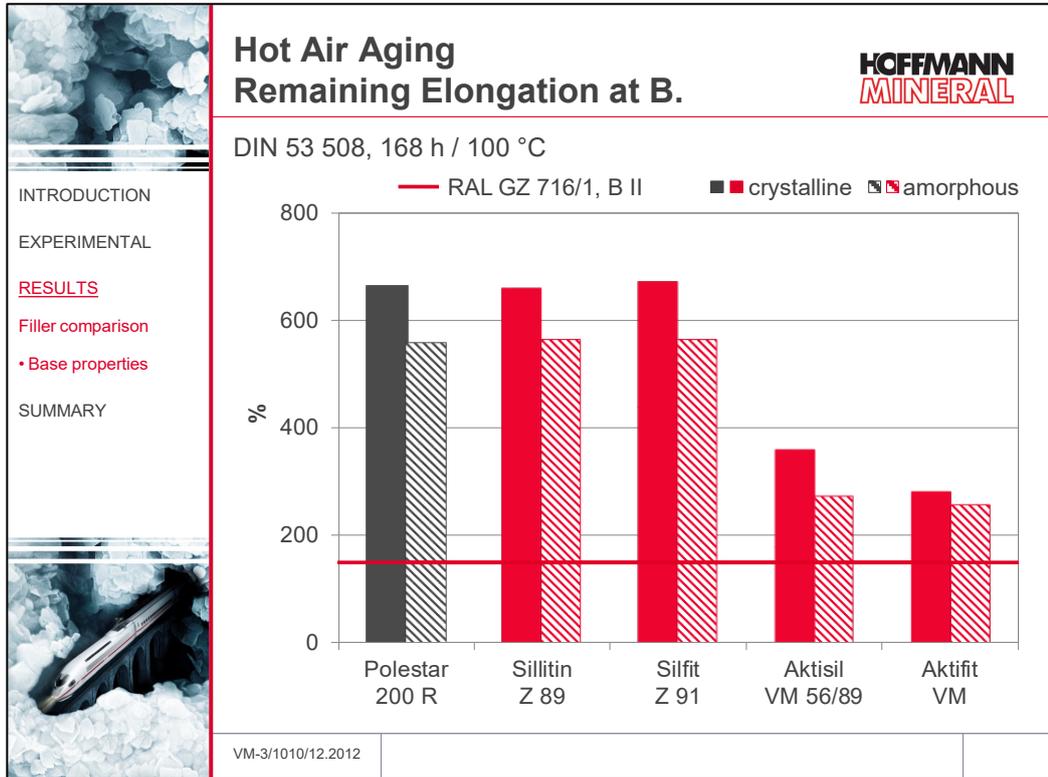
However, the requirement of the standard RAL GZ 716/1, B II, which is in the focus of this study, can also be met with the crystalline EPDM grade.



The hardness change after hot air aging 168 hours at 100 °C does not show any significant differences neither between the individual fillers nor between the two EPDM grades. All compounds remain largely below the maximum specified limit of the standard.



Likewise, between the changes of the tensile strength after hot air aging no big difference can be observed between the the fillers respectively the elastomer grades. The requirements of the standard are satisfied without problems in all cases.



As already found before the hot air aging, the non-treated fillers show comparable results, which according to expectations, come out above the results for the surface treated fillers. The amorphous EPDM gives slightly lower results than the crystalline grade.

The specification limits of the standard are easily met in all cases.

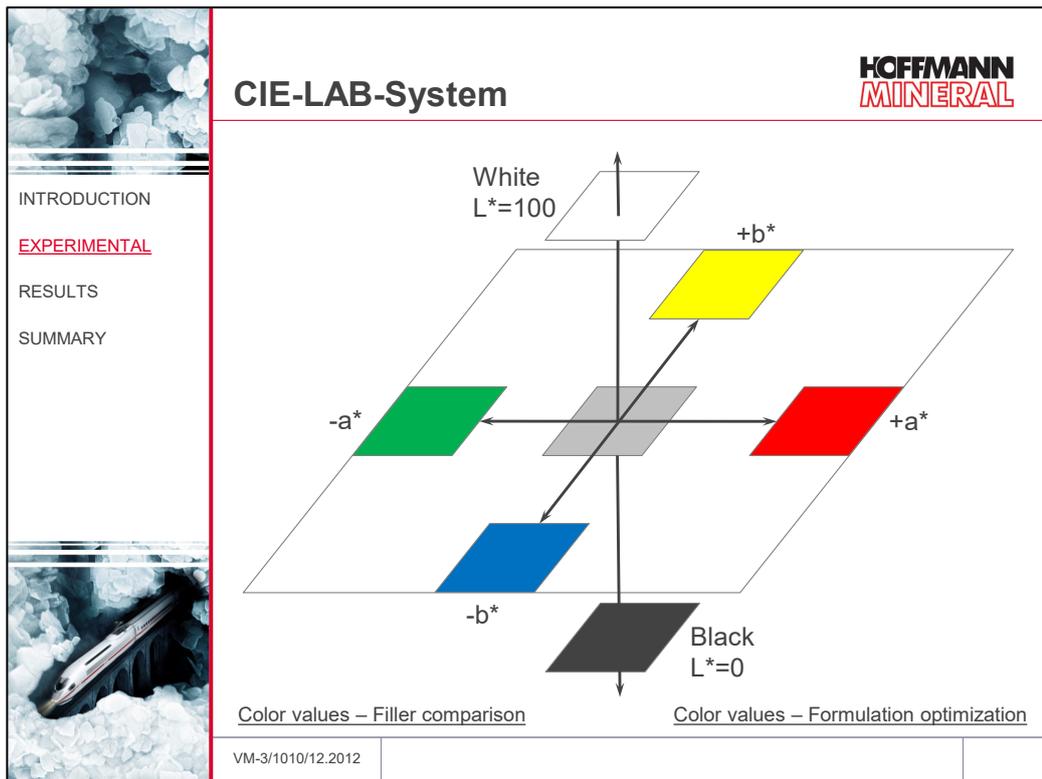
### 3.4 Color

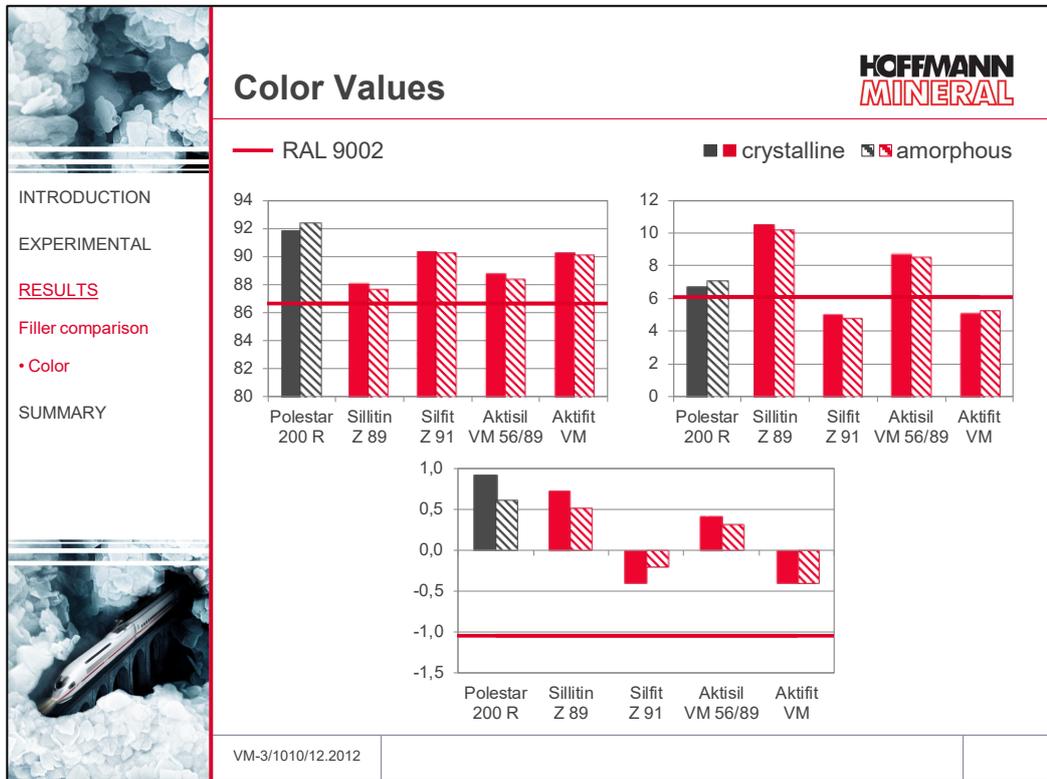
Until recently, it was not possible to produce light-colored profiles without a yellowish tint with the standard grades of Neuburg Siliceous Earth. This way, the use of Neuburg Siliceous Earth was in fact limited to black or grey profiles.

With Calcined Neuburg Siliceous Earth it is now possible to extend the application portfolio of Hoffmann Mineral products to include light-colored sections due to their high brightness and color neutrality.

This will be demonstrated in the following. The assessment was based on the RAL 9002 standard.

The color determinations were carried out with the spectral photometer Luci 100 (Dr. Lange) with light D65 and a measuring geometry  $d/8^\circ$  (without gloss trap) and a normal observation angle of  $10^\circ$ . Each time, the  $L^*$ ,  $a^*$  and  $b^*$  values were registered.





The three graphs clearly indicate that Polestar 200 R allows to come very close to the  $b^*$  requirement of the RAL 9002 specification, but this with a too high brightness (as shown by the  $L^*$  value), and instead of a required greenish, a reddish tint is evident, as confirmed by the positive  $a^*$  result.

Likewise, with the standard Neuburg Siliceous Earth grades Sillitin Z 89 and Aktisil VM 56/89, a reddish tint is obvious. In addition, there is a high yellowish tint, as evident from the comparison of the  $b^*$  values. Just only the  $L^*$  brightness comes close to the RAL 9002 figures.

With the Calcined Neuburg Siliceous Earth grades Silfit Z 91 and Aktifit VM, the brightness levels come out somewhat above the specified level, but they still remain below the too bright results with Polestar 200 R. Furthermore, the  $a^*$  readings of the cured compounds with these two fillers, as shown in the plot, come closer to RAL 9002. Also the yellowish tint with Silfit Z 91 and Aktifit VM is clearly minimized (graph  $b^*$ ), which means the cured compounds are definitely more color-neutral than those loaded with standard Neuburg Siliceous Earth grades or with the calcined clay Polestar 200 R.

### 3.5 Summary section 3

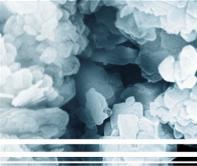
At a loading of 200 phr, Silfit Z 91 is able to satisfy the requirements of the RAL GZ 716/1, B II when working with a crystalline EPDM grade without any problems. The use of the amorphous rubber grade just requires an optimization of the tensile strength. Compared with Sillitin Z 89, Silfit Z 91 delivers a more neutral color. This advantage also remains in comparison with the calcined clay Polestar 200 R. In addition, further benefits concern a shorter conversion time  $t_{90}$ , a tendentially higher tensile strength and a lower compression set.

Aktifit VM at a loading of 200 phr allows to satisfy all requirements of the standard both when using the crystalline or the amorphous EPDM elastomer. In comparison with Aktisil VM 56/89 and Polestar 200 R, the color of the cured compounds comes out more neutral. Furthermore, Aktifit VM will lead to shorter conversion time  $t_{90}$  and improved mechanical properties, such as higher tensile strength and a lower compression set compared with Aktisil VM 56/89 or Polestar 200 R.

The results with Aktifit VM offer themselves for optimization efforts with regard to the filler loading and also to the peroxide concentration. In addition, cost considerations speak for making the most of the performance potential of Aktifit VM. This will be demonstrated in the following.

## 4 Optimized formulation - performance potential of Aktifit VM

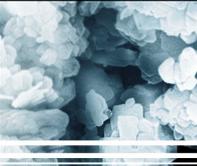
### 4.1 Formulation and variations

|  | <b>Formulation</b>                                      |                  |
|--|---|------------------|
|  |   | <b>in phr</b>    |
| <br>INTRODUCTION<br><u>EXPERIMENTAL</u><br>Formulation optimization<br>RESULTS<br>SUMMARY<br> | Keltan 778 Z – crystalline or Keltan 8340 A – amorphous | 100.00           |
|  | Zinkoxyd aktiv  | 5.00             |
|  | Stearic acid  | 0.50             |
|  | Vulkanox HS/LG  | 0.75             |
|  | Lipoxol 3000  | 2.00             |
|  | Kronos 2222   | 15.00            |
|  | Kezadol GR  | 10.00            |
|  | Aflux 42  | 2.00             |
|  | Trigonox 29/40 B pd                                     | 2 or 3           |
|  | Perkadox 14/40 B pd                                     | 2 or 3           |
|  | Rhenofit TRIM/S   | 1.00             |
|  | Primol 352  | 45 - 95          |
|  | <b>Mineral filler</b>                                   | <b>200 - 400</b> |

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The starting point largely followed the base formulation of the filler comparison. The filler loading was varied from 200 to 400 phr. In order to arrive at compounds with similar hardness and thus comparable properties, increases of the filler loading were compensated by higher oil additions. Furthermore, the peroxide concentrations were reduced. Also the work in this test series included the crystalline EPDM rubber Keltan 778 Z and the amorphous grade Keltan 8340 A.

|   | <b>Formulation Variations</b> |                    |            |                  |            |            |
|---|-------------------------------|--------------------|------------|------------------|------------|------------|
|   | Loadings in phr               |                    |            |                  |            |            |
|   |                               | <b>crystalline</b> |            | <b>amorphous</b> |            |            |
| <br>INTRODUCTION<br><u>EXPERIMENTAL</u><br>Formulation optimization<br>RESULTS<br>SUMMARY<br> | Polestar 200 R (Reference)    | 200                |            |                  |            |            |
|   | <b>Aktifit VM</b>             |                    | <b>400</b> | <b>300</b>       | <b>300</b> | <b>300</b> |
|   | Primol 352                    | 45                 | 95         | 70               | 70         | 70         |
|   | Trigonox 29/40 B pd           | 3                  | 3          | 2                | 3          | 2          |
|   | Perkadox 14/40 B pd           | 3                  | 3          | 2                | 3          | 2          |

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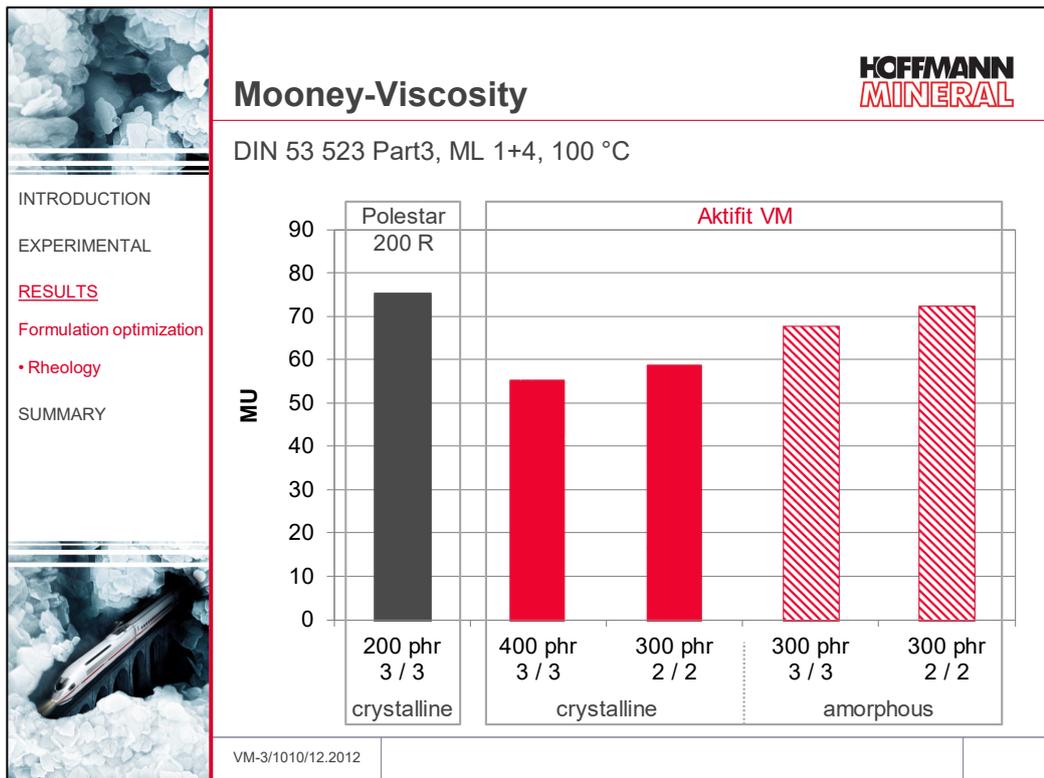
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The reference compound contained 200 phr Polestar 200 R, 45 phr plasticizer and a 3 / 3 blend of Trigonox 29/40 B pd and Perkadox 14/40 B pd (in the following called "peroxide blend") in the crystalline EPDM polymer. This is the only compound version with Polestar 200 R that is able to meet the mechanical requirements of the RAL GZ 716/1, B II standard.

Aktifit VM will be compared with the control compound in the crystalline rubber at two loadings, i.e. at 400 phr with 95 phr plasticizer and the 3 / 3 peroxide blend, respectively at 300 phr with 70 phr plasticizer and a 2 / 2 peroxide blend.

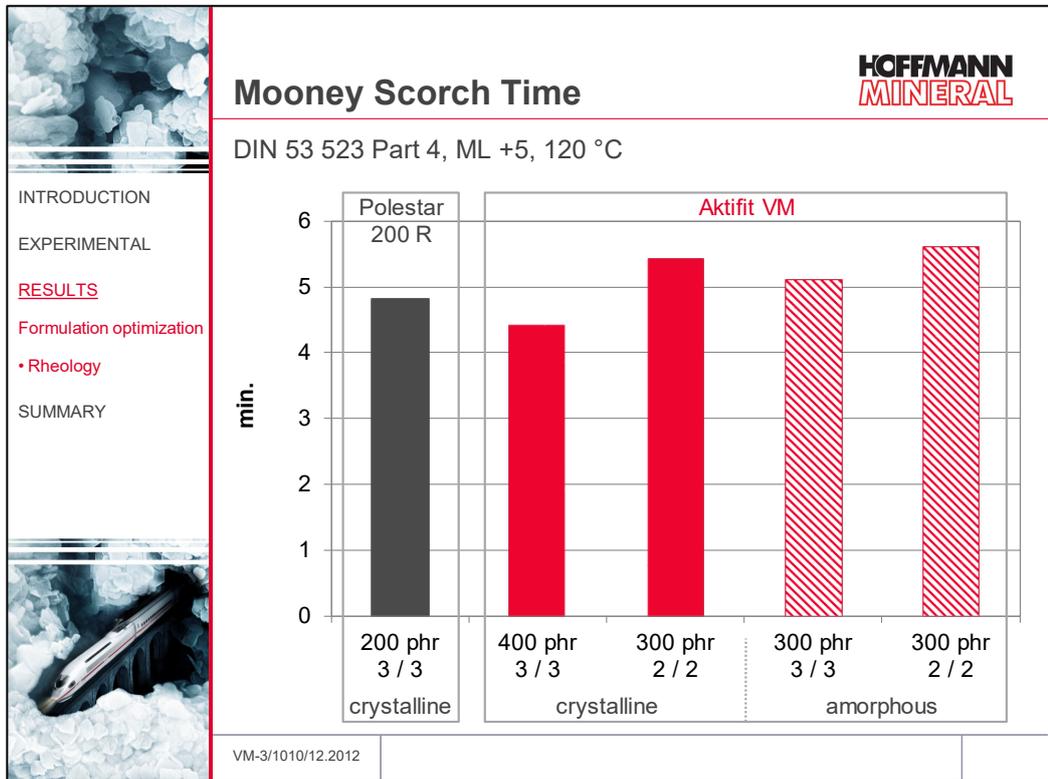
The Aktifit VM loading of 300 phr with 70 phr plasticizer is shown here for the amorphous EPDM rubber with the 3 / 3 and 2 / 2 peroxide blends.

## 4.2 Viscosity, scorch and cure characteristics

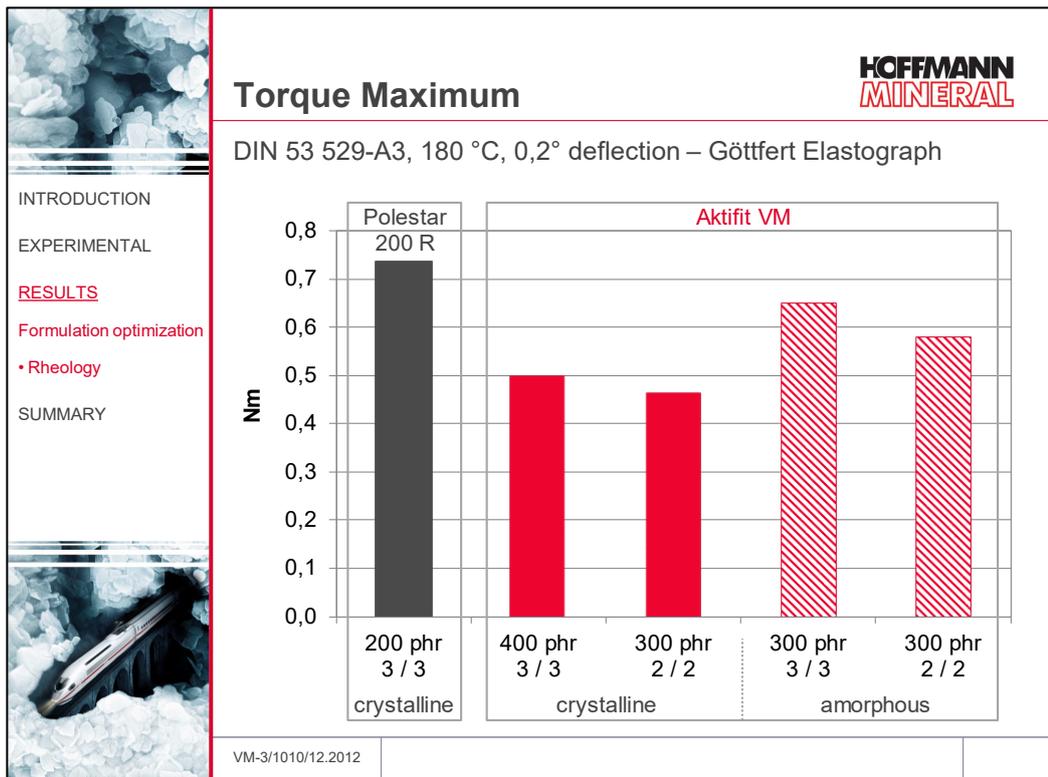


The Mooney viscosities of the two Aktifit VM compounds in the amorphous EPDM come out at a comparable level somewhat below the reference compound. With the crystalline grade, the viscosities with Aktifit VM are still somewhat lower.

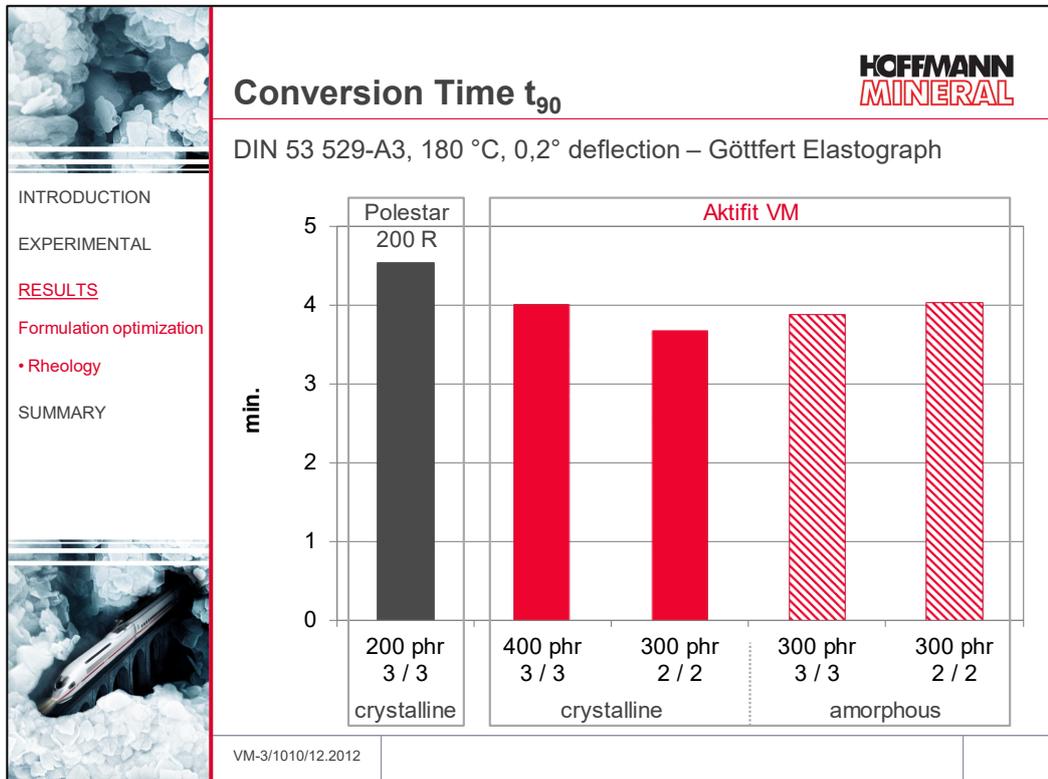
Between the loadings of 300 and 400 phr no significant difference can be observed.



As shown in the graph, the scorch times show only marginal differences. Even the reduction of the peroxide does hardly affect the scorch properties.



In general, the torque maximum goes down with an increased filler and oil loading, quite as should be expected in view of the decreased polymer content. The same trend, and stronger than for the increased filler loading, is true for the reduced peroxide dosing, as confirmed by the comparison of 300 phr Aktifit VM with the 2 / 2 blend vs. 400 phr Aktifit VM with the 3 / 3 peroxide blend in the crystalline EPDM grade.

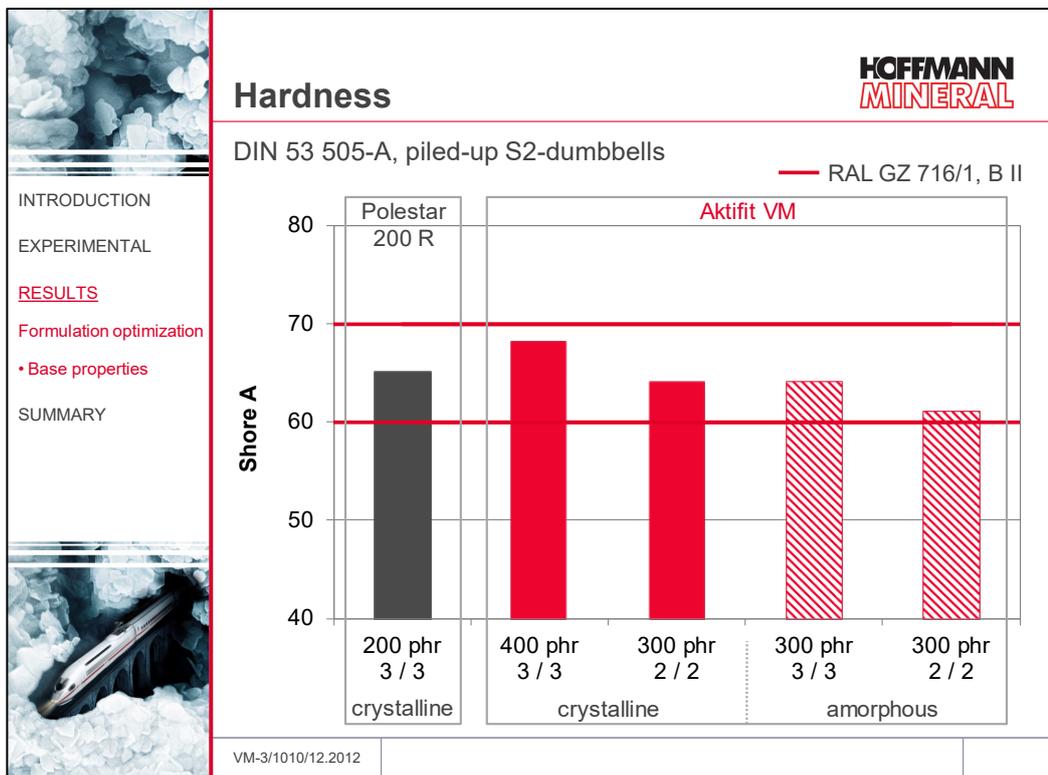


The conversion times  $t_{90}$  of the various Aktifit VM compounds are somewhat shorter in comparison with the reference compound, which should in practice be appreciated as a favorable effect.

### 4.3 Mechanical properties

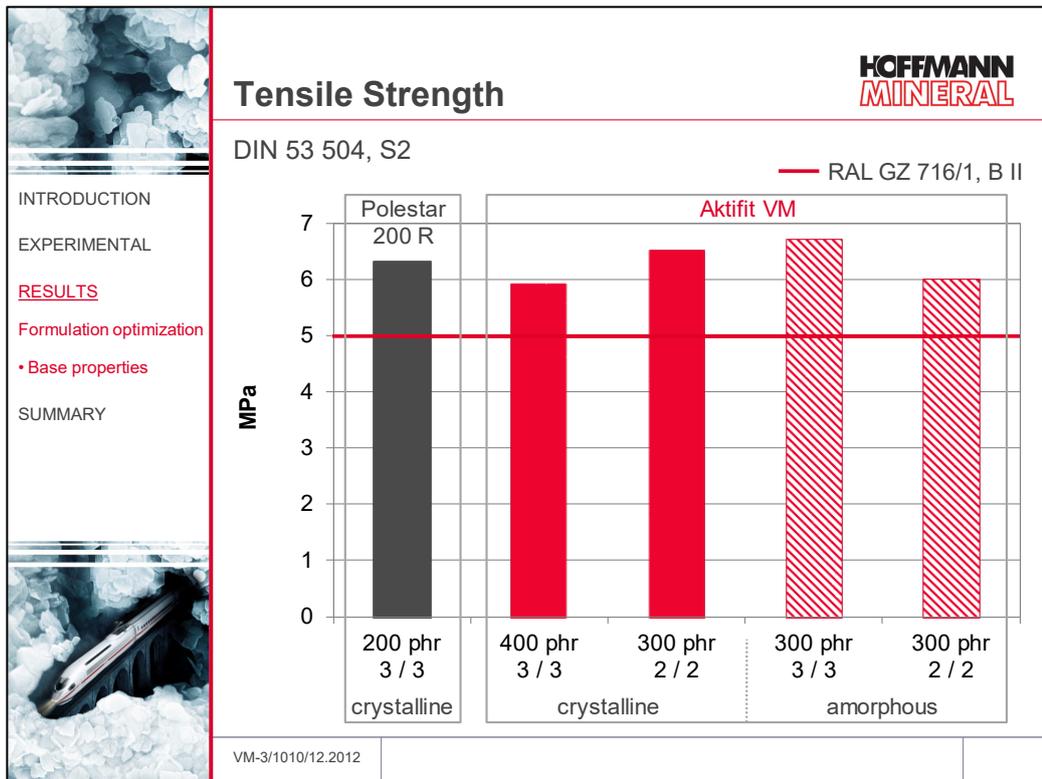
The compound were press-cured at 180 °C

As the conversion time  $t_{90}$  was always shorter than 5 minutes, the curing time for alls compounds with their different sample geometries was fixed at 5 minutes.

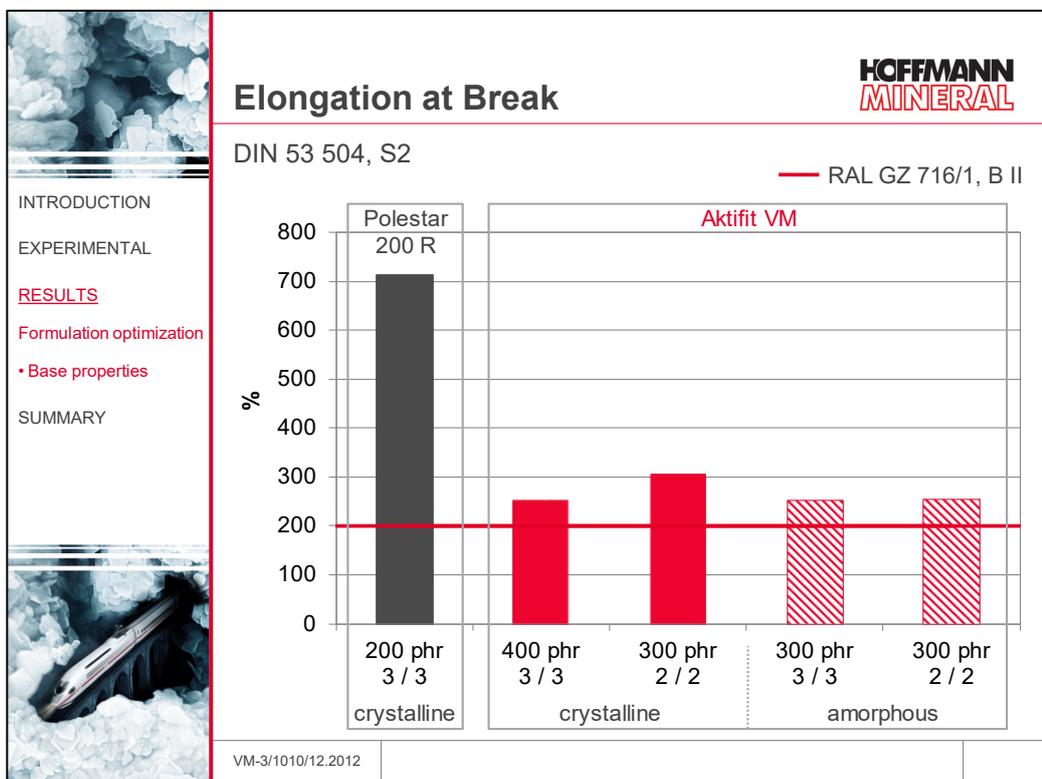


Hardness was determined on three piled-up S2-dumbbells.

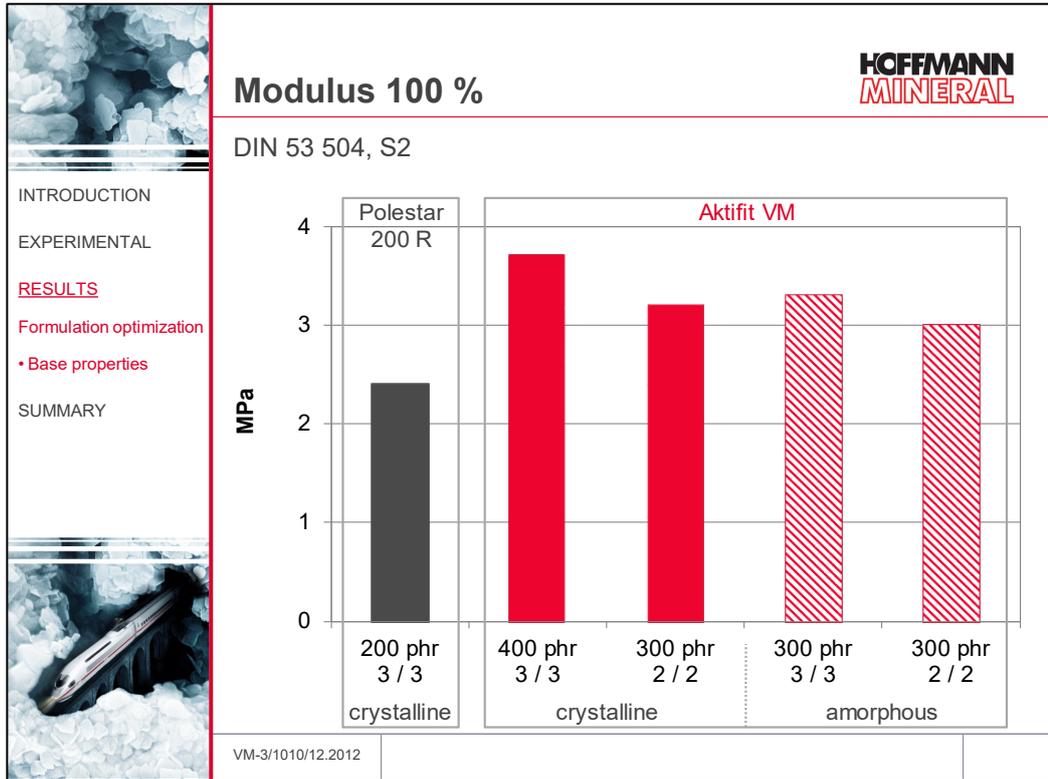
All compound variations yield hardness results in the required range. The highest level is obtained with 400 phr Aktifit VM in combination with the 3 / 3 peroxide blend in the crystalline EPDM polymer, the lowest result with 300 phr Aktifit VM and the 2 / 2 peroxide blend in the amorphous EPDM rubber.



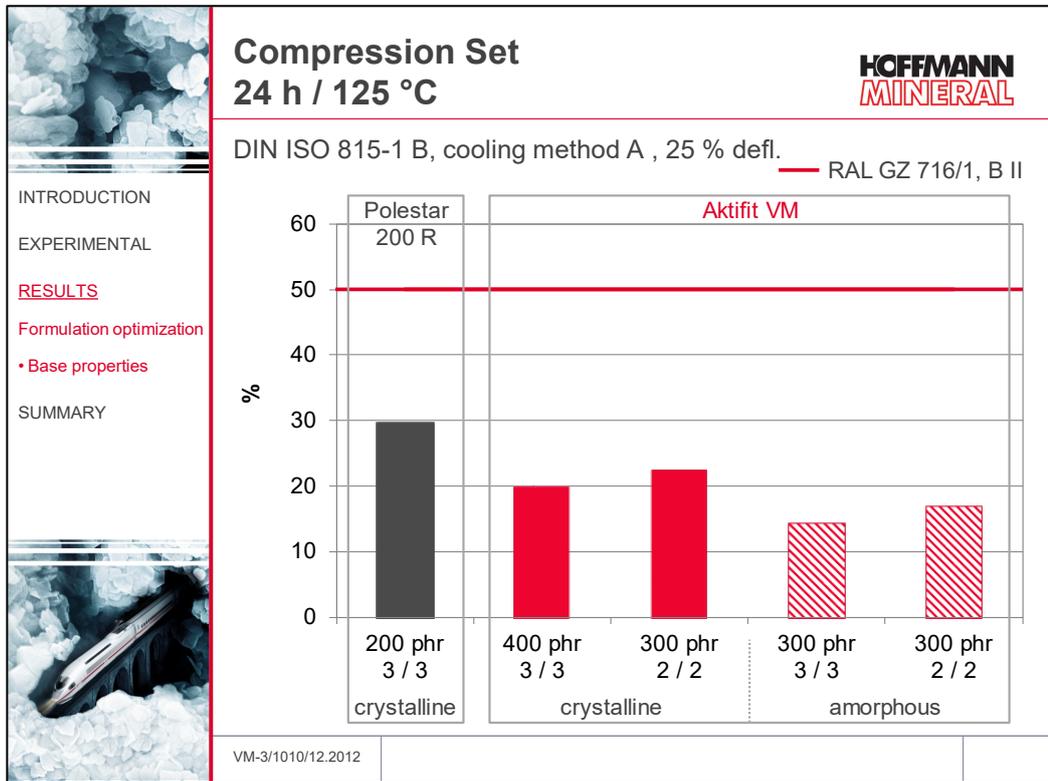
All compounds come out with a tensile strength comparable with the lowly loaded reference with Polestar 200 R, and all meet the level specified in RAL GZ 716/1, B II.



The results for the elongation at break of the compounds loaded with Aktifit VM, as a result of the surface treatment of the filler, are lower than for the control which contains the non-treated calcined clay Polestar 200 R. All compound variations with Aktifit VM surpass the minimum value of 200 % as specified in the standard.

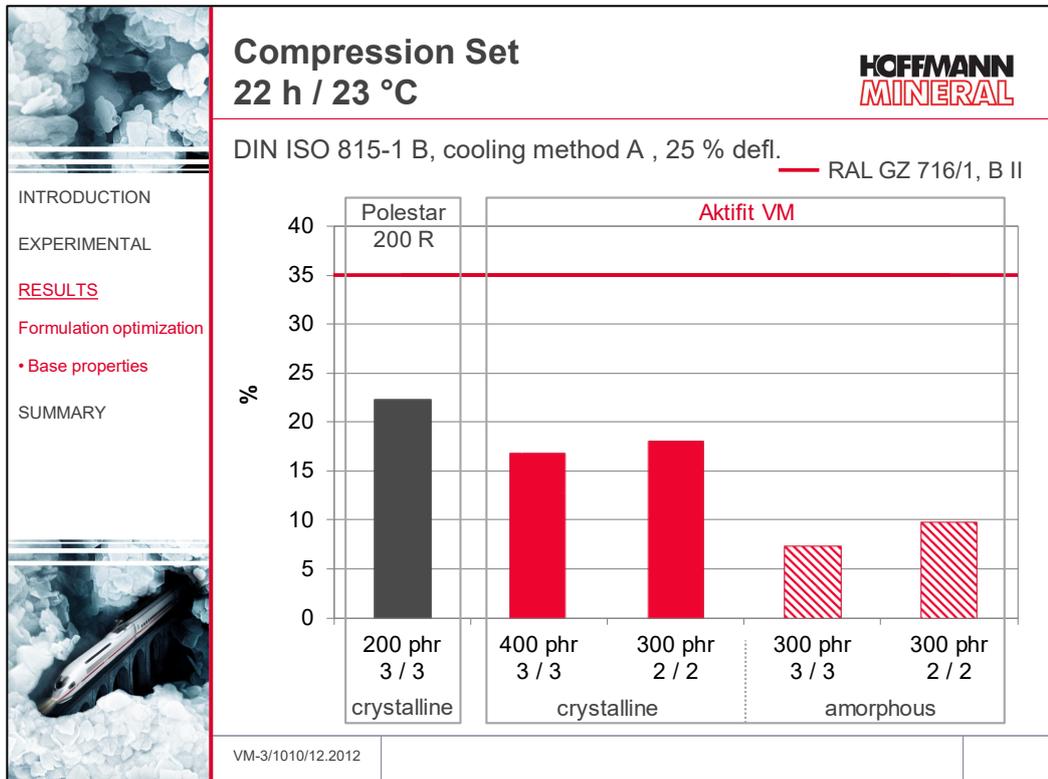


All compounds loaded with Aktifit VM surpass the 100 % modulus of the reference compound despite the higher filler and oil loading and the reduced peroxide concentration. The highest modulus is obtained with 400 phr Aktifit VM in combination with the 3 / 3 peroxide blend in the crystalline EPDM elastomer.

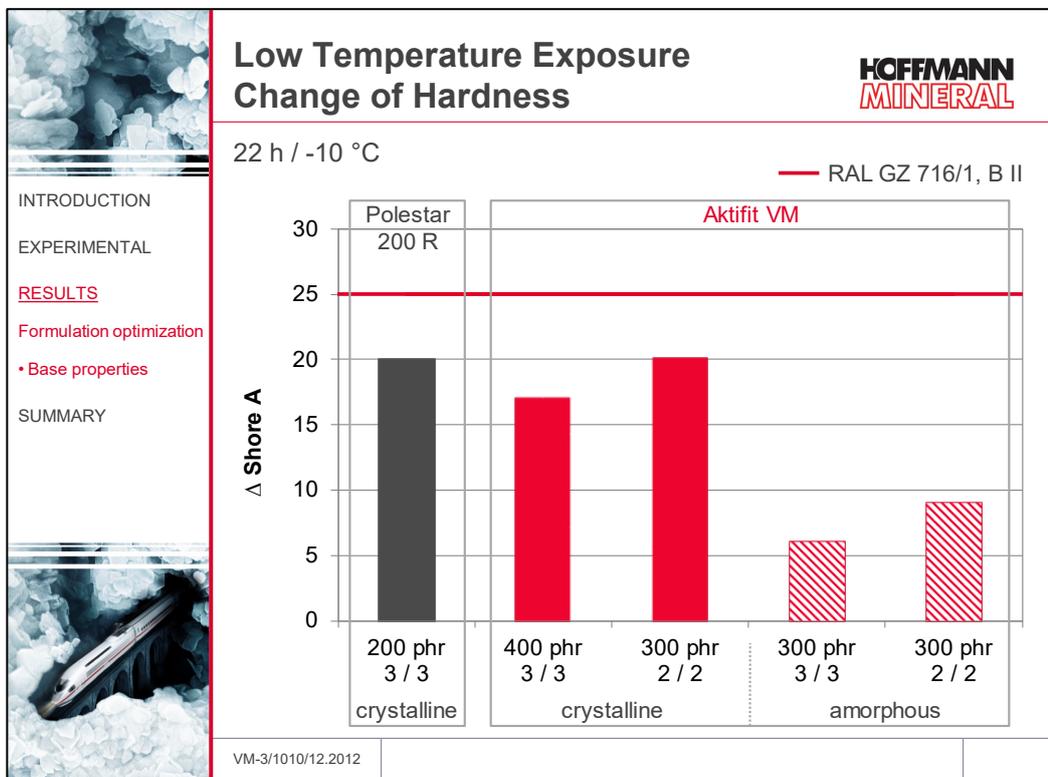


The determination of the compression set was run under the more stringent conditions at 125 °C, and the results in the graph are compared with the requirements of the RAL GZ 716/1, B II for 22 hours at 70 °C. It is very evident that even at the higher test temperature results largely below 50 % can be obtained. This means, the principal function "sealing over long periods of time" has been further improved.

In the crystalline EPDM grade, the increased loading of Aktifit VM results in an evident improvement of the compression set compared with the reference compound. This time, the result with 400 phr Aktifit VM and the 3 / 3 peroxide blend comes off comparable with the 300 phr plus 2 / 2 peroxide blend compound. By using the amorphous polymer, the compression set can be further lowered. The lowest compression set, i.e. about half the level of the reference compound, is obtained with 300 phr Aktifit VM and the 3 / 3 peroxide blend.



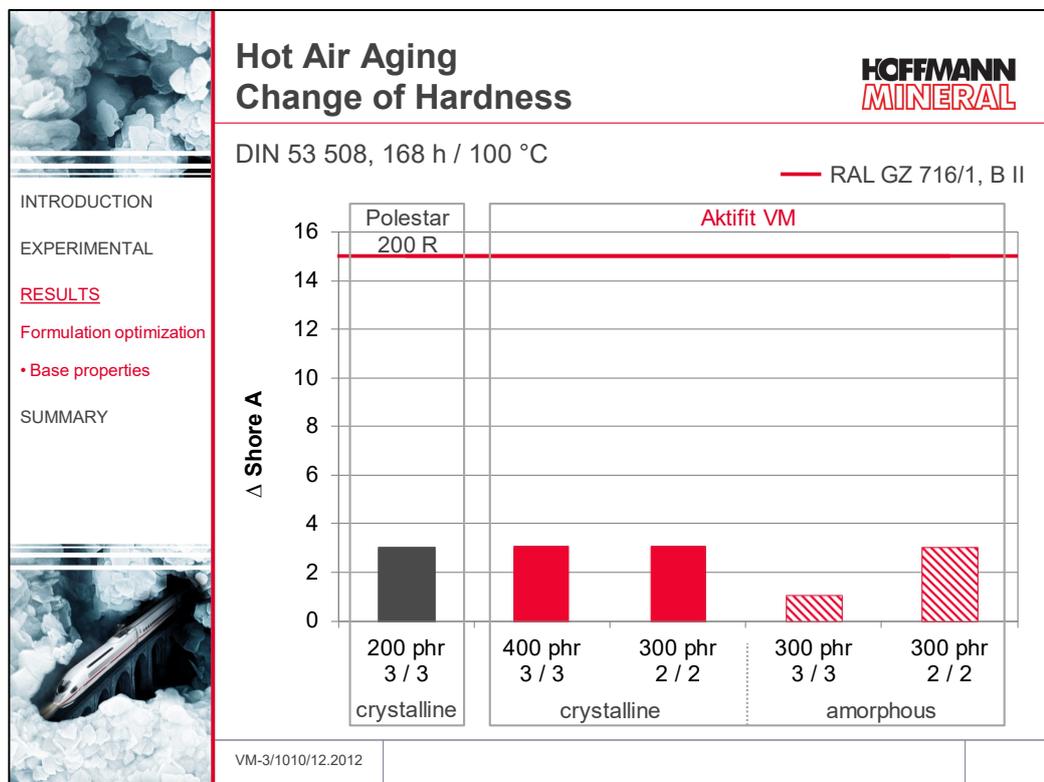
Likewise, all compounds are able to satisfy the specification at the deflection temperature of 23 °C. The results at lower level are widely comparable with what has been found at 125 °C. However, here marked difference becomes evident between the crystalline and the amorphous EPDM grades. When using the 3 / 3 peroxide blend in the amorphous polymer with a loading of 300 phr Aktifit VM, a compression set of distinctly below 10 % can be realized.



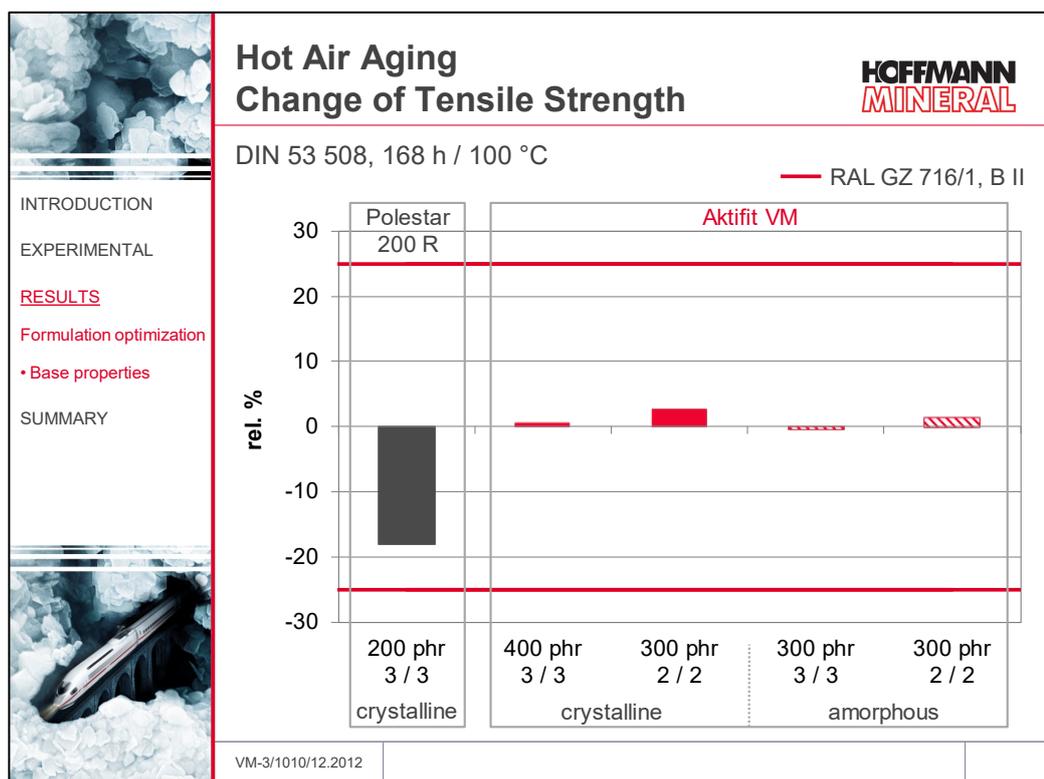
Still more evident is the influence of the polymer grade during the exposure to low temperature. 300 phr Aktifit VM along with the 2 / 2 peroxide blend in the crystalline EPDM come up to the same level of hardness change after exposure to cold temperature as the reference compound, which still is able to meet the requirement of the RAL GZ 716/1, B II standard. By increasing the filler loading to 400 phr and increasing the addition of the peroxide blend to 3 / 3, the change in hardness can be somewhat reduced. Largely better

results, however, are obtained by replacing the crystalline with an amorphous EPDM rubber.

Again for the hardness change during low temperature exposure, the compound with 300 phr Aktifit VM and the higher peroxide concentration ranks best with only an increase of 6 Shore A. In general, the higher peroxide content seems to lead to lower levels of hardness change.

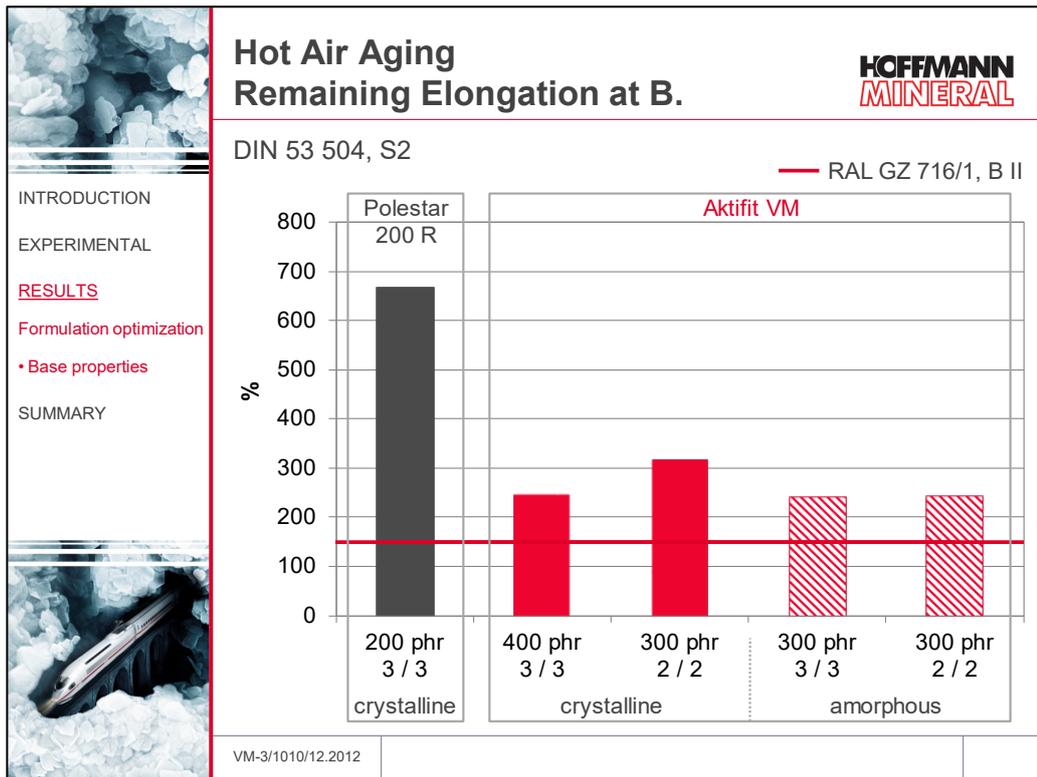


With regard to hardness changes after hot air aging, no significant difference can be observed between the individual compound variations. All compounds are able to satisfy the requirement of the standard without problems.



All compounds loaded with Aktifit VM during hot air aging give  $\pm 0$  % changes in tensile strength, whereas the reference compound comes out with a double-digit negative result.

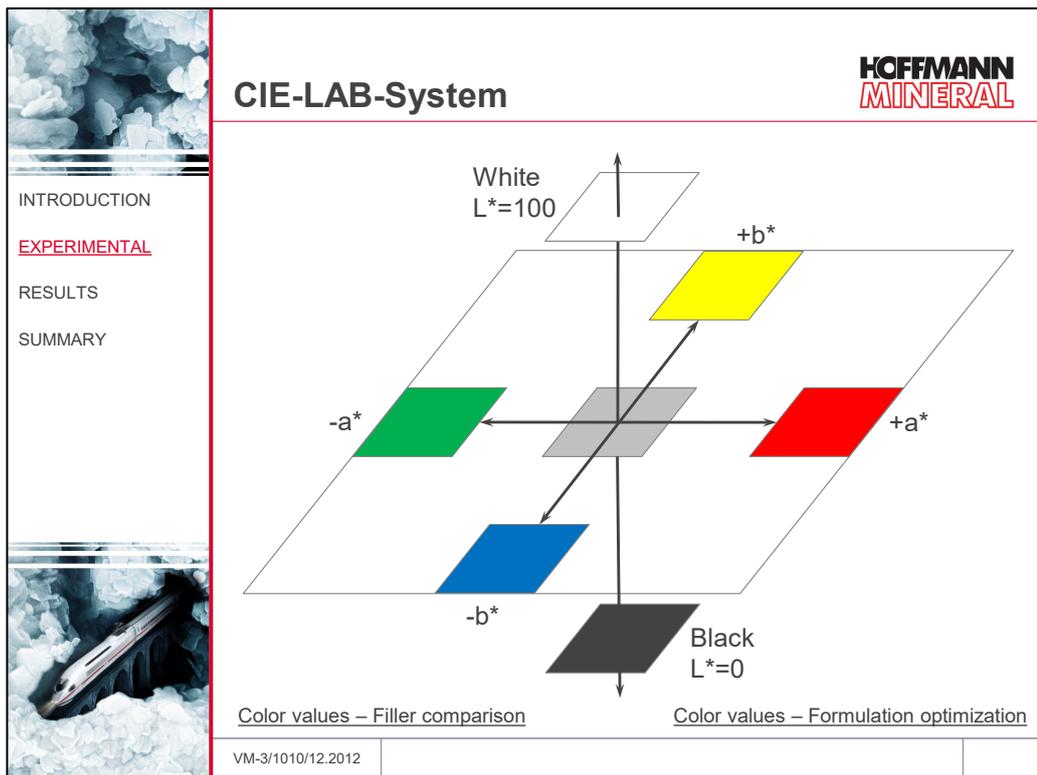
This means, the requirement of RAL GZ 716/1, B II can be easily met with all compounds containing Aktifit VM.

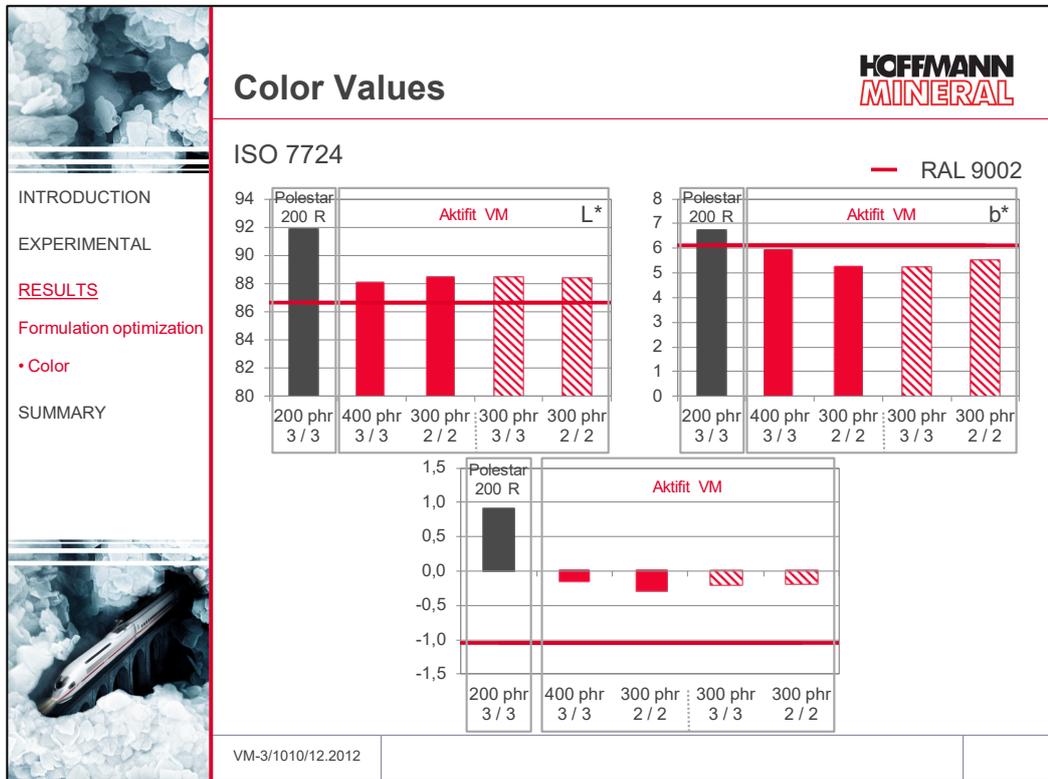


Also after hot air aging all compounds loaded with Aktifit VM reach the elongation at break specified in the pertinent standard. The overall picture is very similar to the situation prior to aging, almost without any change.

#### 4.4 Color

The color determinations were carried out with the spectral photometer Luci 100 (Dr. Lange) with light D65 and a measuring geometry d/8° (without gloss trap) and a normal observation angle of 10°. Each time, the L\*, a\* and b\* values were registered.





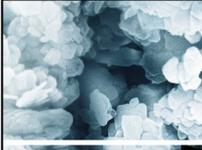
In spite of the loading level increased to 400 phr, Aktifit VM is capable to obtain the  $b^*$  value as required for the grey-white color of RAL 9002. At the same time, the brightness, indicated by the  $L^*$  result, comes very close to the specification requirement, and  $a^*$  remains in the negative region, as also specified in RAL 9002.

The compounds with 300 phr Aktifit VM with regard to brightness and  $a^*$  do not indicate any significant difference, but reach a lower  $b^*$  which stands for a lower yellowish tint.

In difference to the reference compound with Polestar 200 R, the Aktifit VM compounds, therefore, offer a markedly decreased yellowish and no reddish tint, which overall results in a better color neutrality.

## 4.5 Extrusion properties

A qualitative assessment of the extrusion properties was made with the help of the Garvey extrusion test. Apart from the usual determination of the output by length, the special geometry of the die orifice allows to assess simultaneously edge formation, surface quality and die swell of the extrudates.



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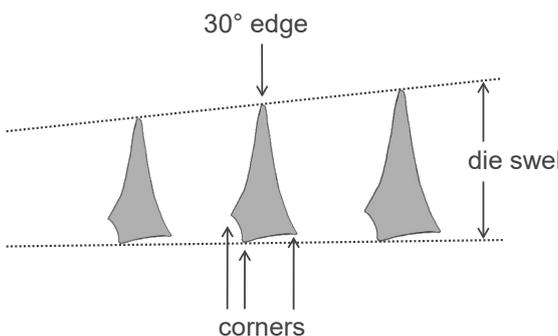
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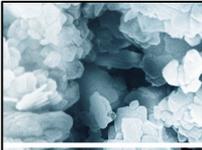
### Garvey Extrusion





VM-3/1010/12.2012

The following table summarizes the parameters of the extrusion test.



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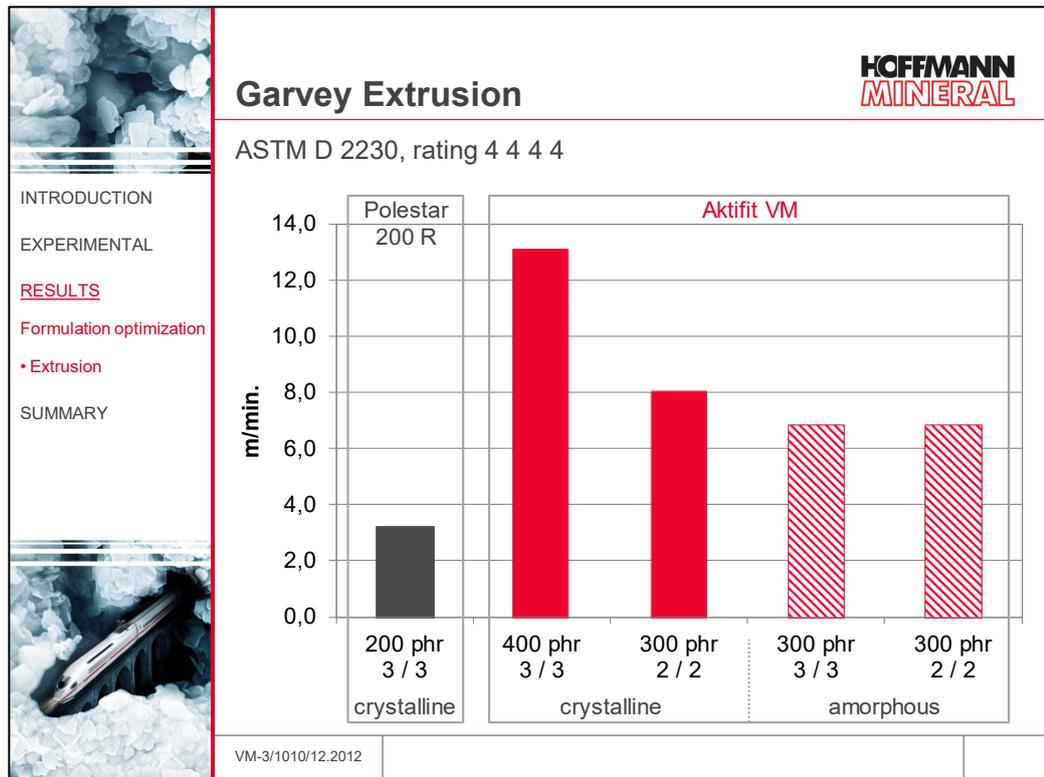
### Garvey Extrusion

|  |       |                               |
|--|-------|-------------------------------|
| Extruder                                     |       | Schwabenthan Polytest 30 R    |
| Screw diameter                               | [mm]  | 30                            |
| Process length                               | [mm]  | 450                           |
| Temperature set point head / zone 1 / zone 2 | [°C]  | 110 / 70 / 70                 |
| Screw speed                                  | [rpm] | adjustable                    |
| Garvey profile                               |       | see picture                   |
| Rating figure 1                              |       | die swell                     |
| Rating figure 2                              |       | 30° edge                      |
| Rating figure 3                              |       | surface                       |
| Rating figure 4                              |       | corners                       |
| Objective of extrusion                       |       | max. output for a 4444-rating |



VM-3/1010/12.2012

For each compound, the maximum possible output for a "4444" rating was determined (4 is the best rating after Garvey, "4444" therefore indicates excellent die swell, 30° edge formation, corners and surface quality).  
 The assessment was carried out on uncured profiles according to ASTM D2230.



The comparison clearly shows that the compounds with Aktifit VM allow markedly higher haul-off speed rates than with the reference compound which contains Polestar 200 R as a filler. The compound with 300 phr Aktifit VM even gives twice the haul-off speed of the control. By using the crystalline EPDM polymer, this rate can further be increased somewhat above the amorphous EPDM grade. With 400 phr Aktifit VM the haul-off speed is increased fourfold, while an optimally shaped profile with an outstanding surface quality is obtained.

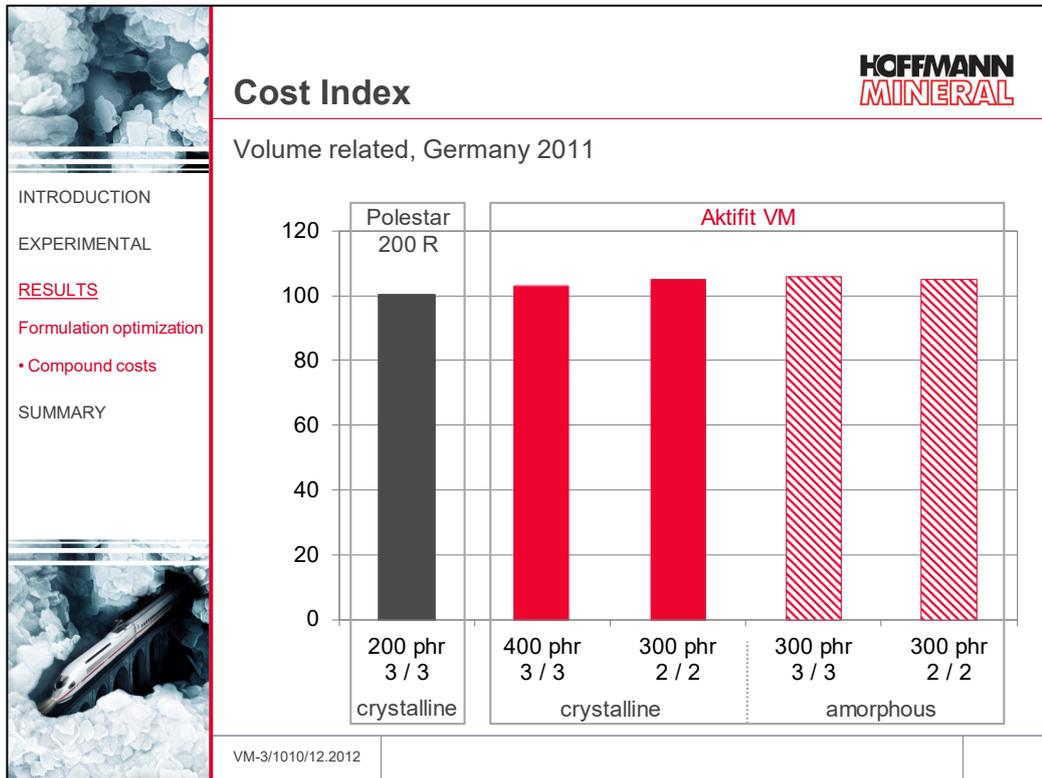
In combination with the short time to full cure - short conversion time  $t_{90}$  - this would allow to increase the throughput within a given time period without having to worry about setback in the profile quality and properties.

## 4.6 Compound raw material costs

A further important consideration besides the processing and cured rubber properties, no doubt, will refer to the costs necessary to produce the compounds.

For this reason, a compound cost calculation relative to volume was carried out for the compounds discussed in this section. The prices mentioned are those valid for Germany in the first half of 2011. At that time, the trend for important components, such as EPDM rubber, plasticizer oils and titanium dioxide was directed clearly upwards and some shortages in supply too.

To simplify the task, cost indices were used with the Polestar 200 R loaded reference compound as the base.



The plot clearly shows that in spite of the higher price of the Aktifit VM, the costs of the individual compound variations change only to a marginal extent. The compounds with 300 phr Aktifit VM come out around an increase of 5 %, and the higher loading of 400 phr allows even to cut that figure in half.

On the other side, potential reductions in the production process, in particular the higher extrusion rates, should be able to more than compensate the slightly higher raw material costs.

## 4.7 Summary section 4

At a loading of 300 phr, Aktifit VM in both EPDM rubbers, with a 2 / 2 peroxide blend gives better mechanical properties, in particular a lower compression set compared with the reference compound loaded with Polestar 200 R. In addition, through reduction of the reddish and yellowish tint, a better color neutrality is offered by Aktifit VM which allows to better approach the RAL color specification.

The best compression set and the minimum hardness change at low temperature will be obtained with 300 phr Aktifit VM and the 3 / 3 peroxide blend in the amorphous EPDM rubber. The reddish and yellowish tint are further reduced in comparison with the reference compound, so with Aktifit VM the RAL 9002 will be more closely approached.

The slight increase of the volume related compound costs vs. the reference compound with Polestar 200 R should be easily compensated by increased productivity in the extrusion step in view of twice the haul-off speed with 300 phr Aktifit VM.

A smaller increase of the compound costs vs. the reference compound with Polestar 200 R along with a fourfold increase of the extrusion throughput is possible by working with 400 phr Aktifit VM and the 3 / 3 peroxide blend in the crystalline EPDM polymer. In addition, despite the twofold increase of the filler loading vs. the reference compound, the reddish and yellowish tint will be minimized, which allows to distinctly come closer to the RAL 9002 specification. Further reasons which speak in favor of this compound variation are the slightly improved hardness change after exposure to low temperature and also the better compression set compared with the reference compound which is loaded with Polestar 200 R.

Overall Aktifit VM offers a combination of properties never realized before, i.e. outstanding extrusion properties, satisfactory tensile strength - even with the amorphous EPDM grade - and an excellent low compression set which by working with amorphous EPDM can also be obtained at low temperatures along with a small change of hardness. In spite of the higher degree of loading, the color neutrality in comparison with the calcined clay is maintained so that the color comes close to the grey-white specification of the RAL 9002 standard.

## 5 Overall summary

The study has shown that Aktifit VM can be used in white or grey-white building profiles without causing a significant increase of the compound costs.

Rather the replacement of calcined clay with the surface treated Calcined Neuburg Siliceous Earth grade Aktifit VM offers improvements of mechanical properties, such as the hardness change upon exposure to low temperature or the compression set. Even a simultaneous reduction of the peroxide level does not constitute a hindrance to realize outstanding properties.

It has further been shown that in spite of a twofold increase of the filler loading with Aktifit VM vs. the calcined clay a lower reddish and yellowish tint is obtained, which means a better color neutrality.

In addition, Aktifit VM makes possible an increased productivity in the plant over calcined clay, as the extrusion performance will be improved, i.e. the output rate can be increased without affecting the profile shaping and smoothness.

In conclusion it can be stated that because of distinct improvements of the property profile Aktifit VM represents an excellent alternative to calcined clay in grey-white building profiles as specified in RAL GZ 716/1, elastomer class B II.

*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.*