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Unternehmensgruppe Hoffmann

Effect of Aktisil VM 56

in radiator hose

according to VW TL 523 61

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VM-2/0500/08.2011

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1 Introduction

Radiator hose in service have to withstand manifold and severe stress and exposure conditions. This explains the stringent requirements as specified by the automotive Industry. The Volkswagen specification TL 523 61 «Radiator water hoses with aramide reinforcement» represents a particularly difficult example.

The materials to be used have to offer, among others, low temperature flexibility and heat resistance, must be resistant against oils and cooling liquids as well as against ozone, and show a low compression set. Furthermore, in the interest of avoiding a possible electrochemical corrosion, a low electrical conductivity is preferred. Straight carbon black loadings, therefore, cannot be used. In order to arrive at such properties, the addition of an insulating filler is required.

The present study will contribute to the development of a suitable formulation, with the selection of appropriate fillers as the main objective.

Aktisil VM 56, a Neuburg Siliceous Earth grade surface treated with a vinyl functional group, offers itself in view of its outstanding extrusion and good mechanical properties in combination with carbon black for this application.

2 Experimental

2.1 Objectives and test design

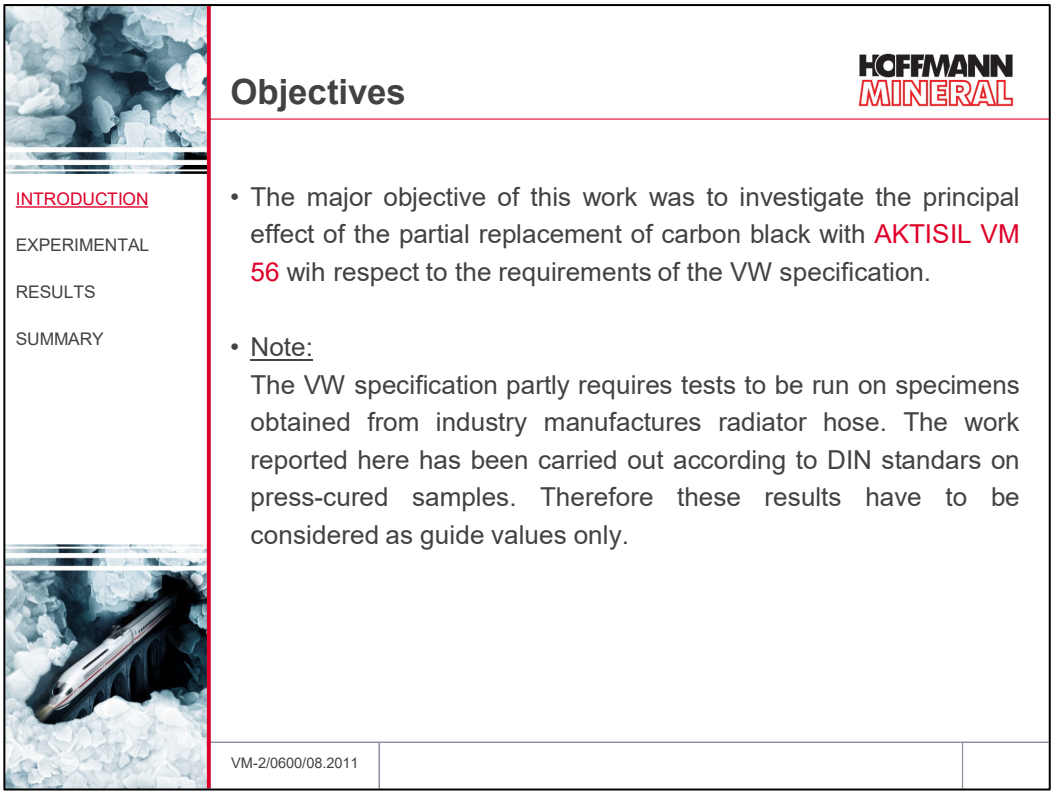


Fig. 1

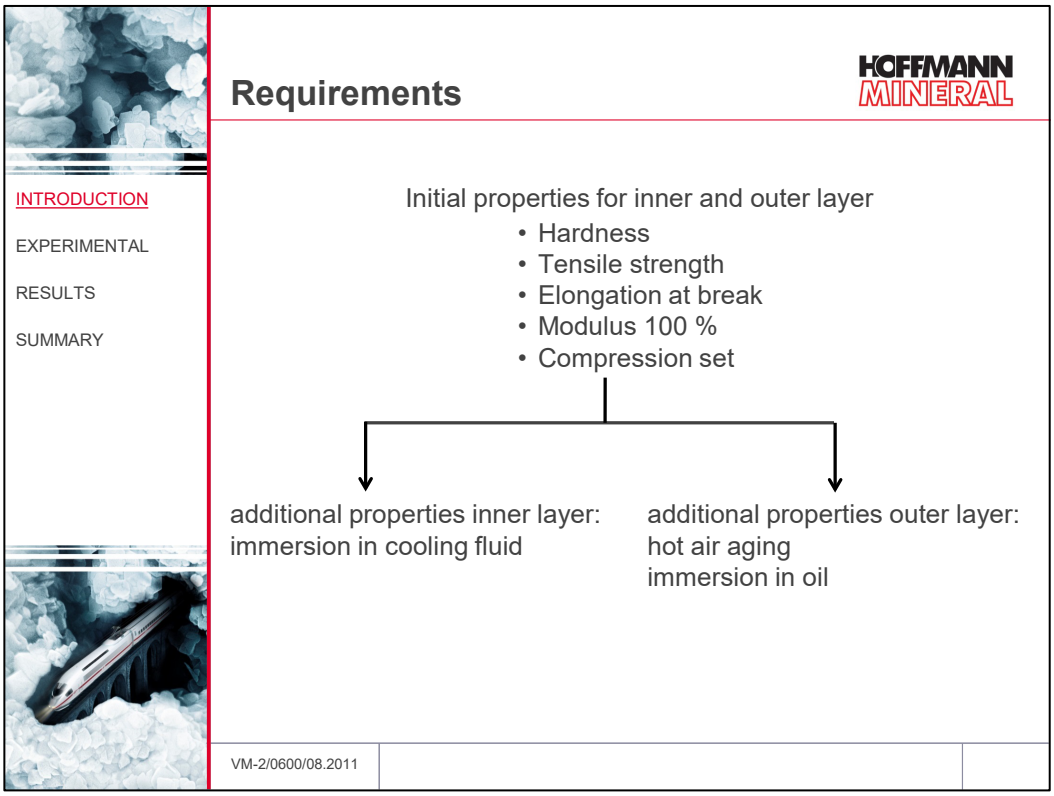
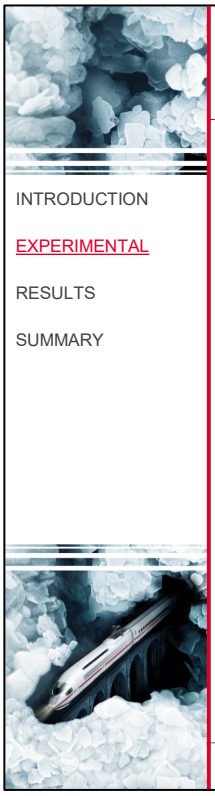


Fig. 2

The use of peroxide cured EPDM for radiator hoses represents the state of art in the industry. Sulfur/accelerator systems are mostly not able to meet the stringent requirements concerning the aging properties. As mentioned above, straight carbon black loadings must be avoided as for minimal electrochemical corrosion a low electrical conductivity is needed. The trials reported here are based on the guide formulation of Exxon Mobil *(Table 1).*



Base Formulation

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EPDM – 65 Shore A

	phr
Vistalon 7500	70.0
Vistalon 3666	52.5
Durex 0	55.0
Corax N 550	55.0
Sunpar 2280	22.5
Flectol TMQ	1.0
Rhenofit EDMA/S (70 %)	0.7
Perkadox 14/40 B	7.0
Total	**263.7**

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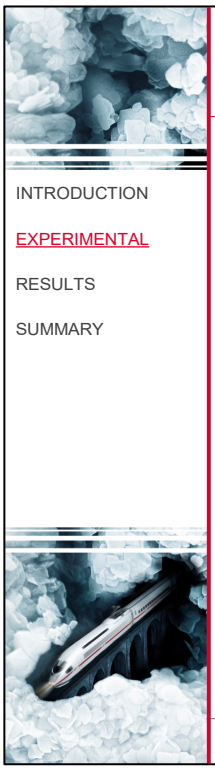
Table 1

The starting point for the filler variations is a 50/50 blend of the carbon blacks Durex 0 and Corax N 550 at a total loading of 110 phr. First, 20 phr carbon black were replaced by 40 phr Aktisil VM 56, and then 40 phr carbon black by 80 phr Aktisil VM 56. These variants were also tested with the straight (unblended) carbon blacks, in order to check the properties of their blends with Aktisil VM 56. The replacement ratio of 1 phr carbon black to 2 phr Aktisil VM 56 was established in preliminary tests which aimed at adjusting a comparable hardness *(Table 2).*



Compound Variations

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The loading of Corax N 550, Durex 0 and AKTISIL VM 56 was varied in the following way:

	phr					
Aktisil VM 56	40			80		
Corax N 550	45	90	0	35	70	0
Durex 0	45	0	90	35	0	70

As a rough guideline, it was confirmed that for equal hardness 1 part of carbon black can be replaced by 2 parts Aktisil VM 56.

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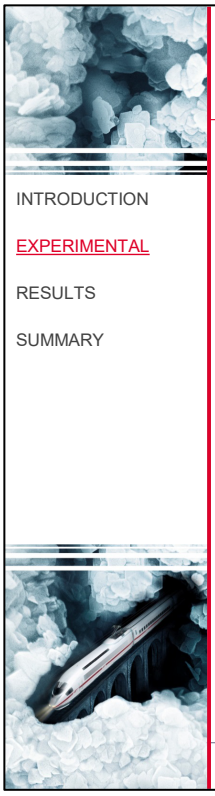
Table 2

Corax N 550 has a distinctly higher BET surface area and DPB absorption than Durex 0 or Aktisil VM 56. For Durex 0, the surface area is only slightly higher compared with Aktisil VM 56 *(Table 3).*



Fillers, Characteristics

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		Corax N 550	Durex 0	Aktisil VM 56
Density	[g/cm³]	1.8	1.8	2.6
Particle size d_{50}	[µm]			2.0
Particle size d_{97}	[µm]			9
Sieve residue >40 µm	[mg/kg]			4
Sieve residue 45 µm (Siev No. 325)	ppm	≤ 300 10	≤ 50 2	
Specific surface area BET	[m²/g]	39	18	11
DBP absorption	[ml/100 g]	121	64	
Functionalization		none	none	Vinyl

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Table 3

2.2 Preparation, curing and testing

The compounding for the cured test sheets was carried out on a laboratory mill, but the compounds for the hose extrusion trials were mixed in a laboratory mixer *(Fig. 3).*

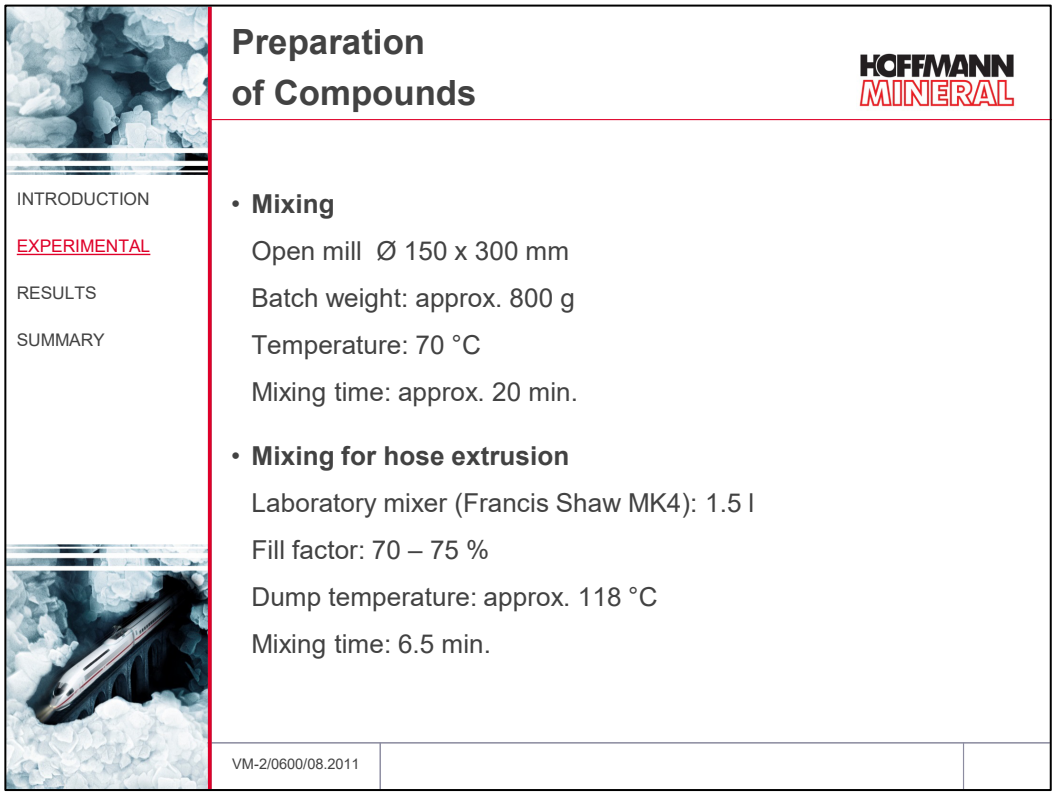


Fig. 3

The test sheets were cured as indicated in *Figure 4*. The tests were run in accordance with DIN standards, but based on the requirement parameters of the VW specification *(Fig. 4).*

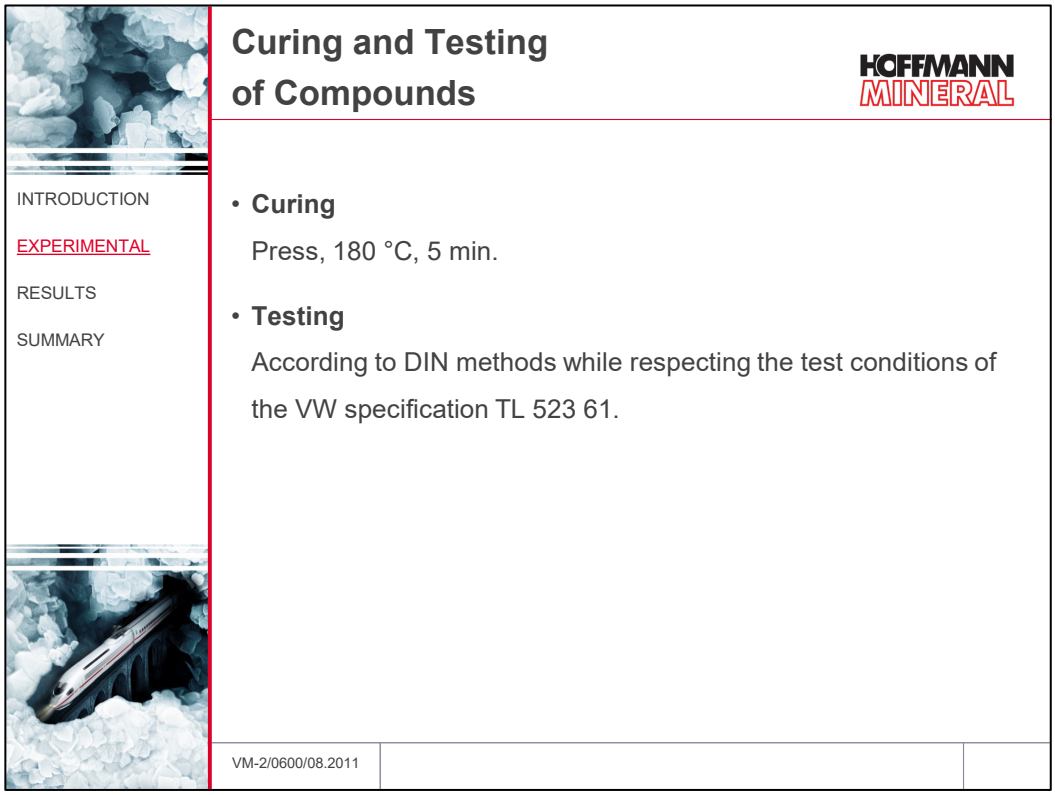
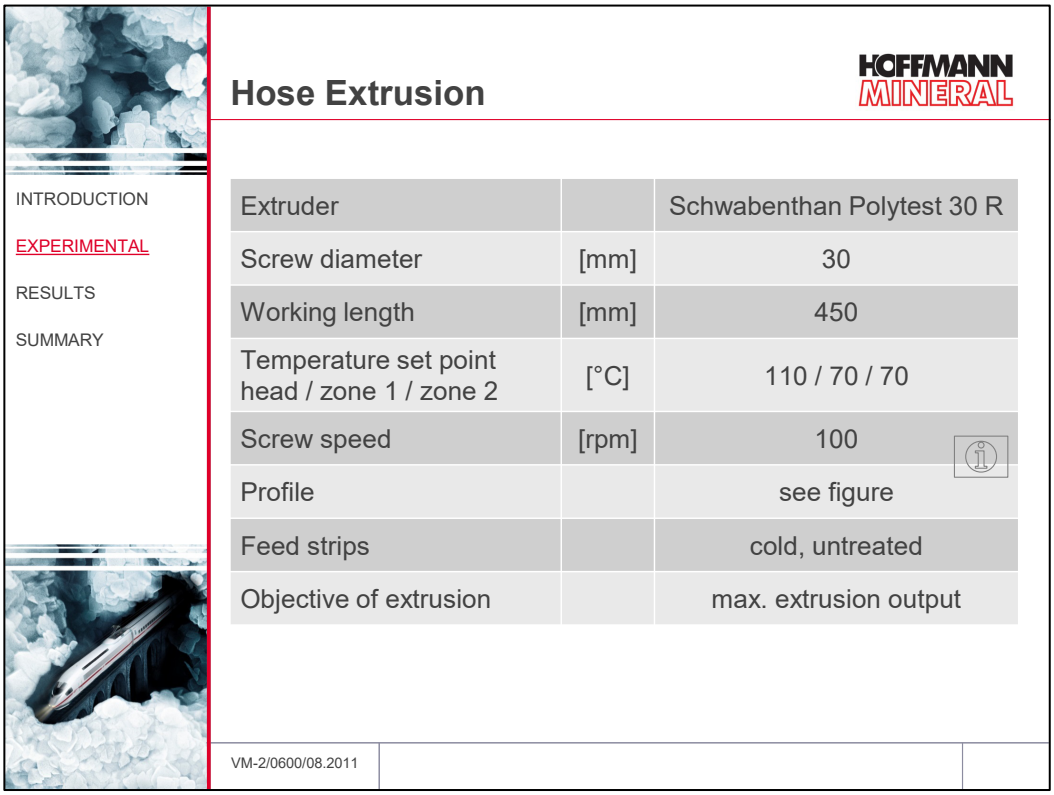


Fig. 4

The extrusion tests were run with the requirement parameters of *Table 4*, and the extruder die is shown in *Figure 5*.



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Hose Extrusion

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Extruder		Schwabenthan Polytest 30 R
Screw diameter	[mm]	30
Working length	[mm]	450
Temperature set point head / zone 1 / zone 2	[°C]	110 / 70 / 70
Screw speed	[rpm]	100
Profile		see figure
Feed strips		cold, untreated
Objective of extrusion		max. extrusion output

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Table 4

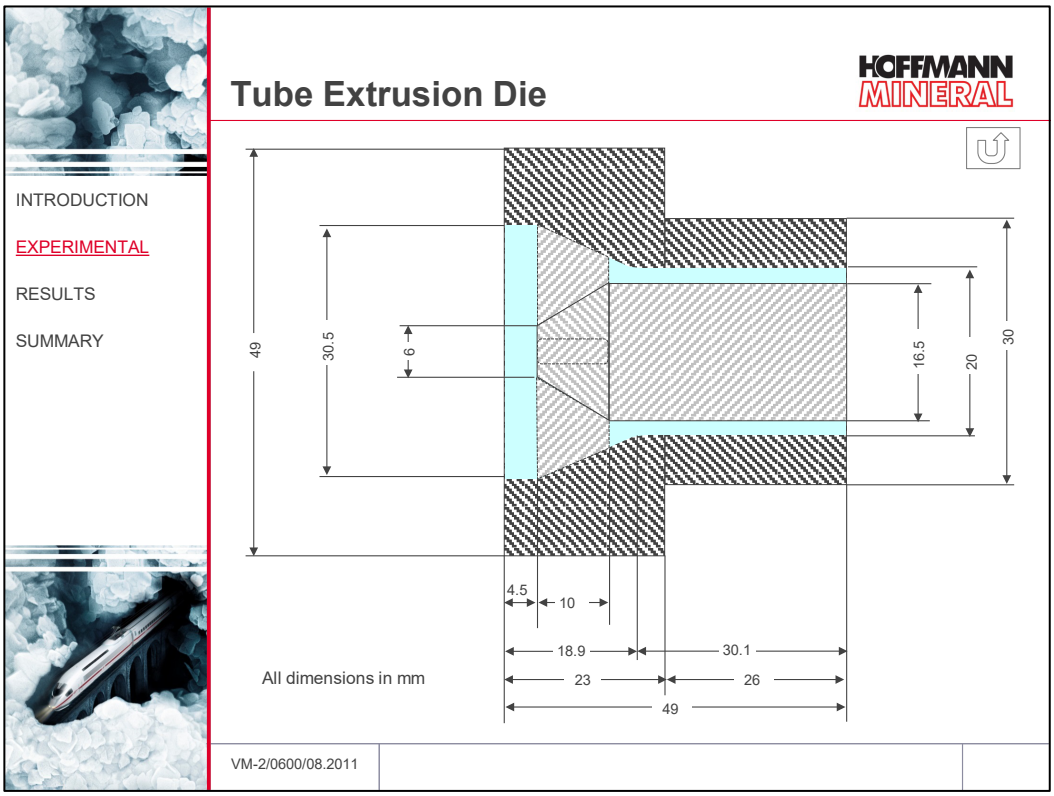
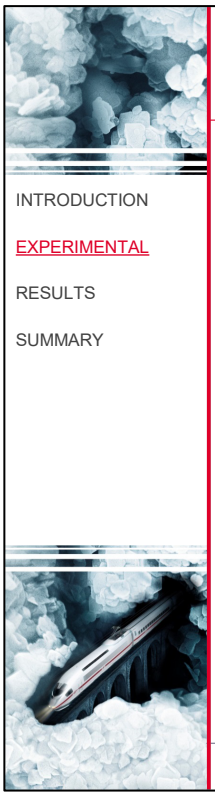


Fig. 5

The electrical volume resistance was tested with the parameters listed in *Table 5*. For the modified test method, a different device was used.



Volume Resistivity

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DIN IEC 93

Measuring instrument		Model HM 307 from Fetronic
Modified test method		Measuring instrument Model Mavo 20 from Co. Gossen
Dimension of plates	[cm]	10 x 10
Thickness of plates	[mm]	2
Electrode set-up		circular plate electrode with protective ring
Test method		voltage / amperage methode
Test voltage	[V]	1
Recording time	[min]	1
Test temperature	[°C]	23
Evaluation		$\rho = R_X * A / h$ ρ= volume resistivity in Ωcm R_X = volume resistance in Ω A = effective surface area of the protected electrode (24 cm²) h = Median thickness of test plate in cm

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Table 5

3 Results

3.1 Rheological properties

Aktisil VM 56 tends to bring about a reduction of viscosity, while the blend with straight Durex 0 leads to a slight increase *(Fig.6)*.

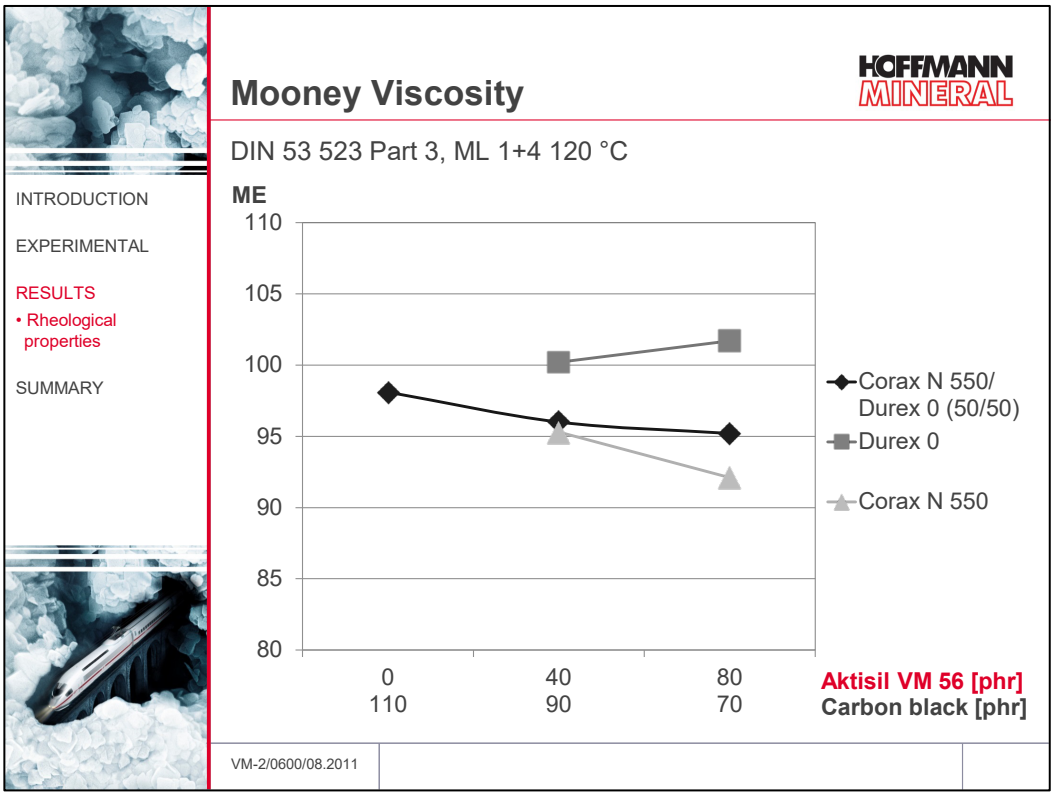

Fig. 6

With increasing addition of Aktisil VM 56 the Mooney scorch time is shortened. In the blend with straight Durex 0, however, the results come out at a markedly higher level. Blended with straight Corax N 550, Aktisil VM 56 shows no effect at all *(Fig. 7)*.

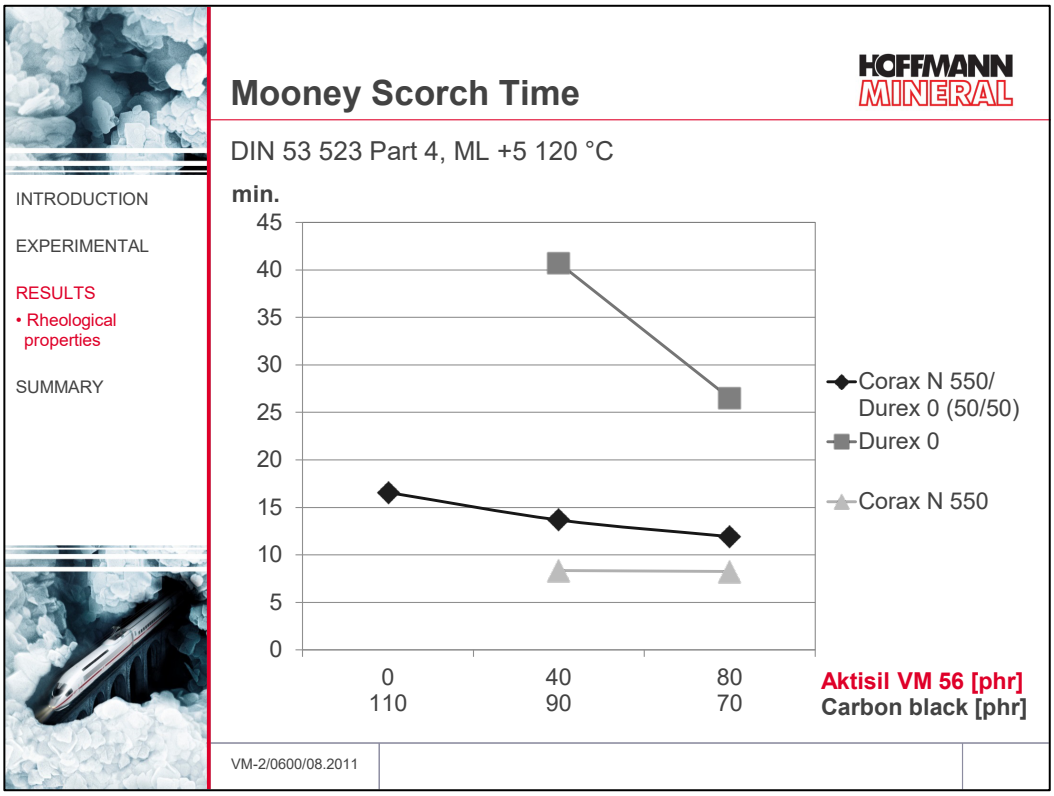

Fig. 7

The conversion time t_5 as an indicator for the onset of cure is slightly reduced by Aktisil VM 56 *(Fig. 8)*.

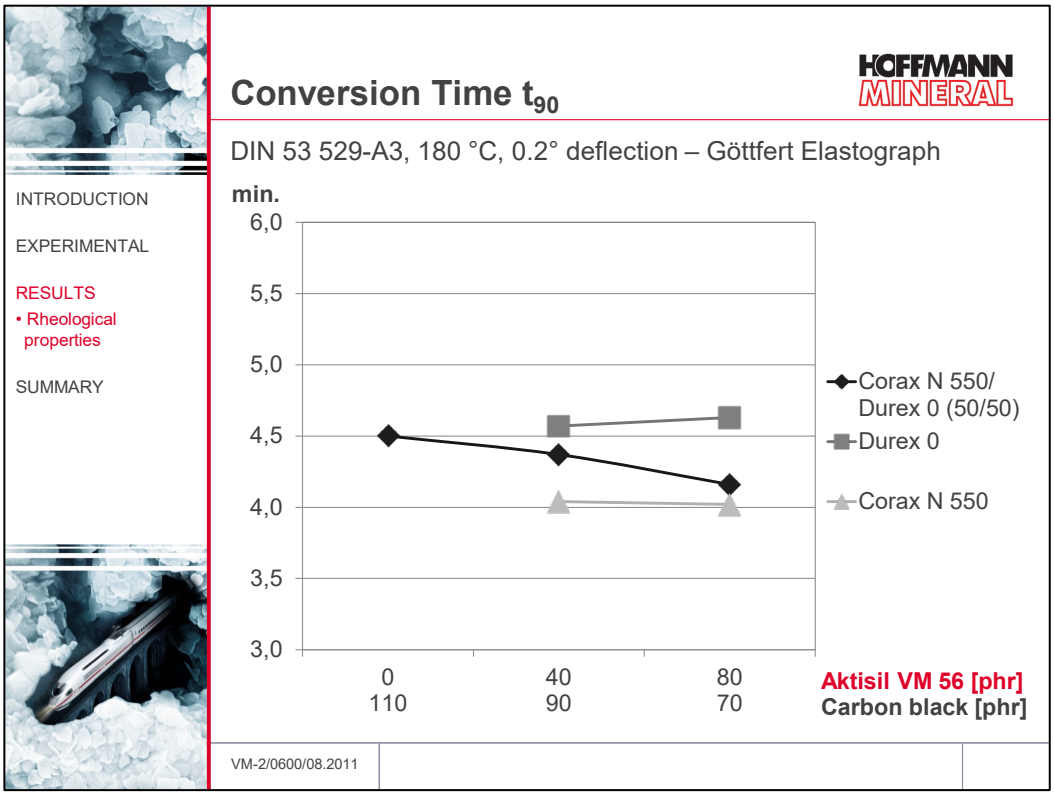

Fig. 8

The maximum cure rate for a given carbon black grade will not be changed significantly by Aktisil VM 56 *(Fig. 9)*.

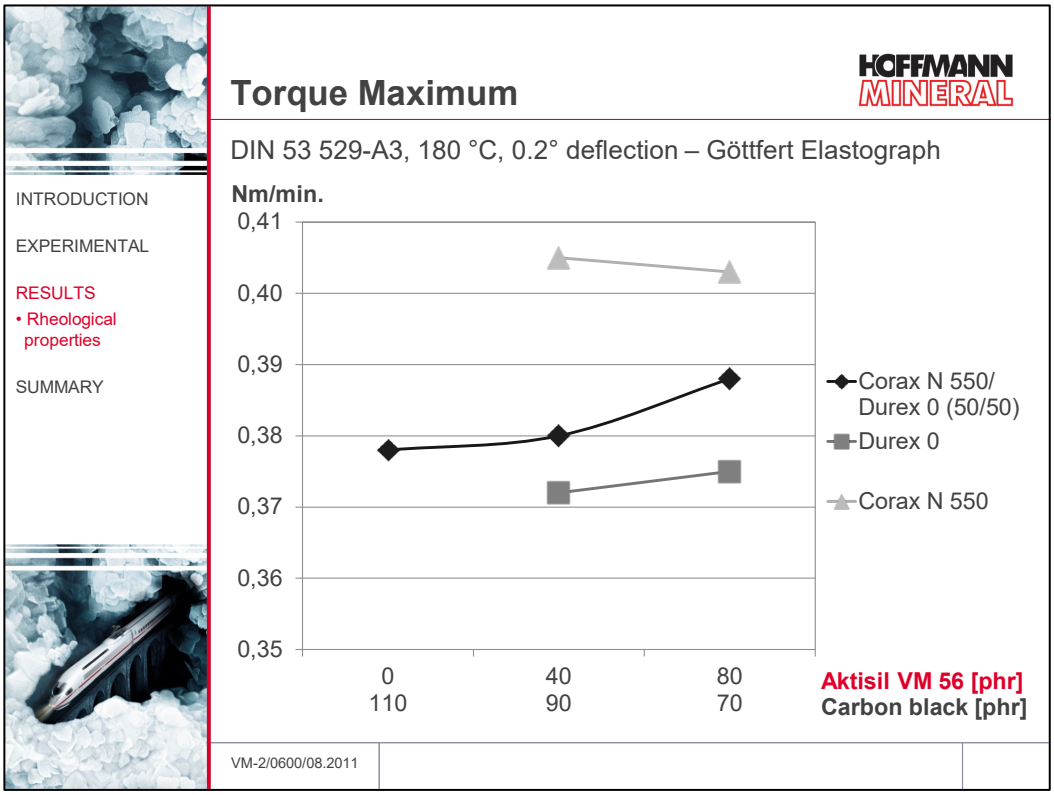

Fig. 9

The conversion time t_{90} as an index for the time to full cure is practically not affected by Aktisil VM 56. Only at the high Aktisil VM 56 loading in the blend with carbon black, a small reduction is observed. Therefore, the cure time for all filler variations and test samples was set at 5 minutes *(Fig. 10)*.

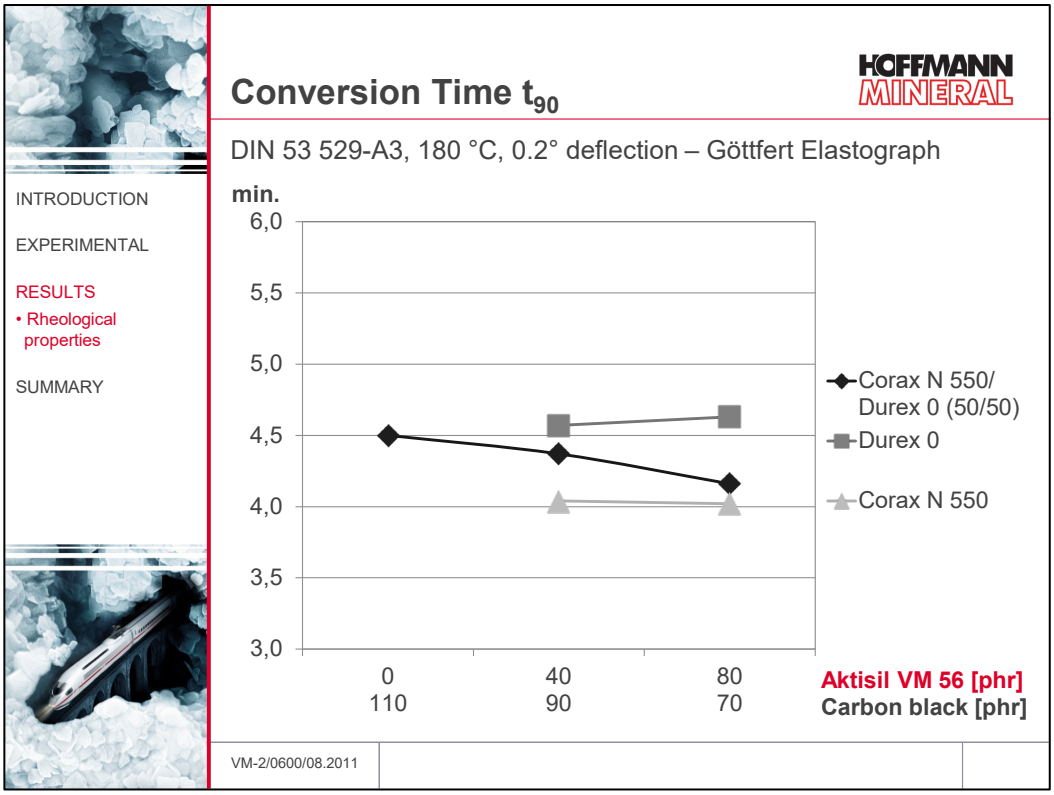

Fig. 10

3.2 Mechanical properties

Nearly equal hardness results are obtained with the chosen replacement ratio of 1:2 for carbon black vs. Aktisil VM 56. Only the blends with Durex 0 generate slightly higher results *(Fig.11)*.

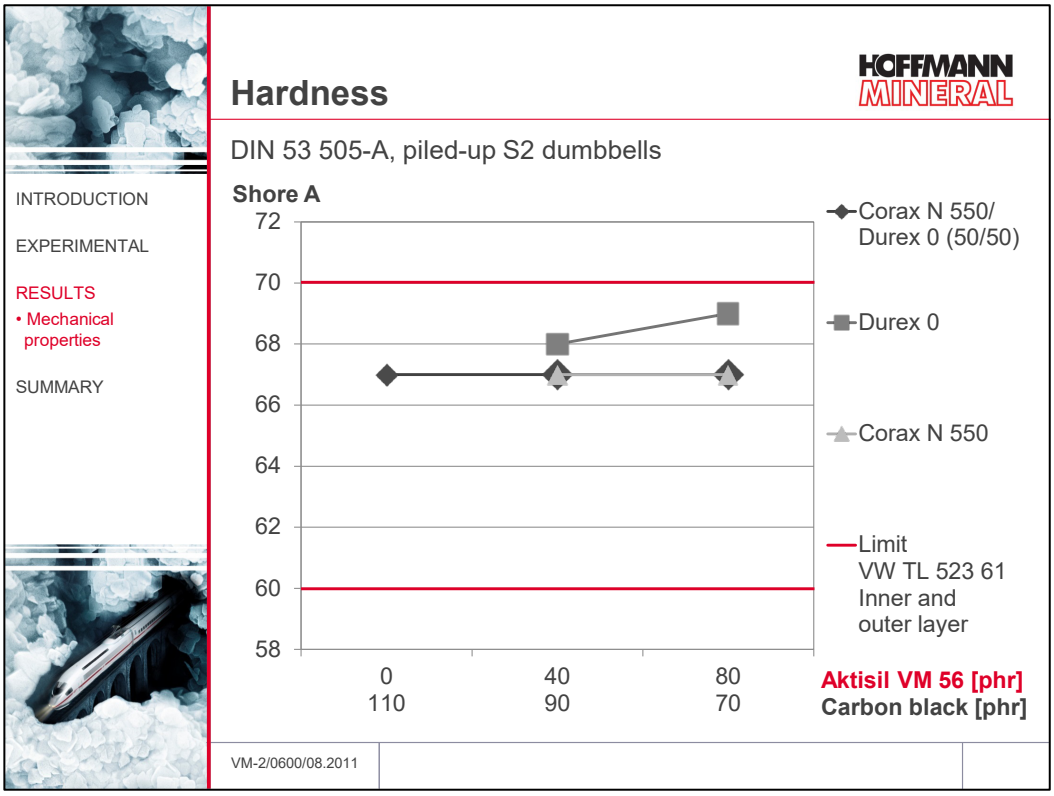

Fig. 11

With increasing amounts of Aktisil VM 56, there is a trend in the direction of lower tensile strength levels, but the results still remain above the specification limit of 10 MPa *(Fig.12)*.

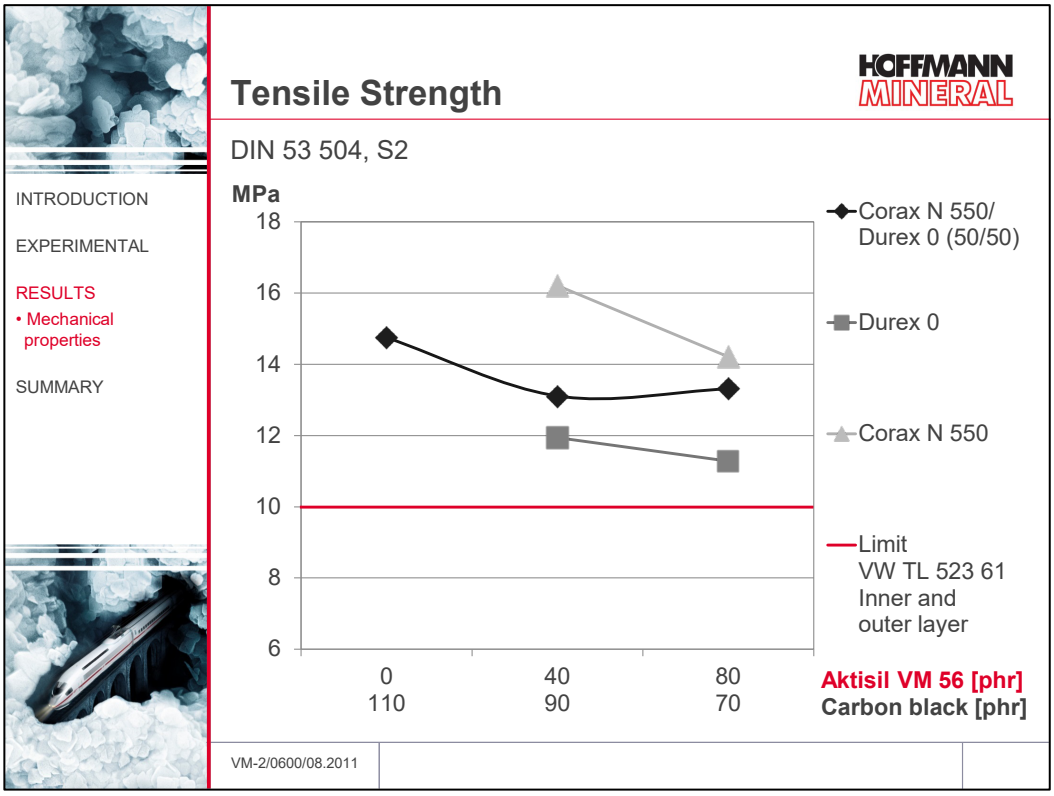

Fig. 12

The elongation at break is reduced with Aktisil VM 56, but even at high additions in combination with straight Durex 0 or the carbon black blend it remains above the specified limit of 300 % *(Fig.13)*.

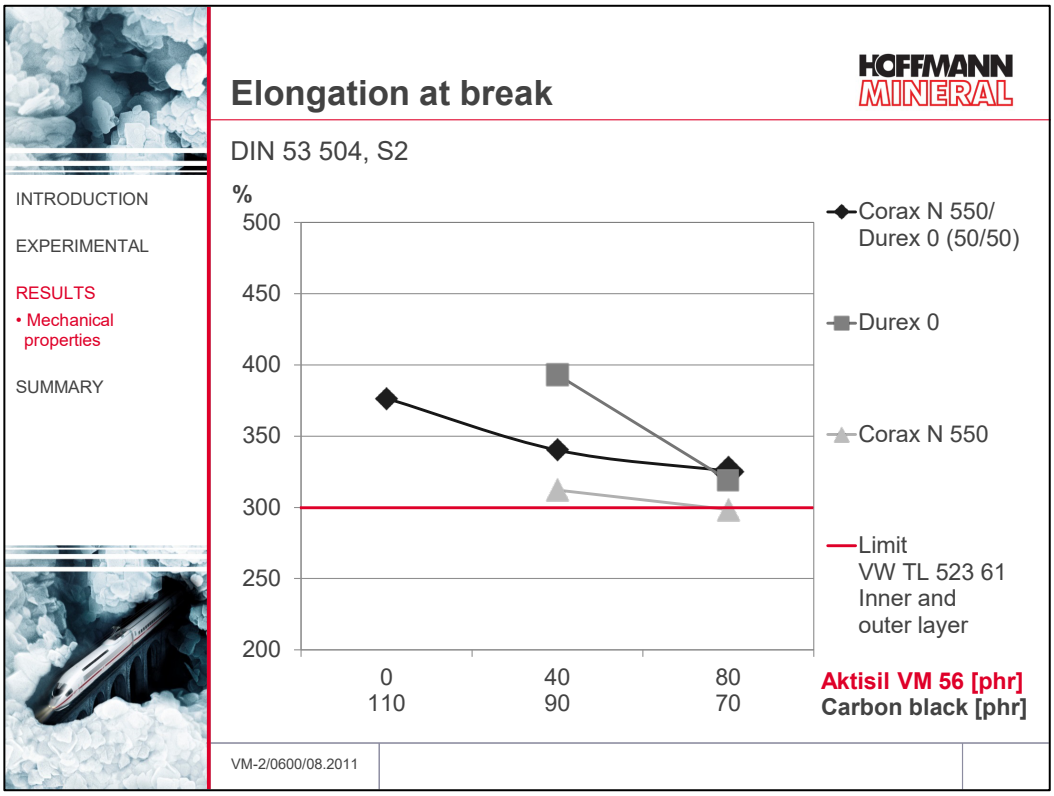

Fig. 13

The blend of Durex 0 with Aktisil VM 56 shows very favorable results with regard to the tensile modulus. This opens chances to apply changes in the formulation which tend to reduce the modulus. For instance, the plasticizer loading could be increased, which would have positive effects on compound viscosity, hardness and elongation at break *(Fig.14)*.

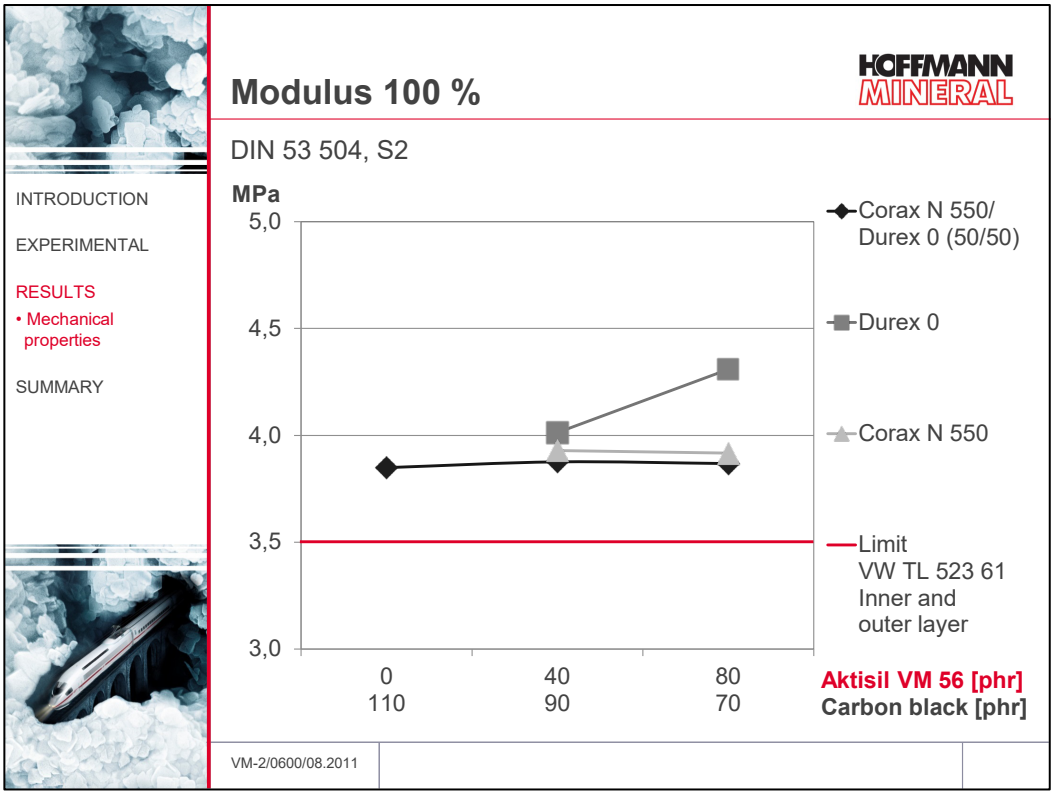

Fig. 14

The tests were run on samples and with the equipment as specified in DIN 53517-I, but with the parameters of the VW standard PV 3307. An exception was made for the time between unloading and measuring the test samples. For working reasons, contrary to the VW specification, a time of about 1 minute had to be observed here.

Aktisil VM 56 gives rise to a trend versus reduced compression set results. The small graphical increase for blends with the straight carbon blacks can be attributed to the repeatibility of the tests.

Most important, the addition of Aktisil VM 56 still gives results below the limit of the specification, which therefore remains fulfilled *(Fig. 15).*

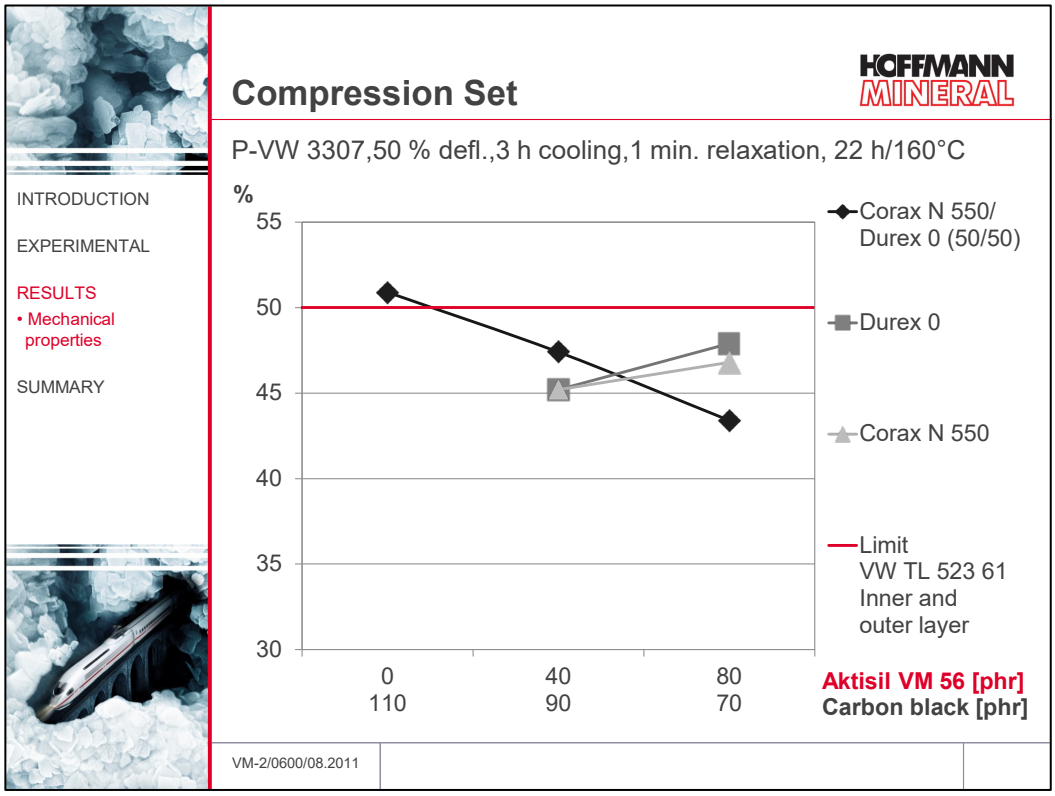

Fig. 15

3.3 Hot air aging

3.3.1 94 h / 160 °C

Aktisil VM 56 seems to give origin to a lower change in hardness, but the differences come out at the limit of test precision. It is noteworthy, however, that the trend with Aktisil VM 56 points away from the specification limit into the region of fulfilled specification requirements *(Fig.16).*

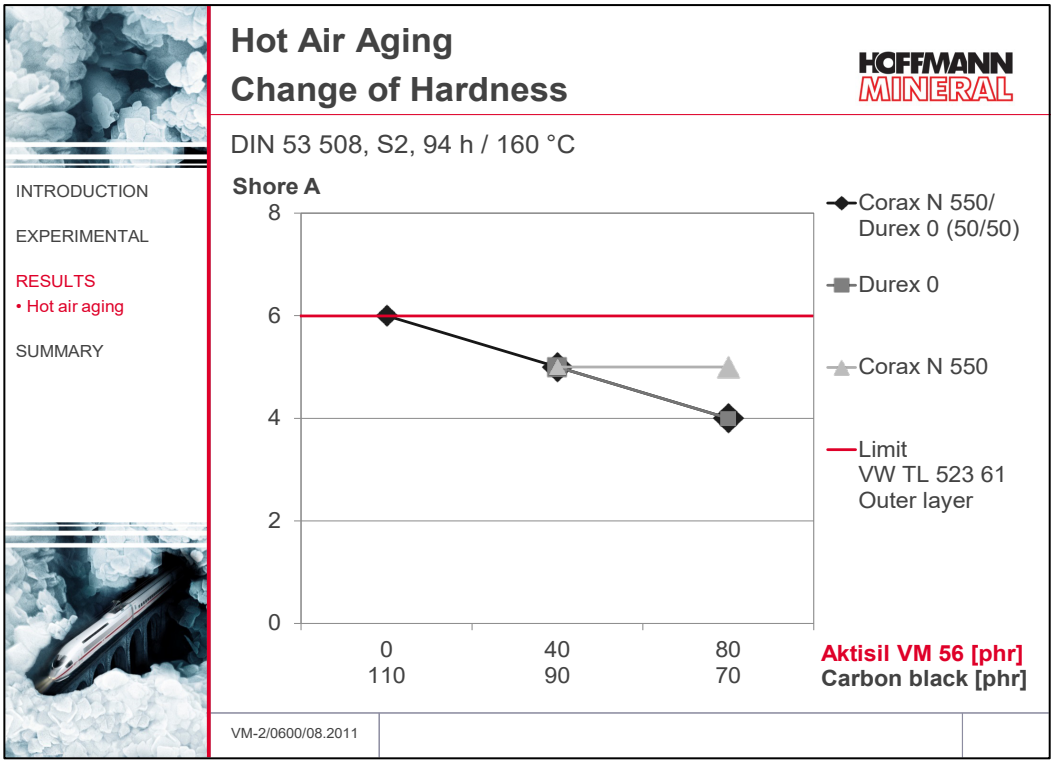

Fig.16

The results give evidence of two tendencies. The addition of Aktisil VM 56 to the carbon black blend practically does not change the tensile strength after hot air aging, while the combination of Aktisil VM 56 with the straight blacks gives rise to a decrease. All results, however, come out above the specification profile *(Fig. 17).*

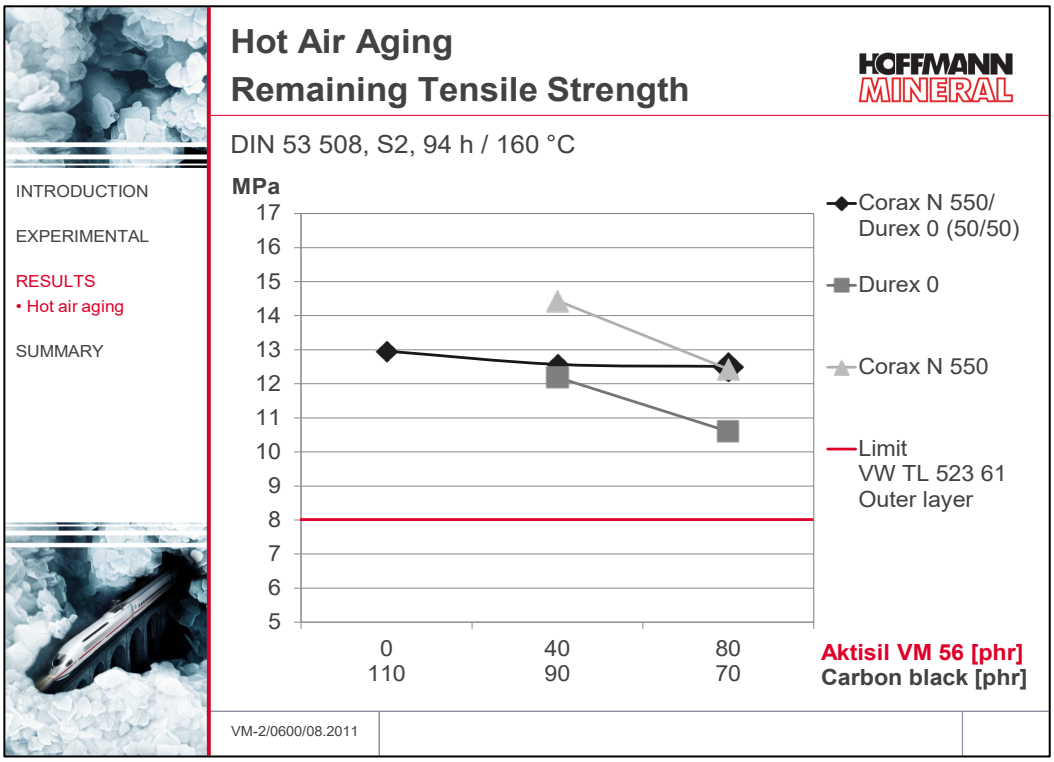

Fig. 17

As already found for the original elongation at break, the results after hot air aging become lower with Aktisil VM 56. However, the blend of 90 phr Durex 0 and 40 phr Aktisil VM 56 is able to meet the specification requirements *(Fig. 18)*.

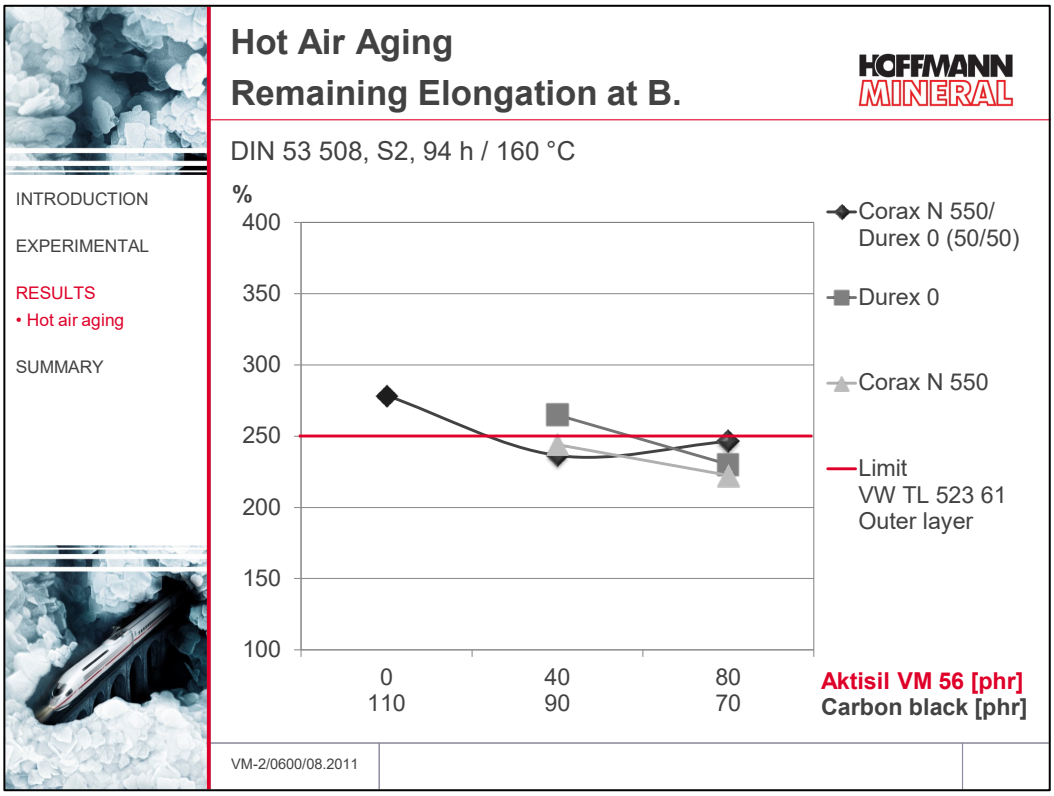

Fig. 18

3.3.2 168 h / 150 °C

Aktisil VM 56 exerts no significant effect on the hardness results after hot air aging *(Fig. 19)*.

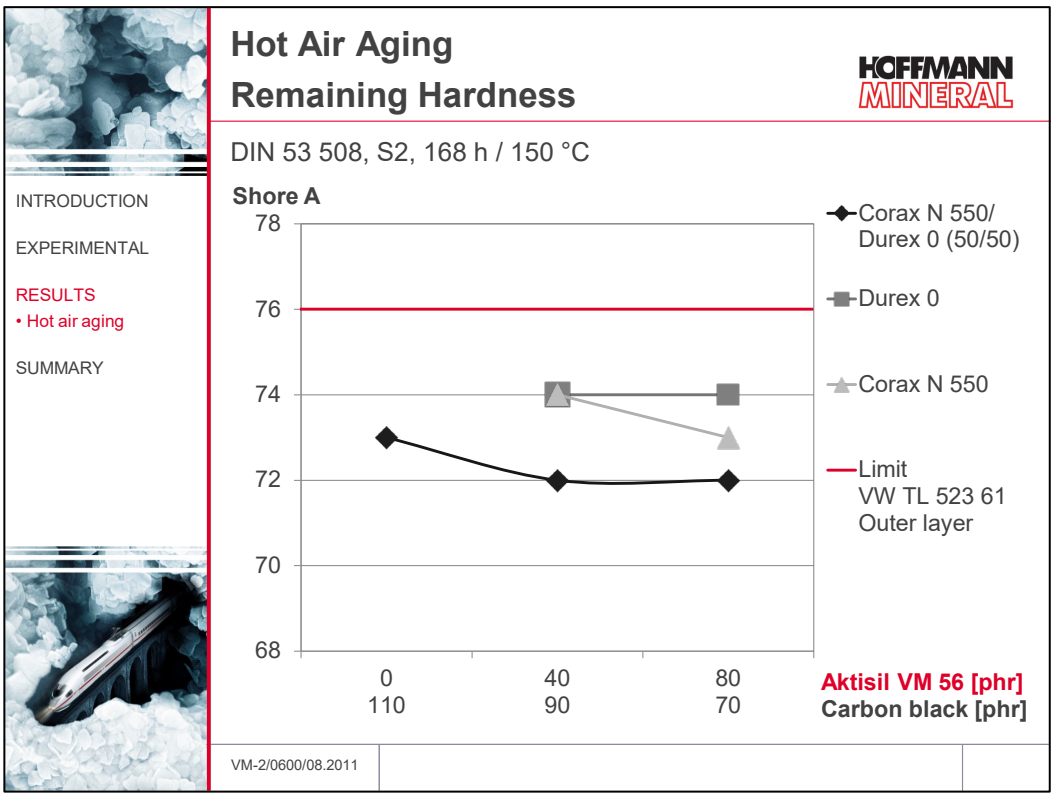

Fig. 19

Again, with increasing Aktisil VM 56 content, there is a trend towards lower results for the tensile strength after hot air aging at 150 °C, which however remain distinctly higher than the specified limit of 8 MPa *(Fig. 20)* .

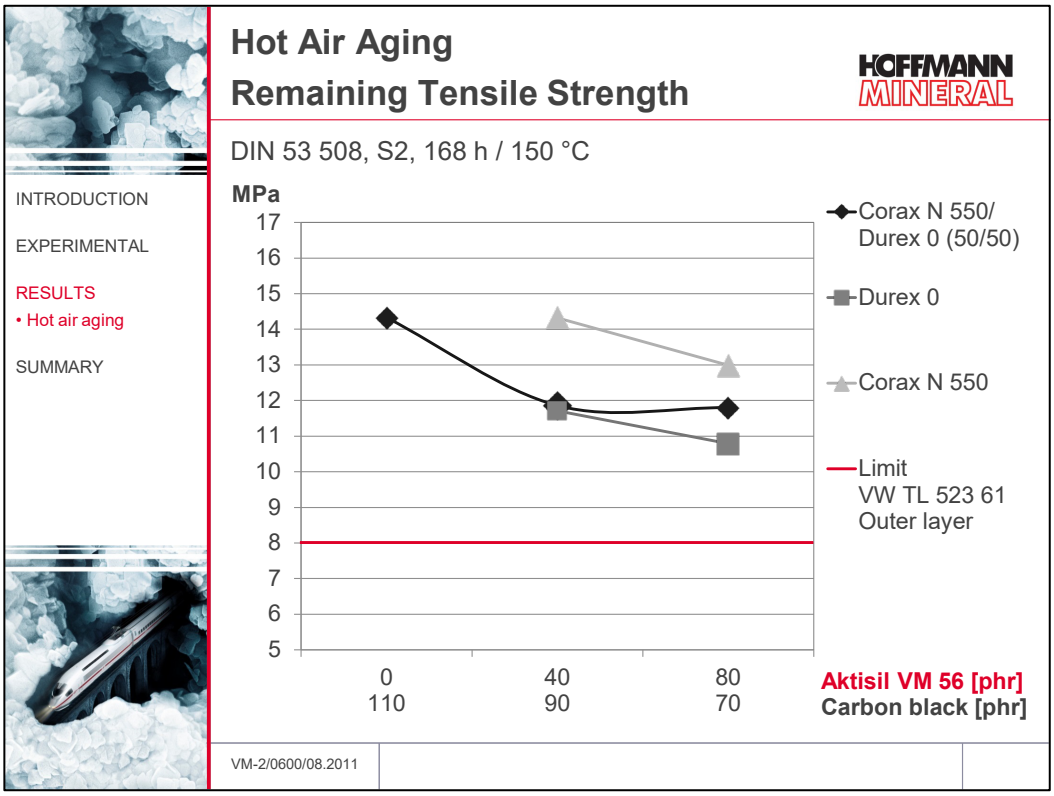

Fig. 20

As already established for the original elongation at break and after hot air aging at 160 °C, the elongation falls off with Aktisil VM 56 also during aging at 150 °C. However, the blend of 90 phr Durex 0 and 40 phr Aktisil VM 56 is able to meet the specification limit *(Fig. 21).*

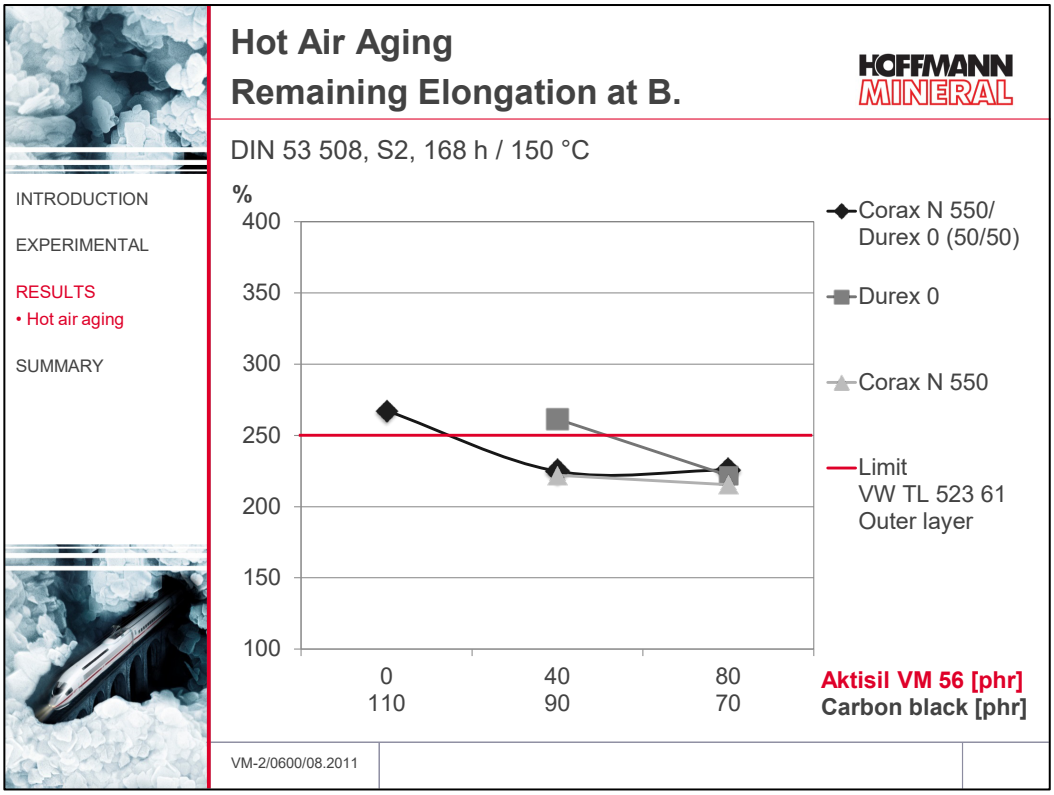

Fig. 21

3.3.3 336 h / 150 °C

The hardness results after hot air aging at 150 °C fluctuate around the limit of 76 Shore A as required in the specification, and the carbon black blend resp. Durex 0 with Aktisil VM 56 come out within the acceptable region. With a modification of the formulation, as already discussed for the 100 % modulus *(Figure 14)*, a larger distance towards the limiting value could be attained *(Fig. 22)*.

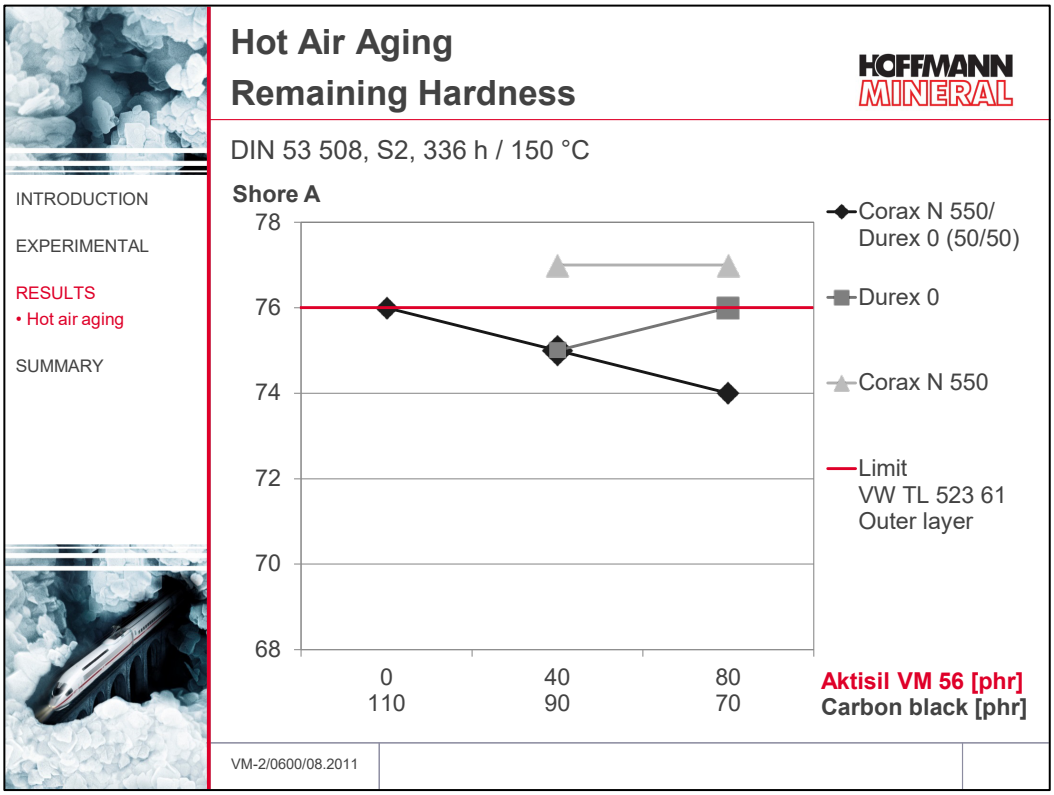

Fig. 22

With increasing Aktisil 56 content, there is a decreasing trend. The results, however, are clearly above the specified limit of 8 MPa *(Fig. 23)*.

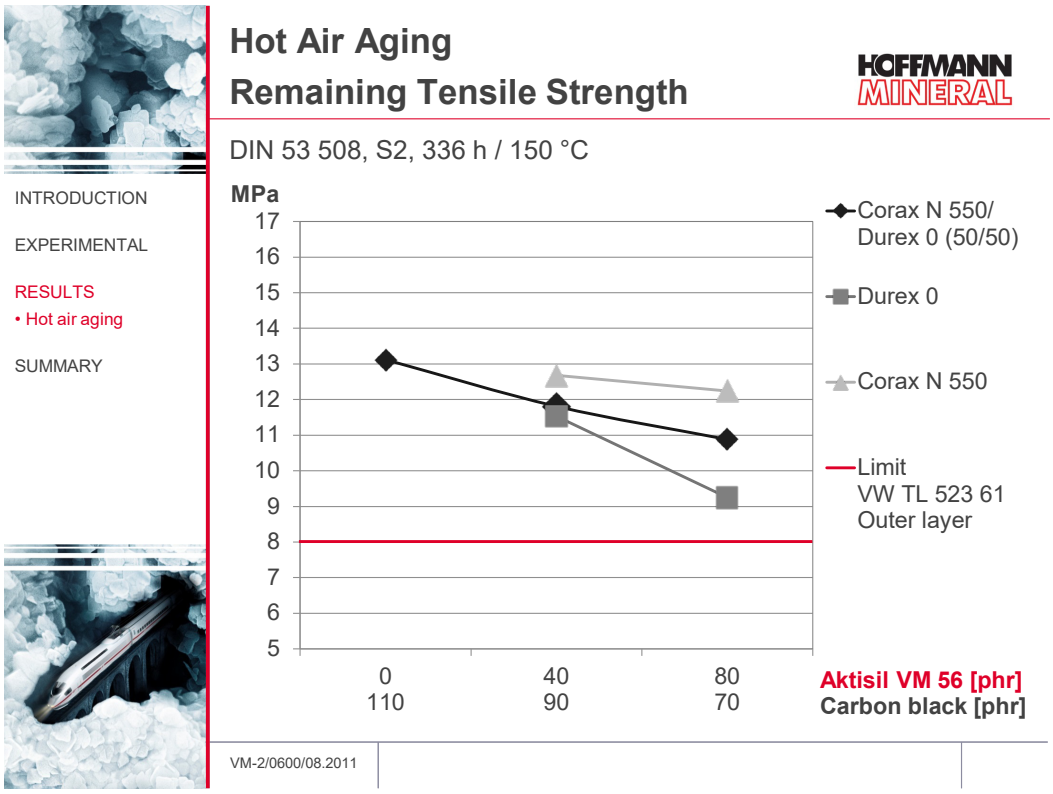

Fig. 23

As already found for the original elongation at break as well as after hot air aging at 160 °C, the elongation goes down also here with additions of Aktisil VM 56. However, the requirements of the standard can be met with a blend of 90 phr Durex 0 and 40 phr Aktisil VM 56 *(Fig. 24)*.

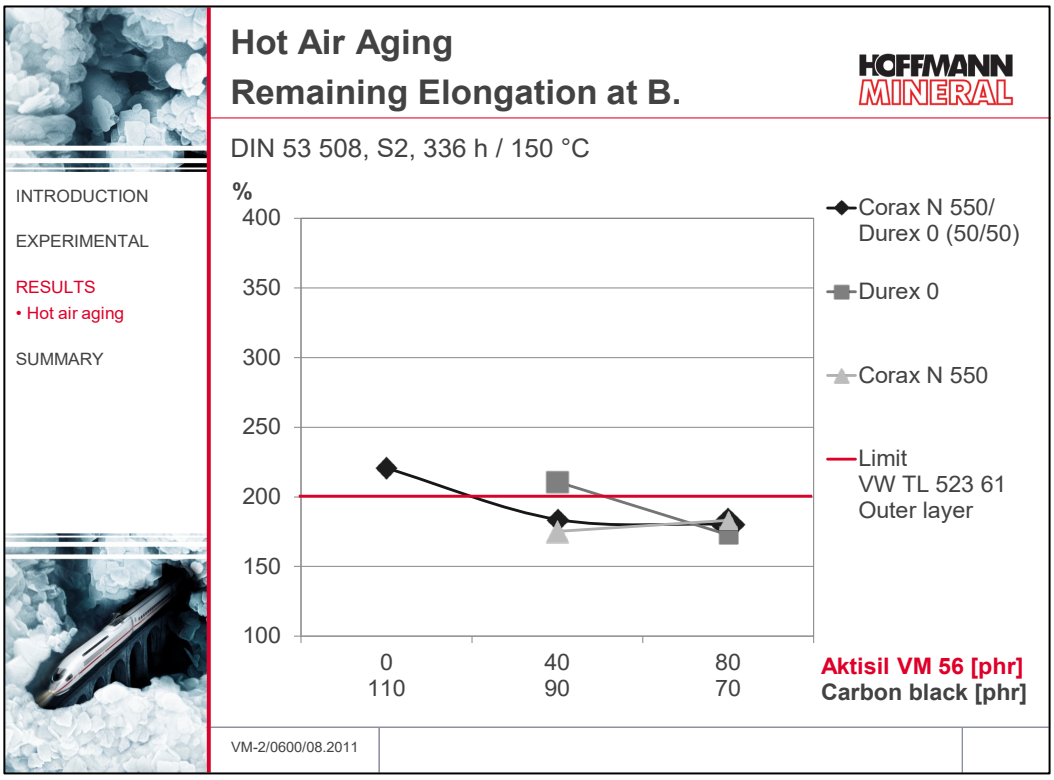

Fig. 24

3.3.4 Comparison 150 °C and 160 °C

In order to illustrate the principal effect of Aktisil VM 56 onto the elongation at break after hot air aging under the two conditions evaluated, here the relative change is plotted vs. the Aktisil VM 56 content. It can be concluded that no negative effects in the relative changes, and thus in the aging properties, can be attributed to Aktisil VM 56 *(Fig. 25)*.

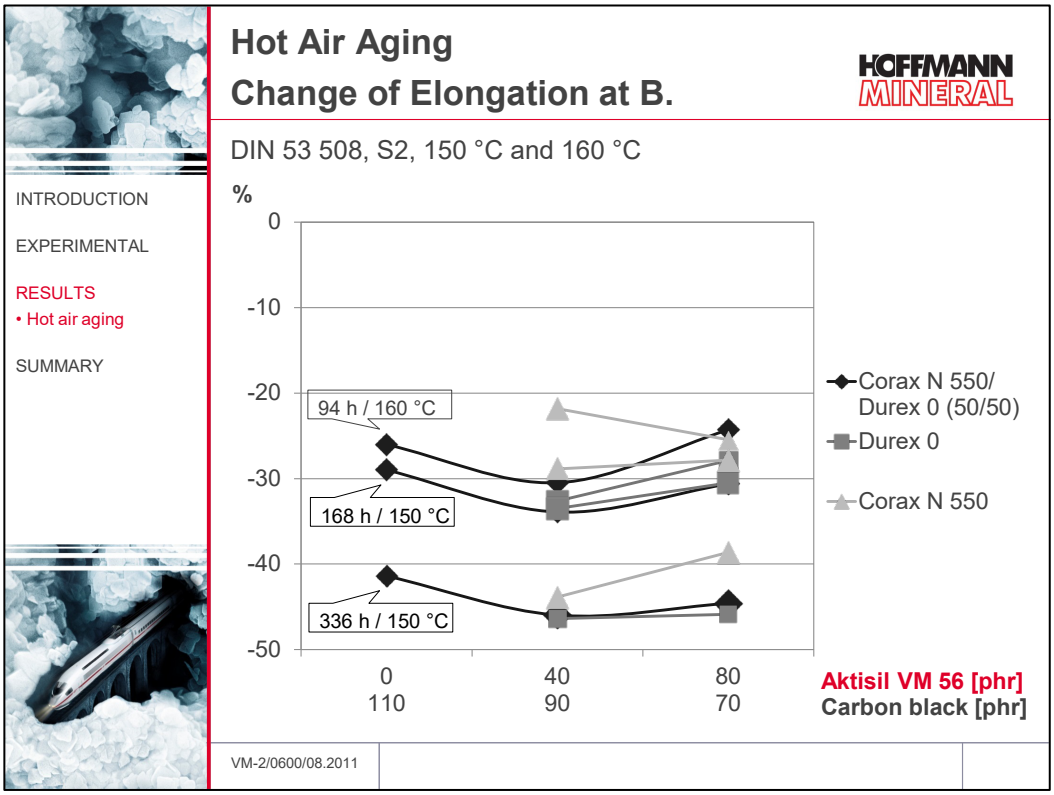

Fig. 25

3.4 Immersion in oil

No negative trend for the hardness after oil immersion can be observed with the addition of Aktisil VM 56. The differences observed have to be explained by the scatter of the test results. All results are found close to the limits of the standard *(Fig. 26).*

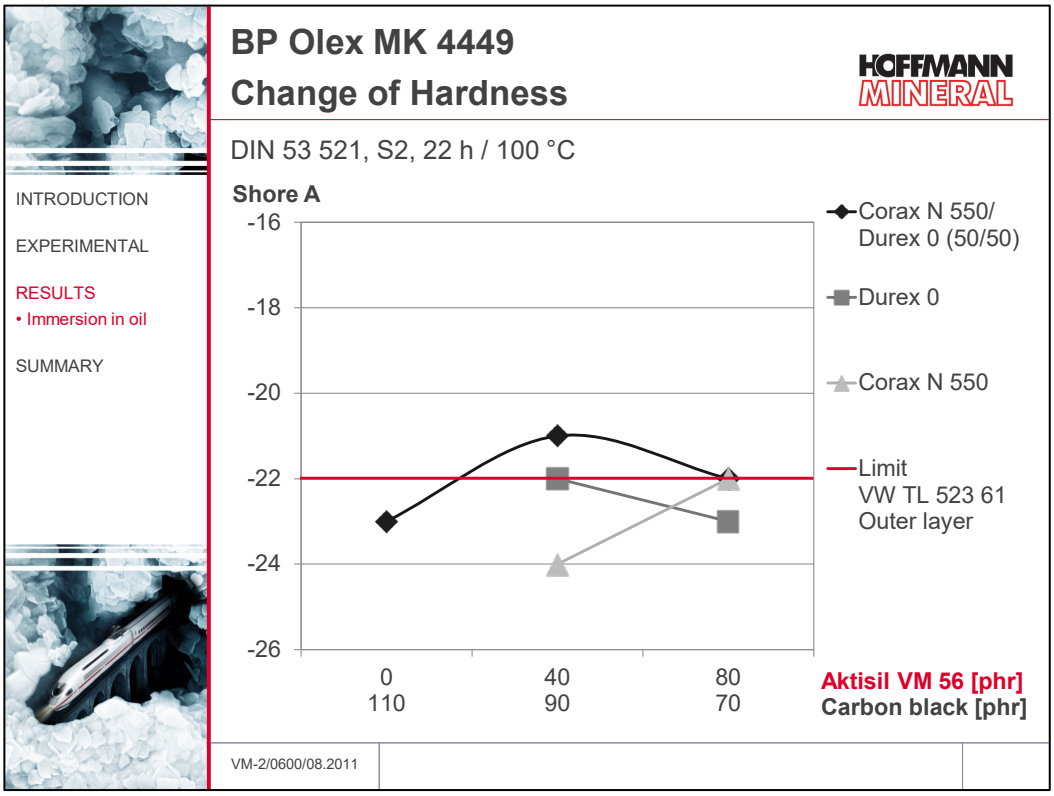

Fig. 26

Increasing Aktisil VM 56 additions give rise to a decrease of the tensile strength after oil immersion, while the results remain above the specified limit of 6.5 MPa *(Fig. 27).*

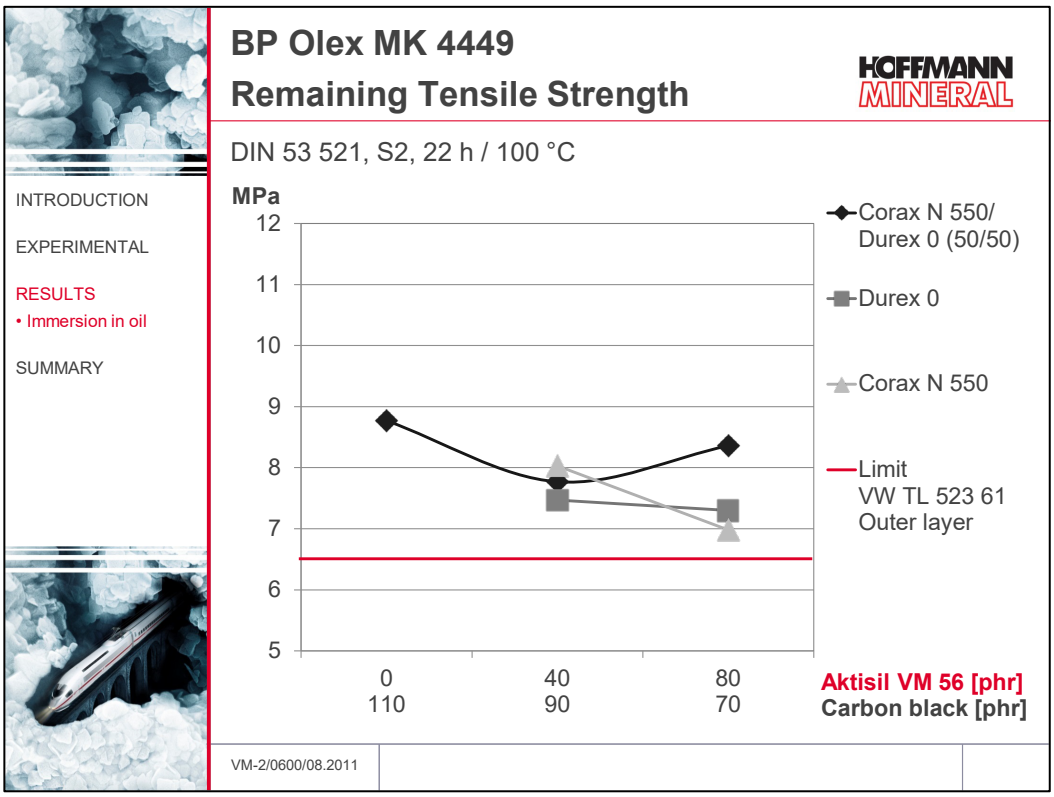

Fig. 27

As already established for the original elongation at break and after hot air aging, also after oil immersion Aktisil VM 56 causes the elongation to fall off. With a blend of Durex 0 and Aktisil VM 56, however, the specified limit can be met *(Fig. 28)*.

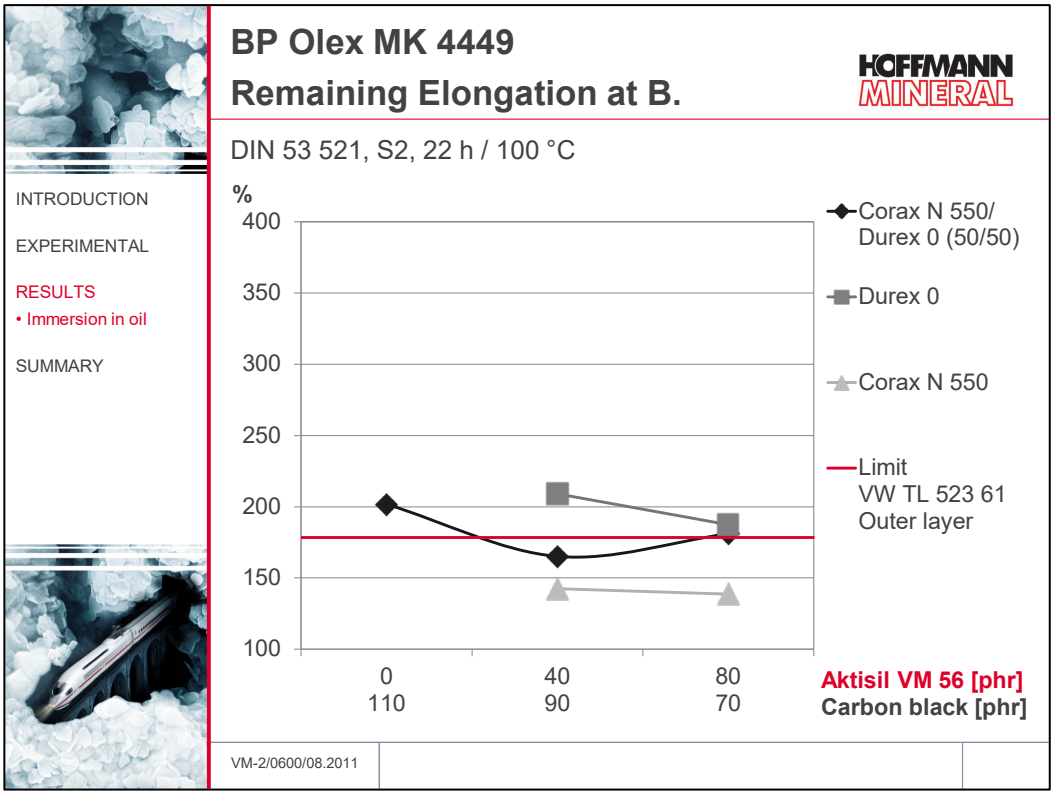

Fig. 28

The addition of Aktisil VM 56 is able to markedly reduce the weight increase during oil immersion. The effect shows a linear relation with the Aktisil VM 56 loading *(Fig. 29)*.

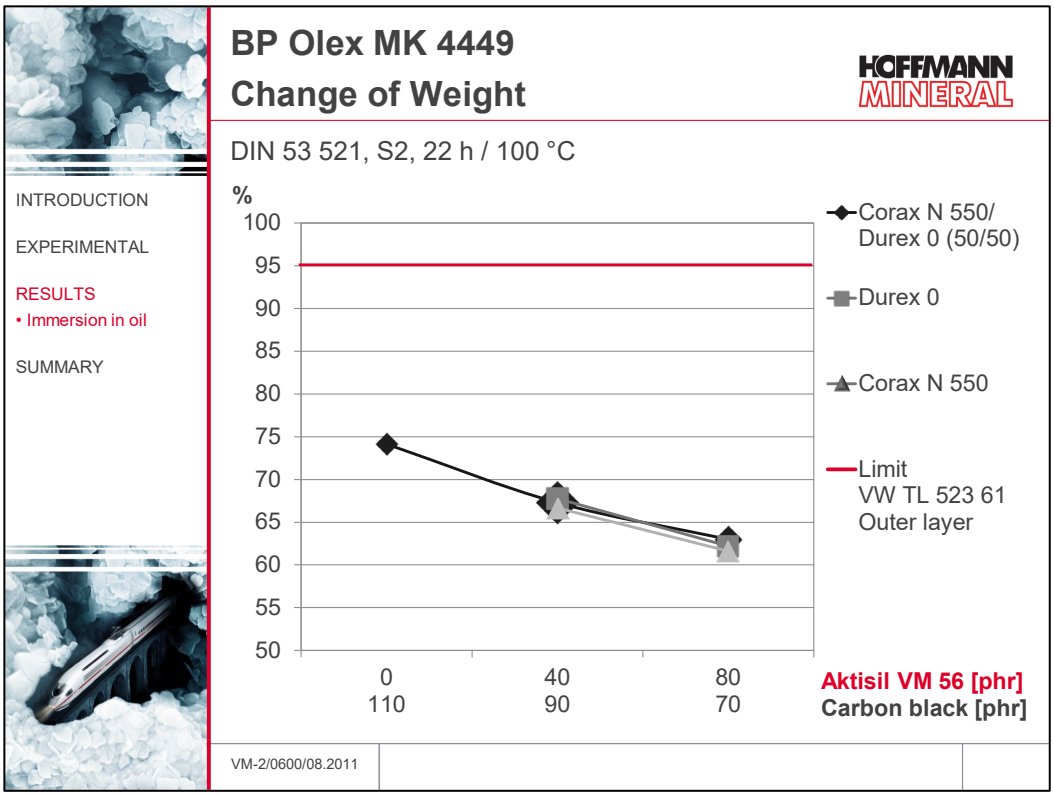

Fig. 29

3.5 Immersion in cooling fluid

With increasing addition of Aktisil VM 56, there is a trend towards a slight decrease of the hardness change during immersion in cooling fluid, while the results remain clearly better than the specified limit of minus 5 Shore A *(Fig. 30).*

For the cooling liquid immersion, the following composition was selected:
50 vol.-% cooling liquid according to VW TL 774 D (VW G12, red)
50 vol.-% distilled water

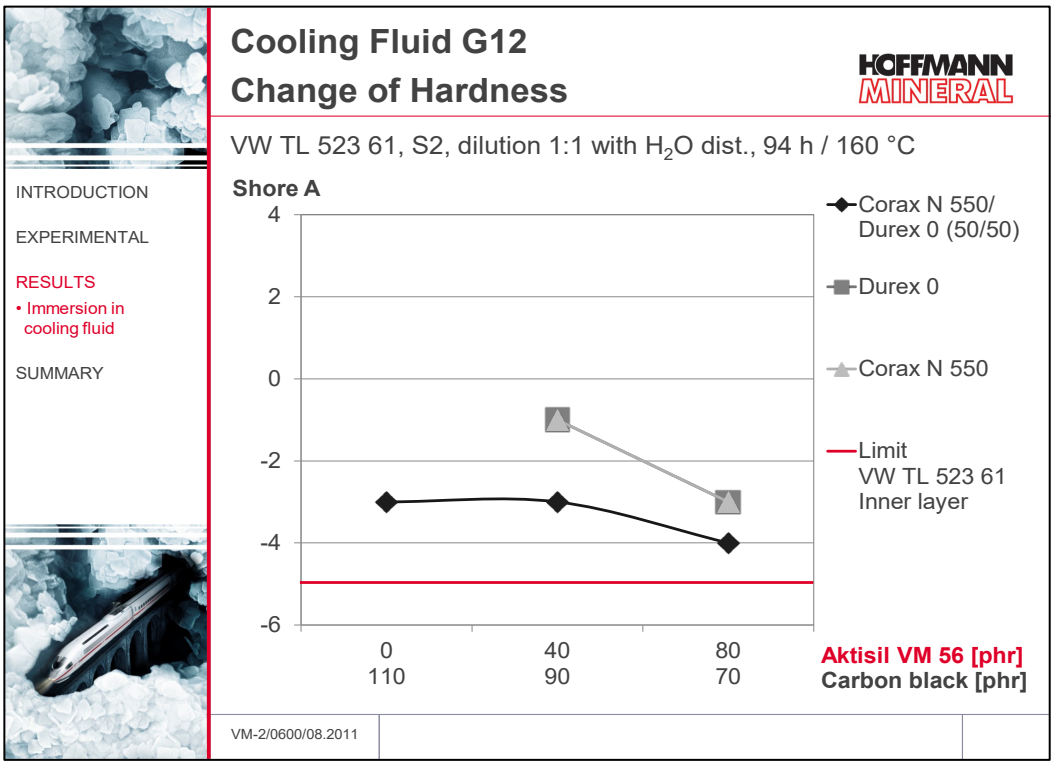

Fig. 30

With increasing Aktisil VM 56 addition, there is a trend towards a reduction of the tensile strength after immersion in cooling fluid, while the results remain clearly above the specified limit of 9 MPa *(Fig. 31).*

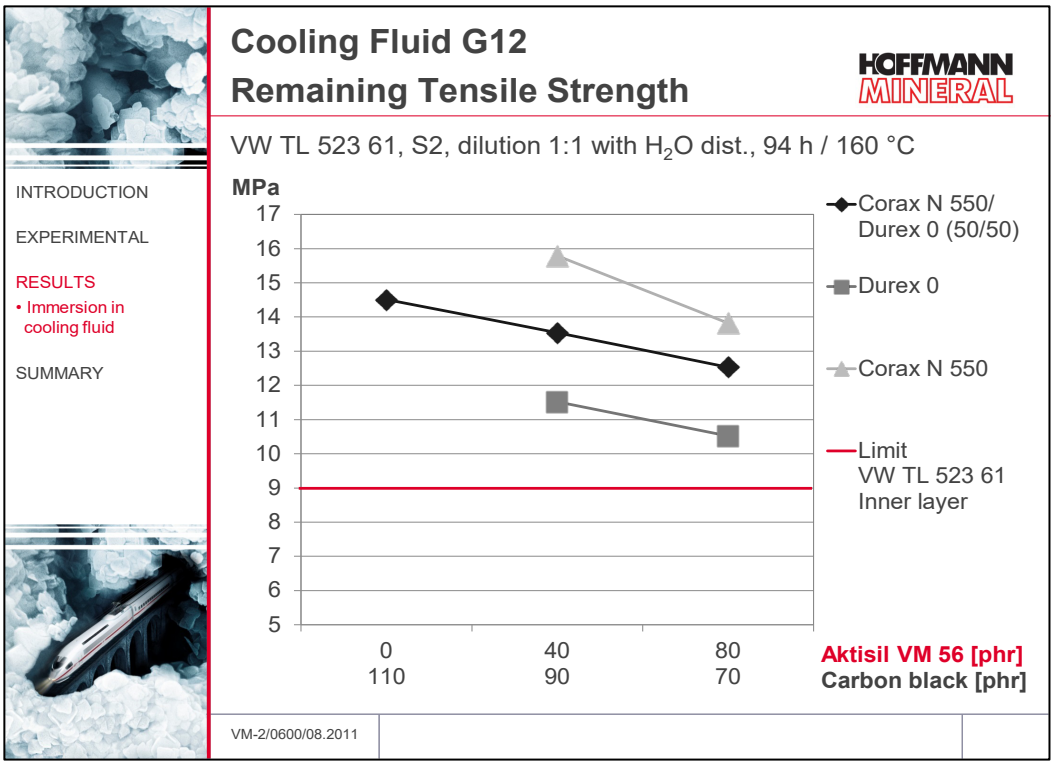

Fig. 31

With increasing Aktisil VM 56 addition, there is a trend towards a reduction of the elongation at break after immersion in cooling fluid, while the results remain clearly above the specified limit of 250 % *(Fig. 32)*.

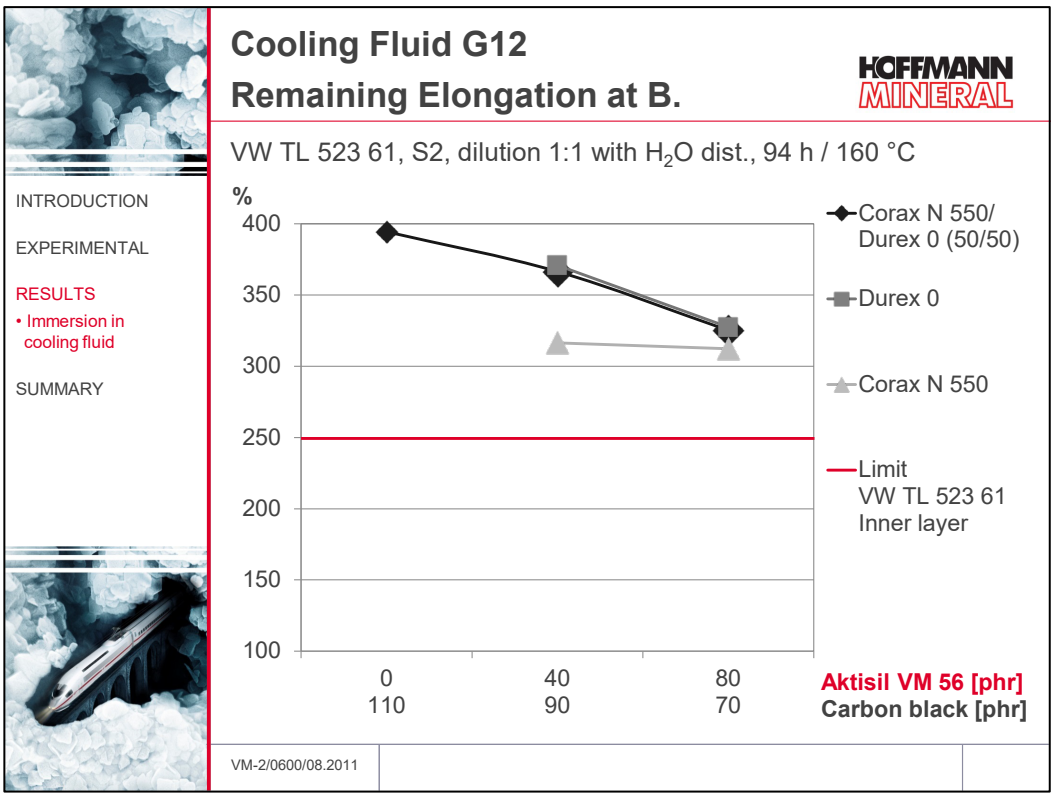

Fig. 32

Via the addition of Aktisil VM 56, the weight increase during immersion in cooling fluid can be markedly reduced. The effect linearly correlates with the Aktisil VM 56 loading *(Fig. 33)*.

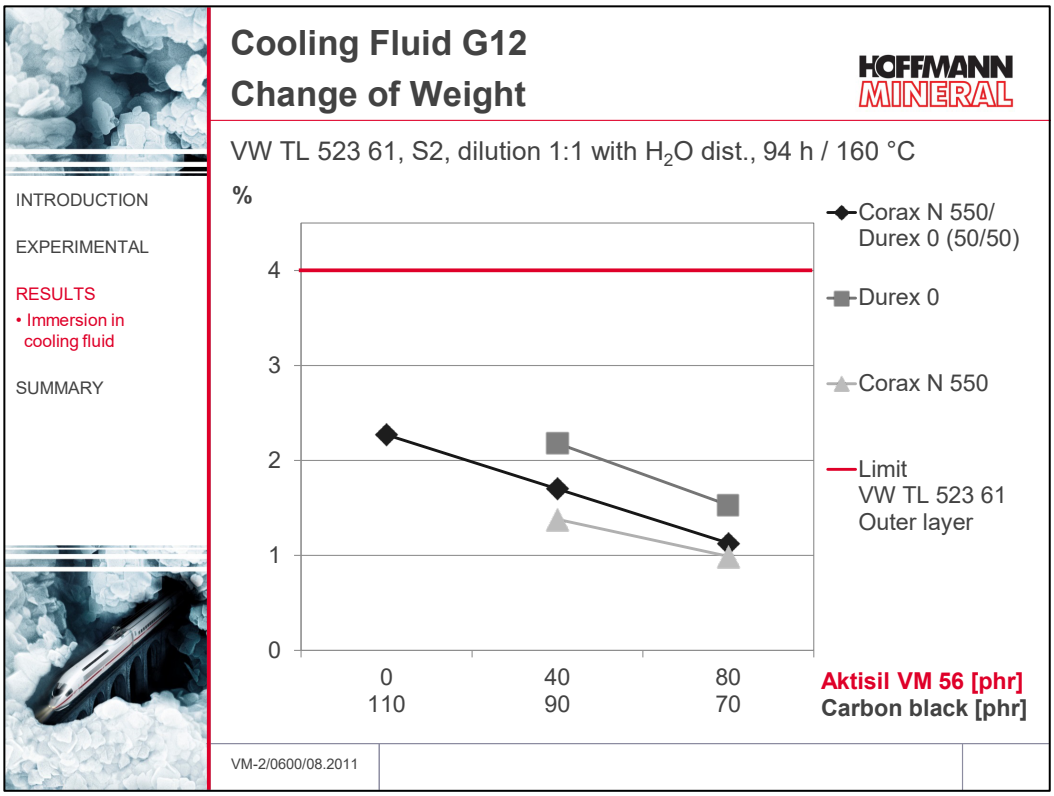

Fig. 33

3.6 Electrical properties

At a high loading, Aktisil VM 56 leads to a markedly increased electrical resistivity. Without Aktisil VM 56 as well as with 40 phr, the results, however, came off outside of the measuring range of the high-ohm measuring equipment *(Fig. 34).*

In *Figure 35*, by contrast a modified measuring arrangement and a different instrument have allowed to evaluate the whole field.

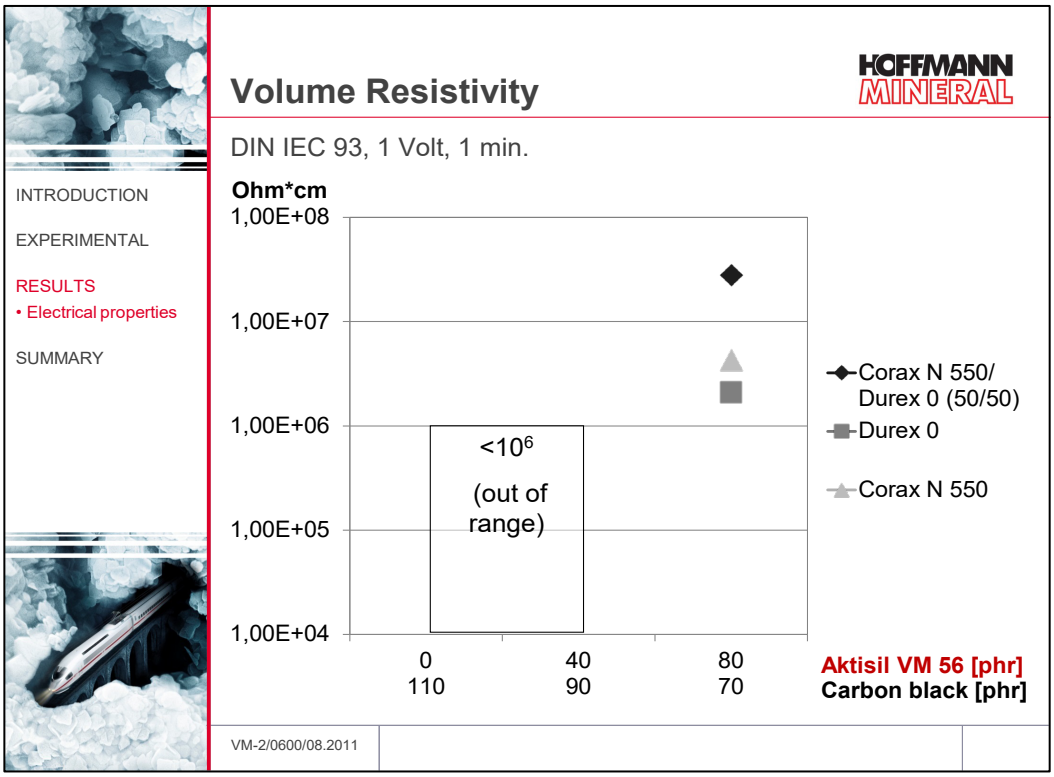

Fig. 34

The addition of Aktisil VM 56 is able to markedly increase the electrical resistivity. The results obtained come close to the insulating region, which means negative effects through electrochemical corrosion should be largely minimized *(Fig. 35).*

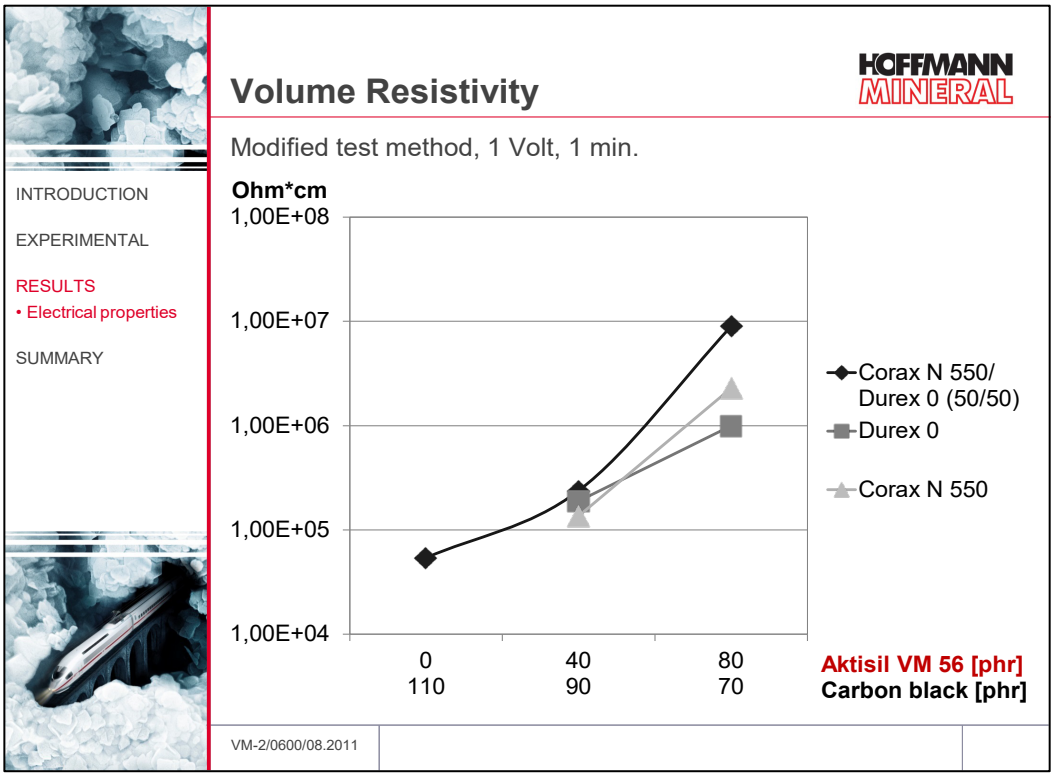

Fig. 35

3.7 Extrusion properties

The addition of Aktisil VM 56 gives rise to a marked increase of the length output during extrusion. The rpm of the extruder was kept at 100. This means Aktisil VM 56 offers a possibility to increase the production rate and consequently the productivity *(Fig. 36).*

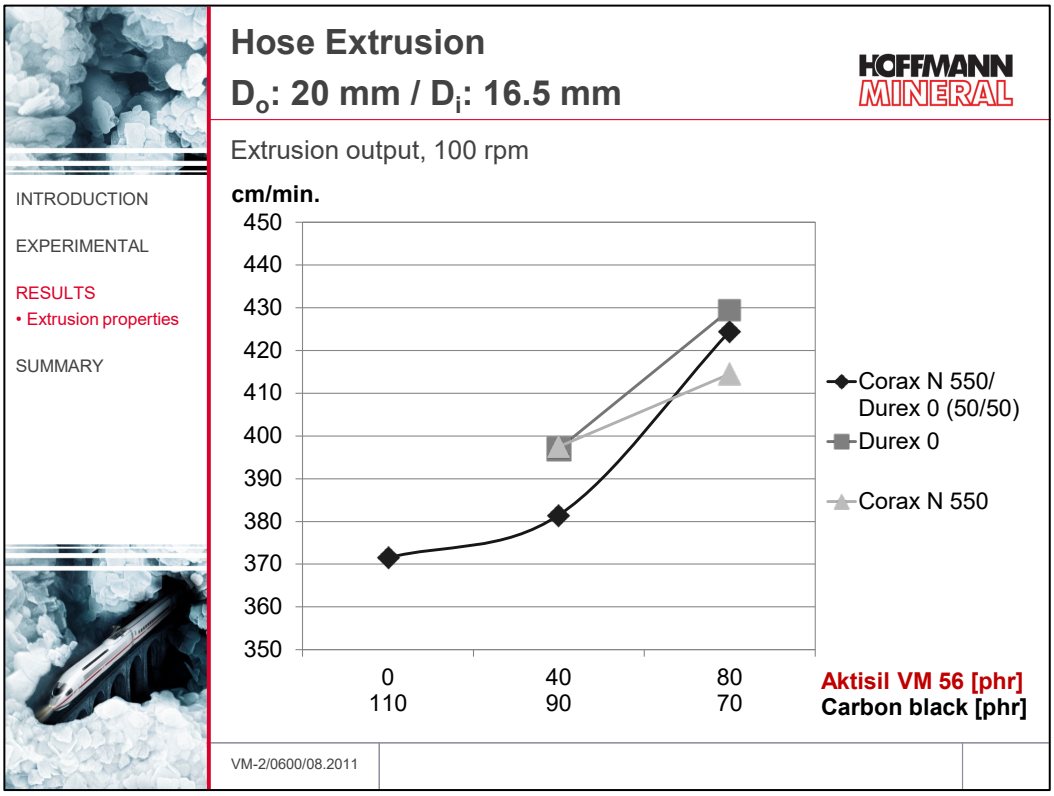

Fig. 36

4 Summary

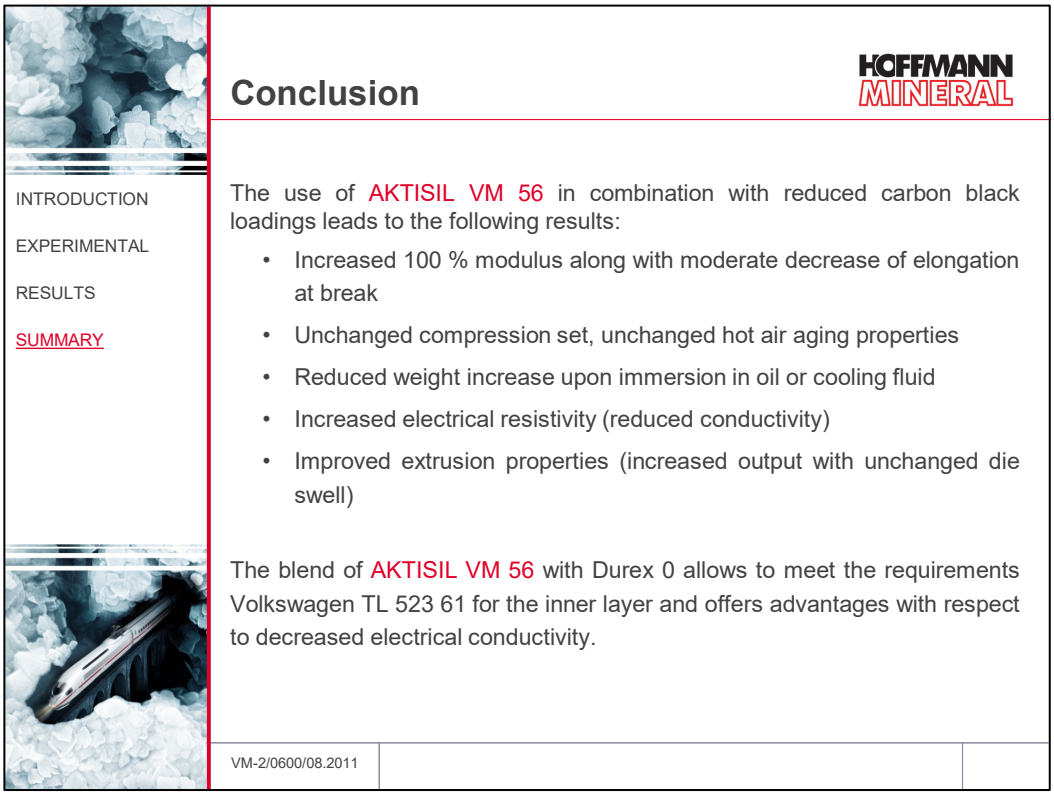

Fig. 37

Electrical conductivity must be reduced in order to avoid electrochemical corrosion. Aktisil VM 56 has proven itself very good in this respect.

It fully meets the requirement profile for the inner layer.

The 40:90 blend of Aktisil VM 56 and lamp black Durex 0 – in laboratory tests always within the stipulated limits of the VW guideline for hardness and elongation at break after ageing and immersion in oil – lends itself superbly to optimizing mixtures for outer layer in order to meet requirements with greater safety margin.