Powder coating:

Polyester/primid for

outdoor coatings (RAL 7016)

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VM-04/1217/03.2019

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1 Introduction

The use of Neuburg Siliceous Earth had already demonstrated positive effects in earlier studies of epoxy- and hybrid-based powder coatings. The optical and mechanical properties were maintained or even improved.

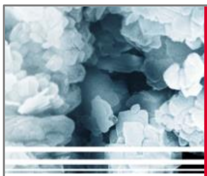
A large number of technical trials and a special series of tests in the powder coating domain (Polyester/TGIC) verify the excellent corrosion protection by using Neuburg Siliceous Earth due to its unique structure.

Thus the question comes up if Neuburg Siliceous Earth is capable of replacing the common used fillers in the field of powder coatings in the area of facades while maintaining or even further improving the optical and mechanical properties, particularly the resistances required by the International Quality Regulations for the Coating of Building Components (GSB).

This question was examined on the basis of an anthracite gray (RAL 7016) standard polyester/Primid formulation.

2 Experimental

2.1 Base formulation



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Base Formulation

	Description	[%]
Crylcoat 2618-3	Polyester resin	75.0
Primid XL 552	Hydroxyalkylamide, crosslinker	3.6
Ceraflour 991	Polyethylene wax, micronized	0.3
Pigments for RAL 7016	Titanium dioxide, pigments red, blue, yellow, black	6.94
BYK 3900P	Anti-crater and leveling additive	1.0
Benzoin	Leveling agent	0.4
Filler		12.76
Total		100

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The study was performed using the base formulation illustrated in *Fig. 1*. Crylcoat 2618-3 is a carboxylated polyester resin (base resin for GSB standard requirements) for use with Primid XL 552, a beta-hydroxyalkylamide crosslinker. Ceraflour 991 is a micronized polyethylene wax that increases surface smoothness and improves surface protection, while BYK 3900P is a leveling and anti-crater additive, and Benzoin improves leveling. The pigment blend used to create the anthracite gray shade, RAL 7016, consisted of titanium dioxide and color pigments. The filler ratio was 12.76 %.
Fig. 1

2.2 Formulation variations

The reference filler for high gloss applications (60° gloss, ~ 90 GU) was a precipitated barium sulfate, the Blanc fixe. For this purpose different grades of Neuburg Siliceous Earth were chosen: the calcined product Aktifit PF 115 (particularly recommended for high gloss) and Silfit Z 91, a calcined product with outstanding mechanical properties.
The reference filler for medium gloss, which tends to be the standard for facades (60° gloss, ~ 75 GU) was an aluminum hydroxide. Concerning this, the selected Neuburg Siliceous Earth grade was Sillitin V 88 (standard product for matting).

2.3 Fillers and characteristics

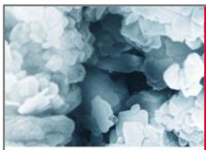
Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural aging, the silica portion exhibits a round grain shape and consists of aggregated primary particles of about 200 nm diameter.

The calcination of the Neuburg Siliceous Earth helps to drive off the crystal water present in the kaolinite portion and to generate calcined kaolinite. The silica portion remains inert under the temperature chosen. The resulting products have an outstandingly high degree of whiteness and color neutrality.

The filler characteristics are shown in *Fig. 2* and *3*.

Blanc fixe, the reference filler for high gloss, is characterized by the highest brightness and color neutrality, followed by the two calcined types of Neuburg Siliceous Earth, Aktifit PF 115 with a special amino silane surface treatment, and Silfit Z 91.

The aluminum hydroxide used as reference filler for the medium-gloss had also highest brightness and the lowest yellowish tint, followed by Sillitin V 88.



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Filler Characteristics

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Filler	Description	Surface Treatment	Color L*	Color b*
Blanc fixe	Barium Sulfate precipitated	none	96.5	0.3
Aktifit PF 115	Calcined Neuburg Siliceous Earth	special amino silane	94.5	0.5
Silfit Z 91	Calcined Neuburg Siliceous Earth	none	95.8	0.6
ATH	Synthetic Aluminum hydroxide	none	95.0	2.2
Sillitin V 88	Neuburg Siliceous Earth	none	93.7	3.0

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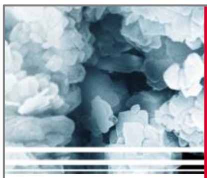
Fig. 2

The density of the Neuburg Siliceous Earth grades was 2.6 g/cm³, the oil absorption was 45 – 55 g linseed oil per 100 g of filler and a BET surface area of 7 to 8 m²/g. The Blanc fixe had the highest density, of 4.4 g/cm³ and an oil absorption of 37 g/100 g and a BET surface area of 4.0 m²/g. The aluminum hydroxide had the lowest density, of 2.4 g/cm³, coupled with the lowest oil absorption value of 26 g/100 g and the lowest BET surface area of 2.5 m²/g.

The division into two gloss categories is clearly visible in the particle size distribution: the first three finer fillers are suitable for high-gloss applications as the average particle size diameter d_{50} is less or equal to 2 µm and the topcut d_{97} is less than 10 µm.

The higher particle size of the fourth and fifth much coarser fillers makes them suitable for medium-gloss applications, the d_{50} ranges from 5 to 13 µm, and the d_{97} is between 16 and 37 µm.



Filler Characteristics

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Filler	Density [g/cm³]	Oil absorption [g/100g]	BET [m²/g]	d_{50}	d_{97}
Blanc fixe	4.4	37	4.0	1.1	4.3
Aktifit PF 115	2.6	~ 55	8.0	2.0	9.3
Silfit Z 91	2.6	55	8.0	2.0	9.0
ATH	2.4	26	2.5	12.7	37
Sillitin V 88	2.6	45	8.3	4.7	16

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Fig. 3

2.4 Preparation

The premix was first blended for two minutes at 1000 rpm in a Prism Pilot 3 mixer before being homogenized in an extruder (Coperion ZSK 18, twin screw, screw speed 800 rpm, heating zones 50°C / 80°C / 120°C / 120°C / 120°C). The formulations were ground in a Retsch AS 200 mill and then passed through a 125 µm sieve. The powder was applied using an automatically powder gun (Corona) at 80 kV and 0.9 bar onto Q-Panel sheets (aluminum chromated Al 48 and steel R 48).

The polyester resin takes 10 minutes to cure at a peak metal temperature (PMT) of 180 °C. Despite using the different fillers, neither the curing time nor the curing temperature had to be increased. Accordingly, all variations of the formulation were sufficiently crosslinked within 10 minutes at a PMT of 180 °C.

The dry film thickness was 70-80 µm.

2.5 Test methods

Color values

The color values CIE L* a* b* were measured using a spectral photometer with d/8° measuring geometry and light type D 65.

Gloss

A micro-TRI-gloss from the company BYK was used to measure gloss. The measuring angle of 20° represents the high gloss range, the angle of 60° the medium gloss range.

Haze

High quality surface finishes are expected to have a clear and bright appearance. Microstructures, which can be caused by insufficient dispersion, for example, create a milky appearance or haze. This effect was measured using the micro-haze plus from the company BYK.

Adhesion/Flexibility (mandrel bending test)

The mandrel bending test according to DIN EN ISO 6860 is used to evaluate the flexibility and adhesion of coatings when exposed to bending stress using a conical thorn. The test result is either stated as the crack length, starting from the thin end of the thorn, or as the thorn diameter, measured at the end of the crack. The lower the result, the better is the flexibility/adhesion of the coating.

Adhesion (cross cut test)

The cross cut test according to DIN EN ISO 2409 was performed using a 2 mm grid distance and evaluated as per the standard.

Flexibility (cupping test)

The cupping test as per DIN ISO 1520 involves slowly pressing a semi-sphere from the rear into the panel at constant speed and evaluating the front coating in respect of crack formation. The test result is the maximum possible cupping as length in millimeters just before cracks become visible.

Flexibility (impact/reverse impact test)

The impact/reverse impact test based on ASTM D 2794 involves dropping weights of 2 lbs (with different spherical diameters of 12.7 mm and 15.9 mm) from different heights onto the coated front side (impact test) and onto the uncoated back side (reverse impact test). The coating on the front side is then examined in each case for cracks.
The result of the reverse impact test is stated in inch pounds just before cracks become visible. The impact test was performed only with 80 inch pounds and the coating examined for cracks (yes or no).
The time span between curing the coating and performing the test was 1 hour or 3 days, as indicated in the results.

Sodium hydroxide test (2N)

As specified by GSB, 1 mL NaOH solution (2N) was dropped onto the coating and covered with a watch glass. After 60 minutes, the watch glass was removed and the excess solution had to be absorbed by using a soft fleece. The surface was rinsed by tap water and dried with a towel. The color was then evaluated: GSB requires level 1, i.e., no visible change in the color of the tested surface compared to the original (not tested) surface.

Water spot resistance

According to GSB regulations, coated aluminum parts in sealed plastic foil packaging must be tested for storage capability when exposed to humidity and temperature. To perform this test, five round filters (diameter 55 mm) were stacked, soaked with 1.5 ml of demineralized water and covered with a watch glass. The watch glass was attached to the specimen with insulating tape to ensure that no moisture can escape. This test specimen was placed in the drying chamber for 4 hours at 58°C. It was then left to cool down for 15 minutes before removing the watch glass and filter papers and then conditioning the specimen for 20 hours at 23°C and 50% relative humidity. The Delta L* change in color was then measured. According to GSB (Standard and Master), it must not exceed 4.

3. Results

3.1 Color

Because of the anthracite gray color, the use of filler does not affect the color values. Accordingly, all coatings, regardless of the used filler, demonstrated a L* value of about 34.5 (*Fig. 4*), an a* value of -1.4 (*Fig. 5*) and a b* value of about -2.9 (*Fig. 6*).

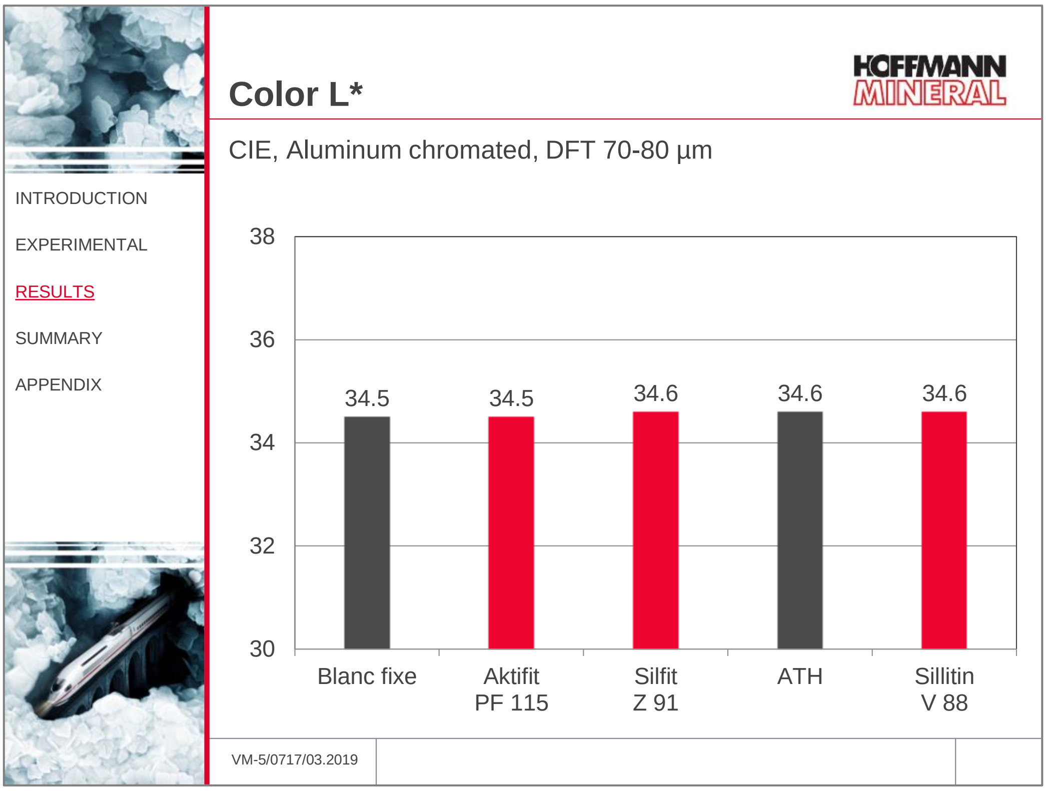
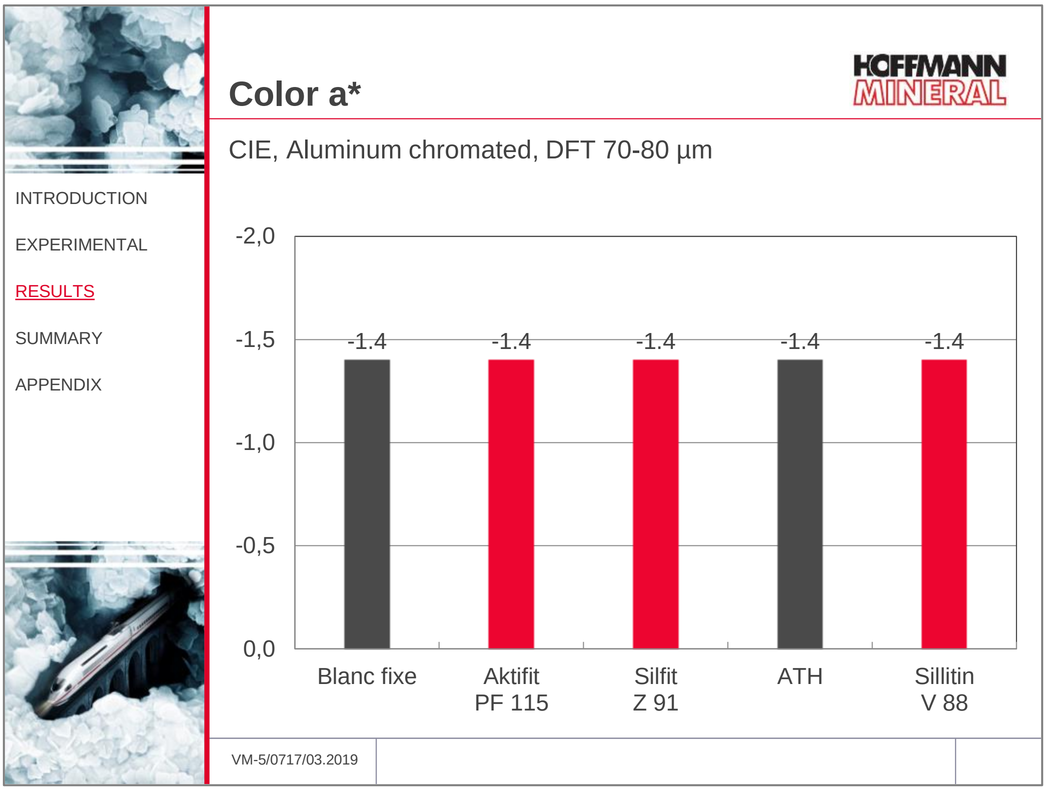


Fig. 4


HOFFMANN MINERAL
Color a*
CIE, Aluminum chromated, DFT 70-80 µm
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-2,0
-1,5
-1,0
-0,5
0,0
-1.4
-1.4
-1.4
-1.4
-1.4
Blanc fixe
Aktifit PF 115
Silfit Z 91
ATH
Sillitin V 88
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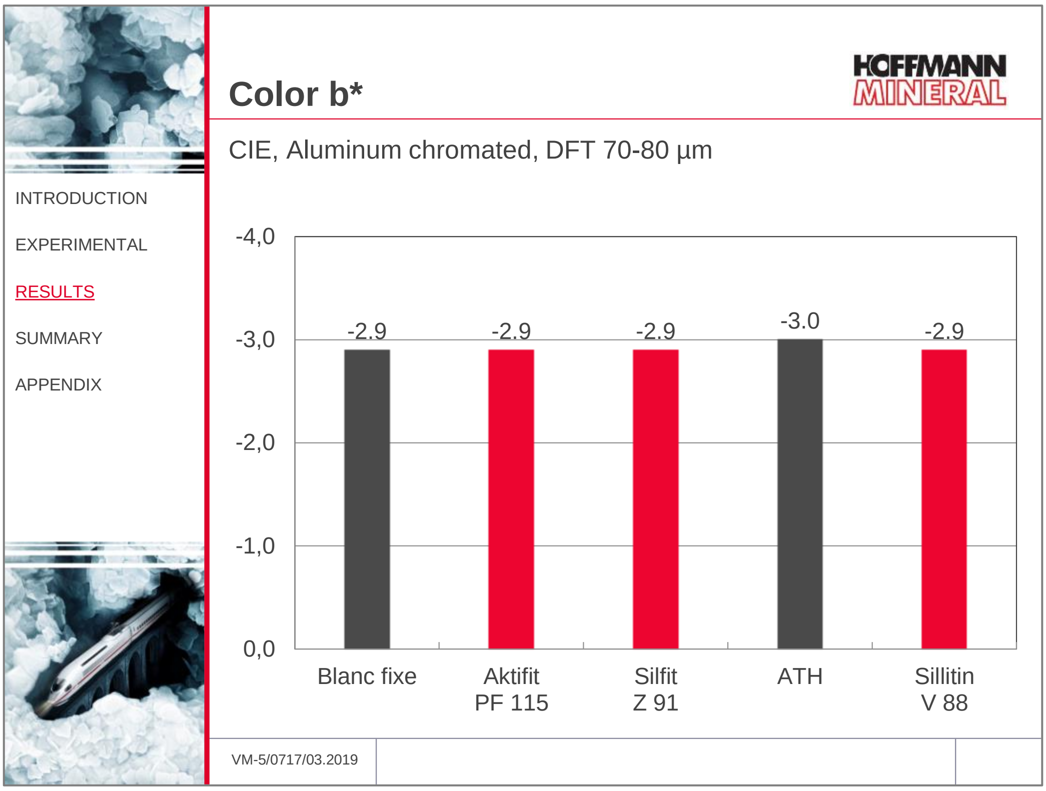
Fig. 5


HOFFMANN MINERAL
Color b*
CIE, Aluminum chromated, DFT 70-80 µm
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-3,0
-2,0
-1,0
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-2.9
-2.9
-2.9
-3.0
-2.9
Blanc fixe
Aktifit PF 115
Silfit Z 91
ATH
Sillitin V 88
VM-5/0717/03.2019

Fig. 6

3.2 Gloss, haze and surface appearance

Through the classification in high and medium gloss arose the following two groups: the first high-gloss group contained the Blanc fixe with the highest gloss of more than 90 GU at 60° gloss, followed by Aktifit PF 115 and Silfit Z 91. Looking at the large difference in density between barium sulfate and Neuburg Siliceous Earth, this only moderate difference in gloss appears even more remarkable. The pigment volume concentration (PVC) with Blanc fixe was 7% and with Neuburg Siliceous Earth 9.6%.
The values for the second, medium-gloss group containing the ATH and the Sillitin V 88 were also very close together (*Fig. 7*).

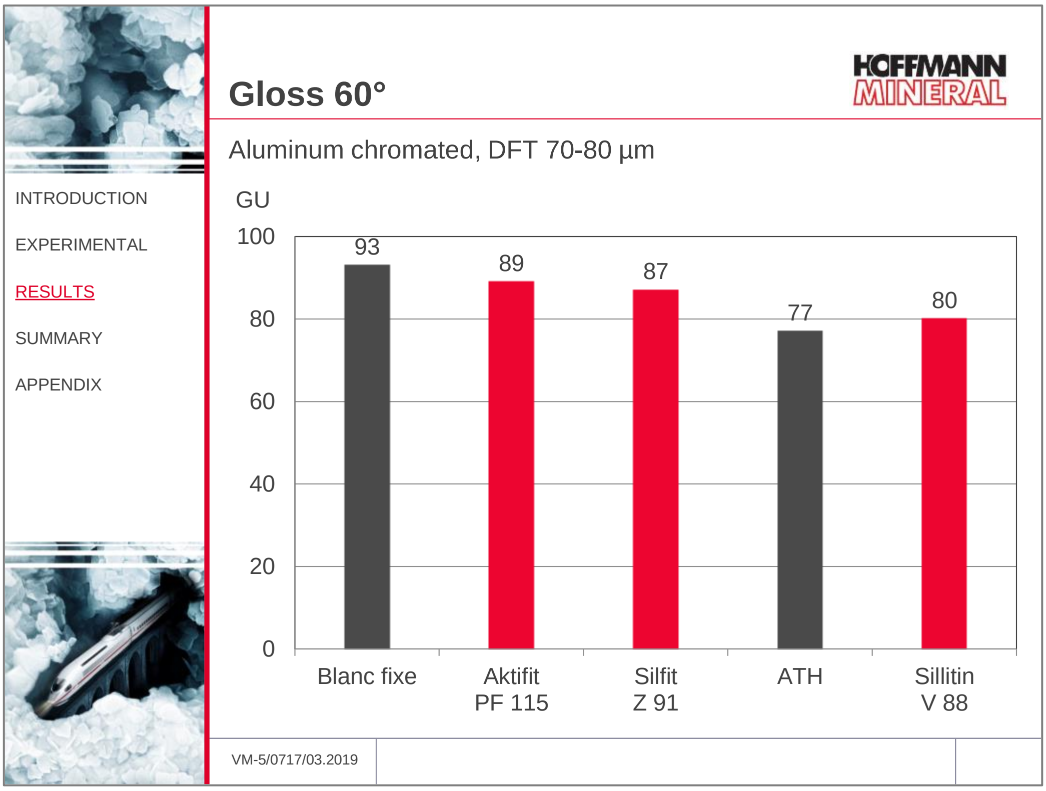


Fig.7

A look at the 20° gloss representing the high gloss shows that the precipitated barium sulfate demonstrated the highest gloss among the glossy coatings, as shown in *Fig. 8*, whereas facades are mainly coated with semi-gloss powder coatings, with only the 60° gloss being relevant.

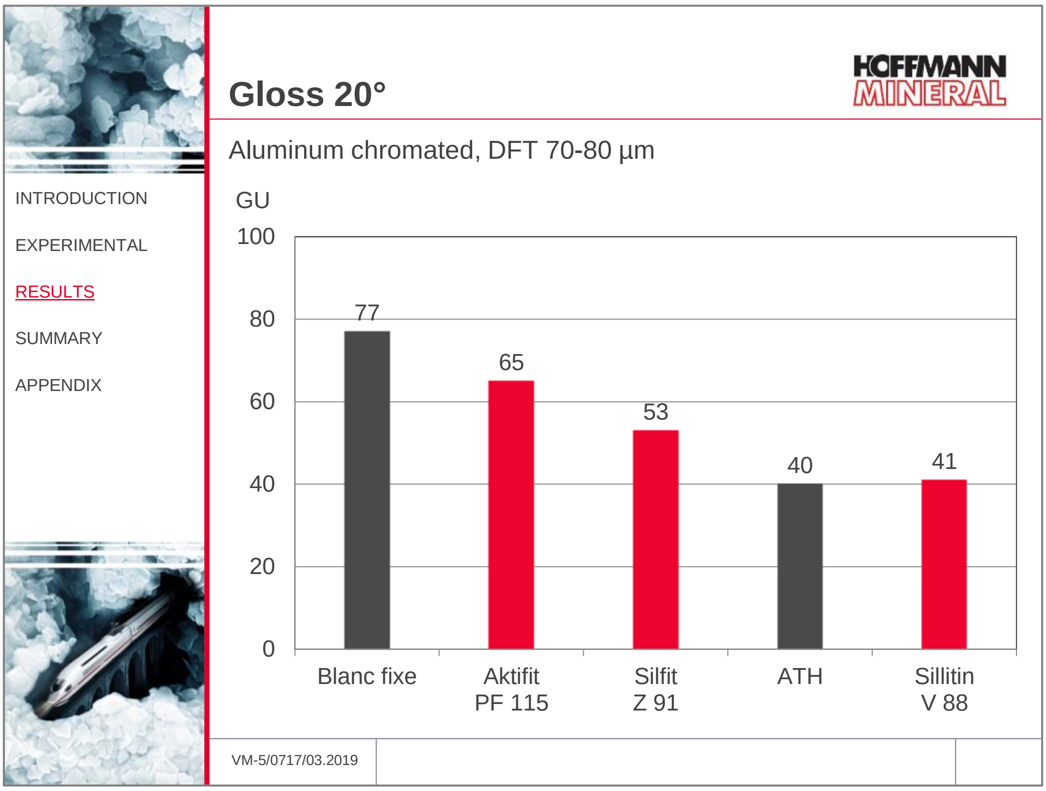


Fig. 8

The findings for the 20° gloss were also reflected in the haze of the high-gloss coatings, Blanc fixe had the lowest haze value, followed by Aktifit PF 115 (*Fig. 9*).

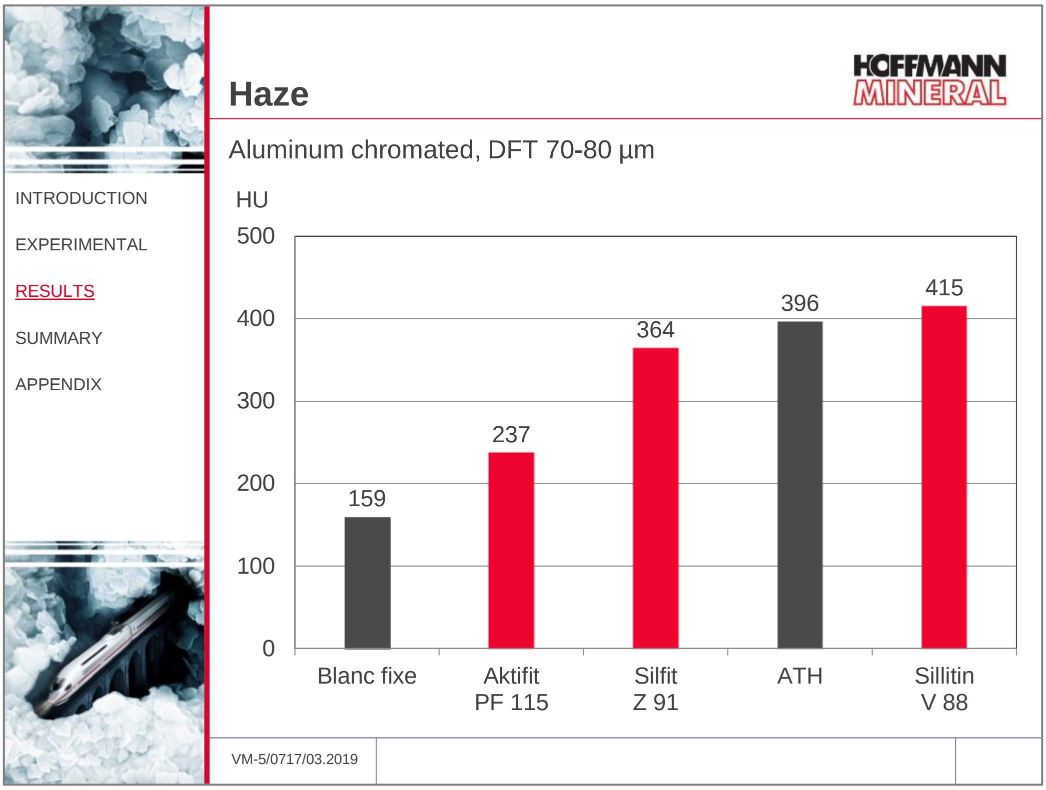


Fig. 9

These measurement results become relative, however, when studying the surface appearance under a microscope. The surface of the coating with Blanc fixe is inhomogeneous, with coarse filler particles/agglomerates clearly visible as elevations. These surface defects were caused ostensibly by insufficient dispersing time in the extruder. By contrast, the comparable types of Neuburg Siliceous Earth, such as Aktifit PF 115, demonstrate a surface that is perfectly smooth and sealed in appearance (*Fig. 10*).

The coating with ATH appears somewhat grainier than with the comparable Sillitin V 88, which may be attributable to the higher topcut d_{97} of the ATH. A comparison of the surface finish reveals a comparable gloss level but a much smoother finish with Sillitin V 88 than with ATH (*Fig. 11*).

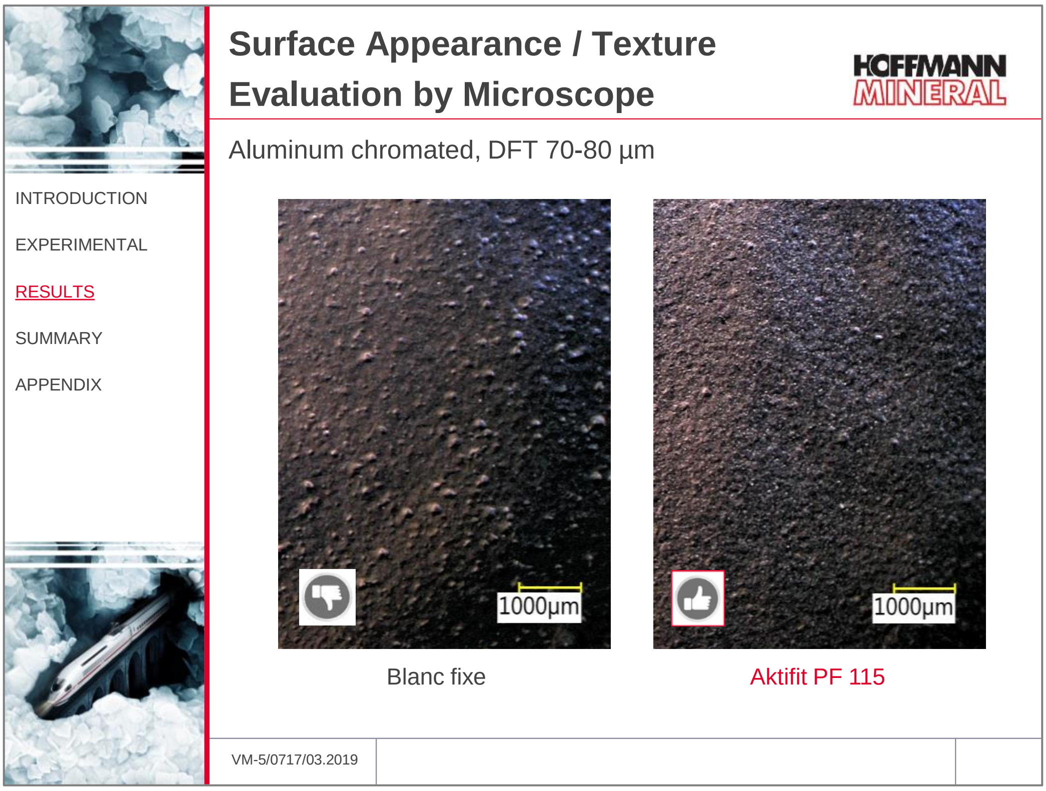


Fig. 10

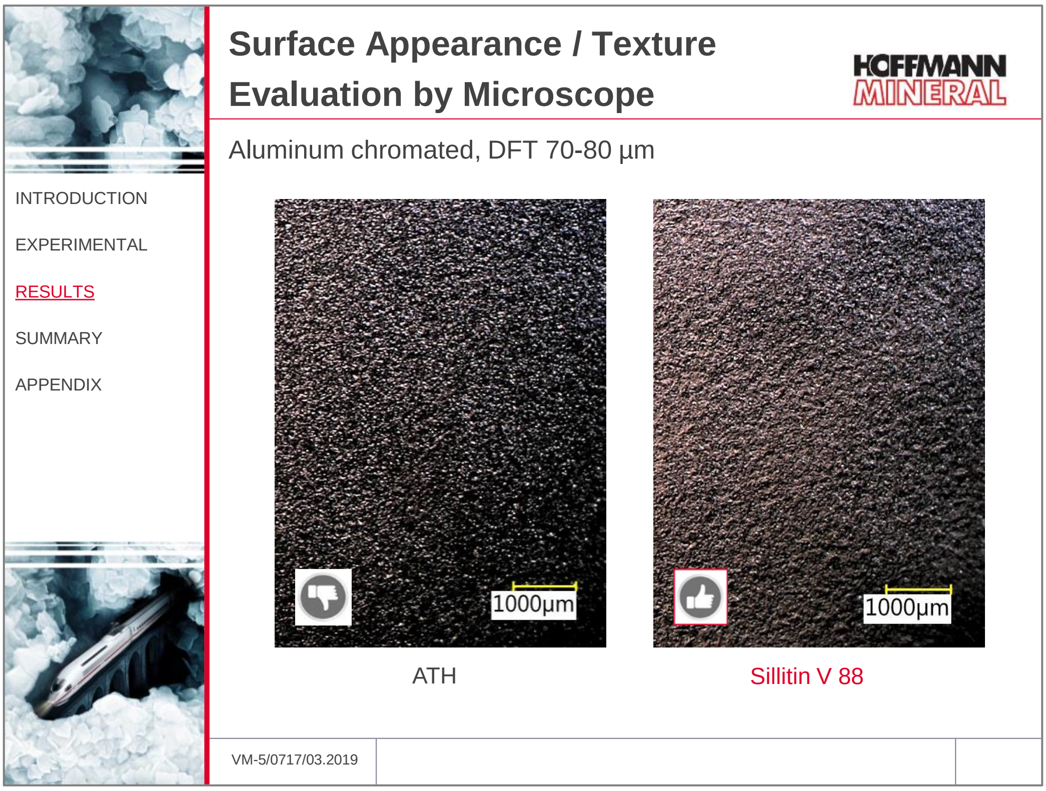
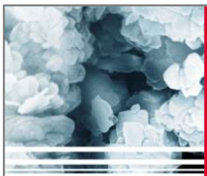


Fig. 11

3.3 Mandrel bending test, cross cut test, cupping test

Fig. 12 shows the mechanical tests on steel substrate, which produced no recognizable differences between the chosen fillers. Both flexibility and adhesion on the substrate were excellent in all tests. Nor were differences recognizable from the cupping test (slow deformation); the cupping values of all formulations were excellent at more than 10 mm.



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Mechanical Properties

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Steel (Q-Panel R 48), DFT 70-80 µm

	Mandrel Bending Test	Cross Cut Test (2 mm)	Cupping Test
Blanc fixe	all 0 mm	all GT 0	all > 10 mm
Aktifit PF 115			
Silfit Z 91			
ATH			
Sillitin V 88			

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Fig. 12

3.4 Impact test and reverse impact test

Testing the coatings for flexibility in respect of fast deformation, however, revealed marked differences, as shown on the following impact test and reverse impact test charts. *Fig. 13* shows the results of the reverse impact test 1 hour after curing and performed with a sphere of 15.9 mm diameter. The GSB requirement is a minimum of 20 inch pounds, which was satisfied by all formulations. However the results of more than 80 inch pounds for the two calcined types of Neuburg Siliceous Earth Aktifit PF 115 and Silfit Z 91 were outstanding.

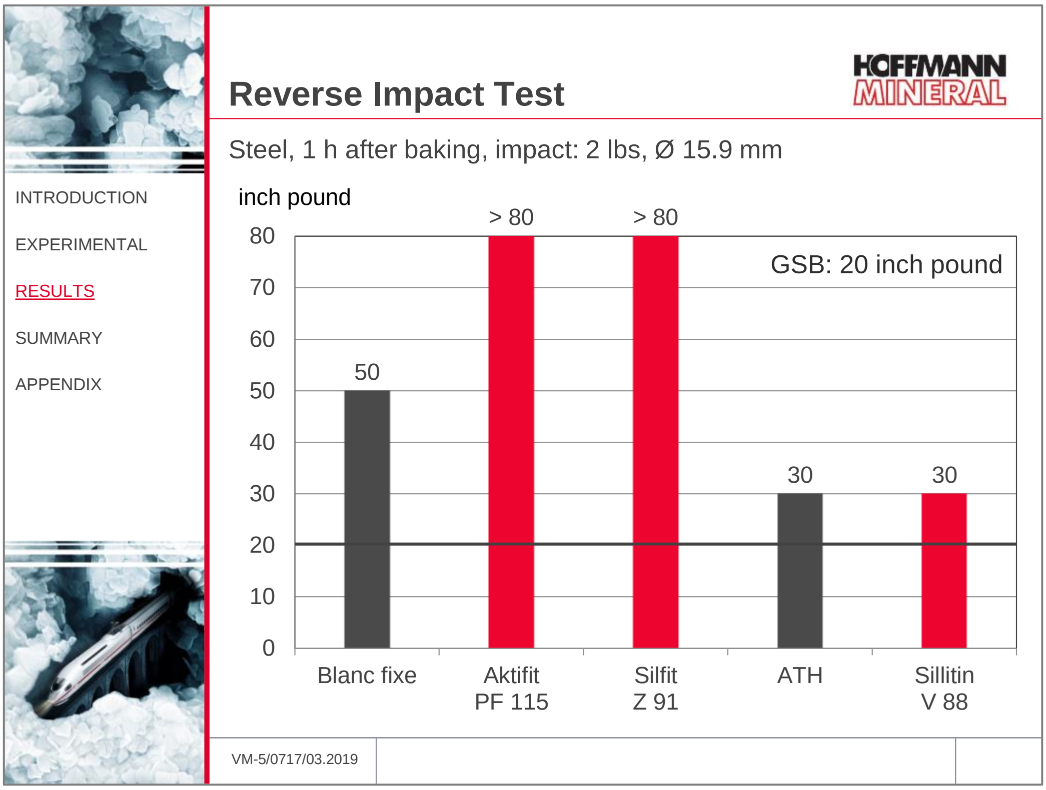

Fig. 13

The result is even more marked if the test is performed after three days with an even smaller sphere diameter of 12.7 mm. Most of the coatings demonstrated virtually no residual flexibility, and cracked at low heights, although Aktifit PF 115 at least achieved 20 inch pounds. The only exception was Silfit Z 91, which sustained an excellent level of flexibility with more than 80 inch pounds (*Fig.14*).

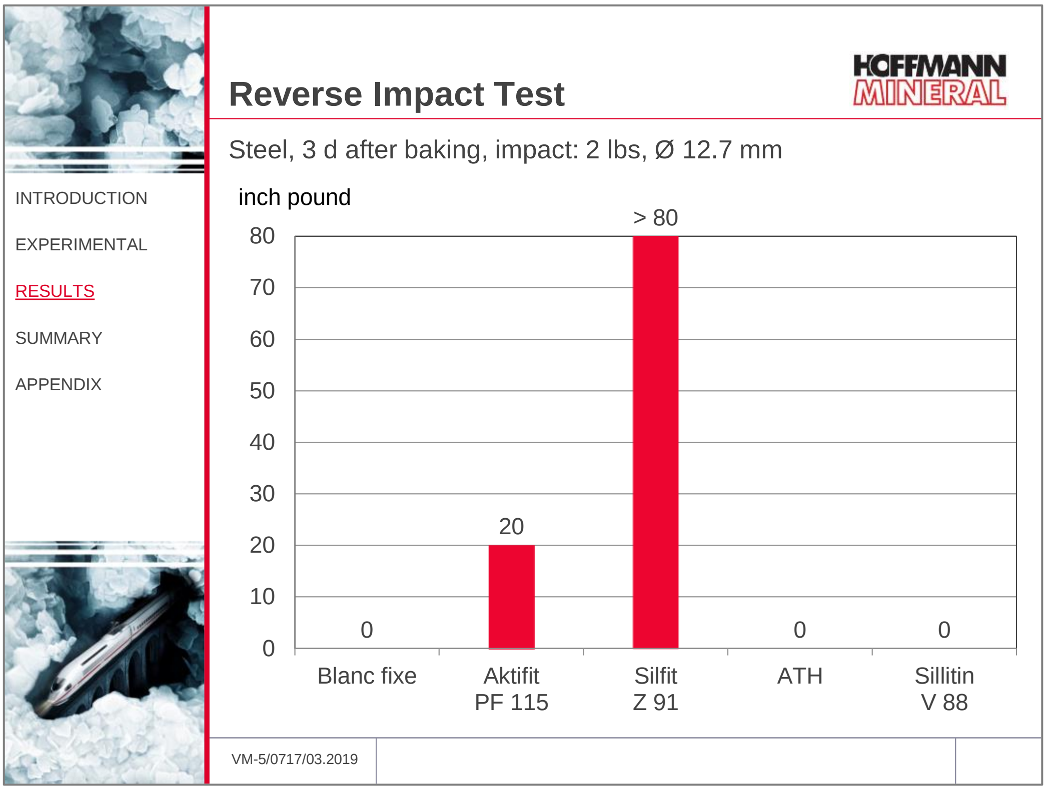

Fig. 14

Additionally, the impact test was also performed by impacting the coated front side with 80 inch pounds and examining the powder coating for cracks. As shown in *Fig. 15*, the only coatings not to demonstrate any cracks under this load were Aktifit PF 115, Silfit Z 91 and Sillitin V 88.

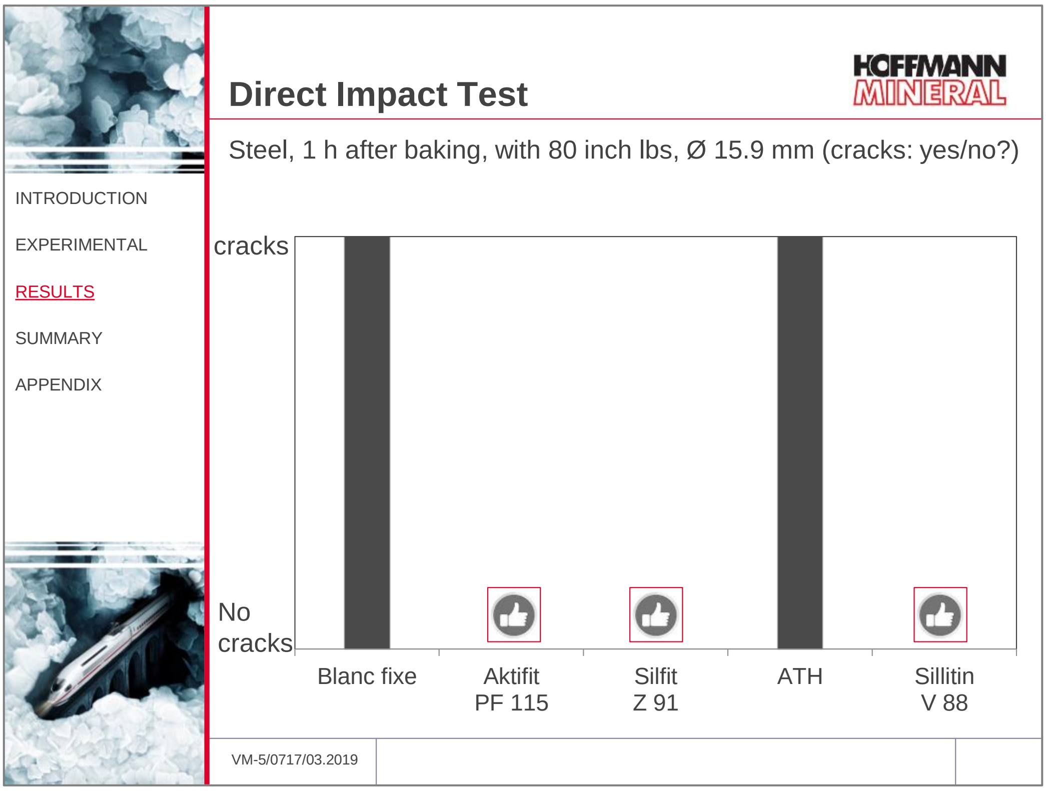

Fig. 15

This test was also repeated after three days and with a smaller sphere diameter. The results were comparable to those of the reverse impact test on the uncoated back side of the specimens, as shown in *Fig. 14*. The coatings containing Aktifit PF 115 and Silfit Z 91 were the only ones without cracks and with continued superlative flexibility, while all other fillers had caused cracks (*Fig.16*).

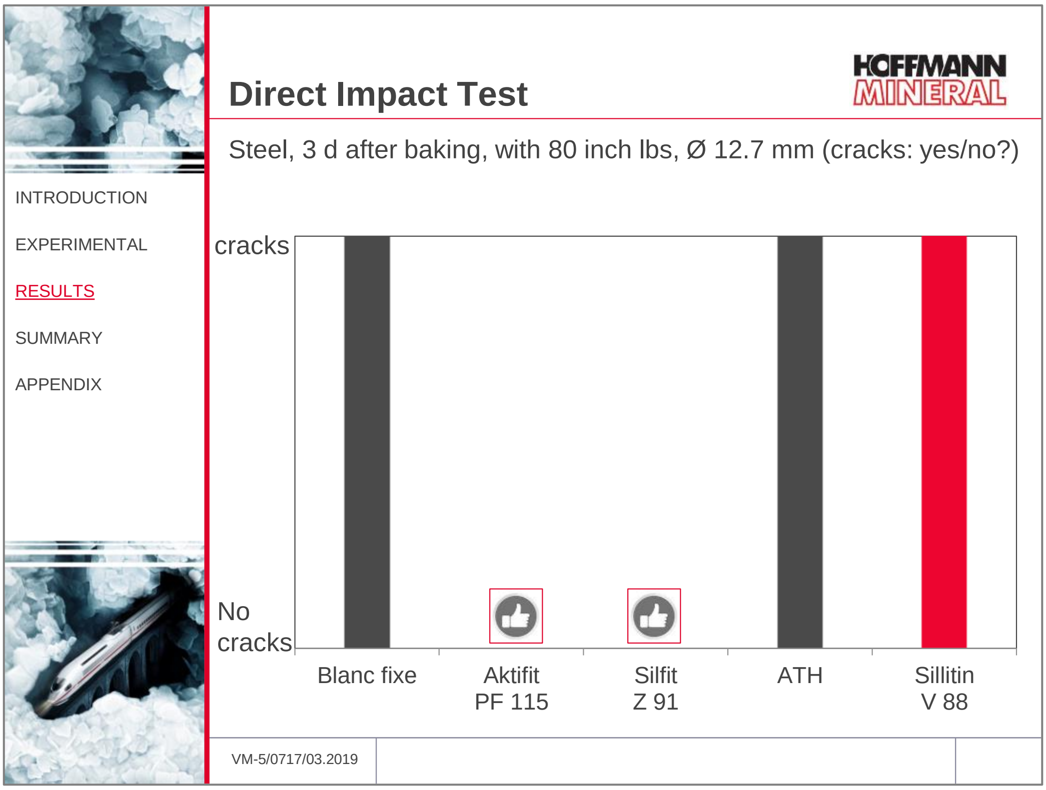

Fig. 16

3.5 Sodium hydroxide test

The caustic soda test with 2-molar solution was performed as per GSB regulations. After 60 minutes, the surface was evaluated for color changes. The resulting shifts in color are shown as Delta E in *Fig. 17*. All tested formulation variations revealed a very slight change in color, with a Delta E of 0.1. As such, no differences were noticeable.

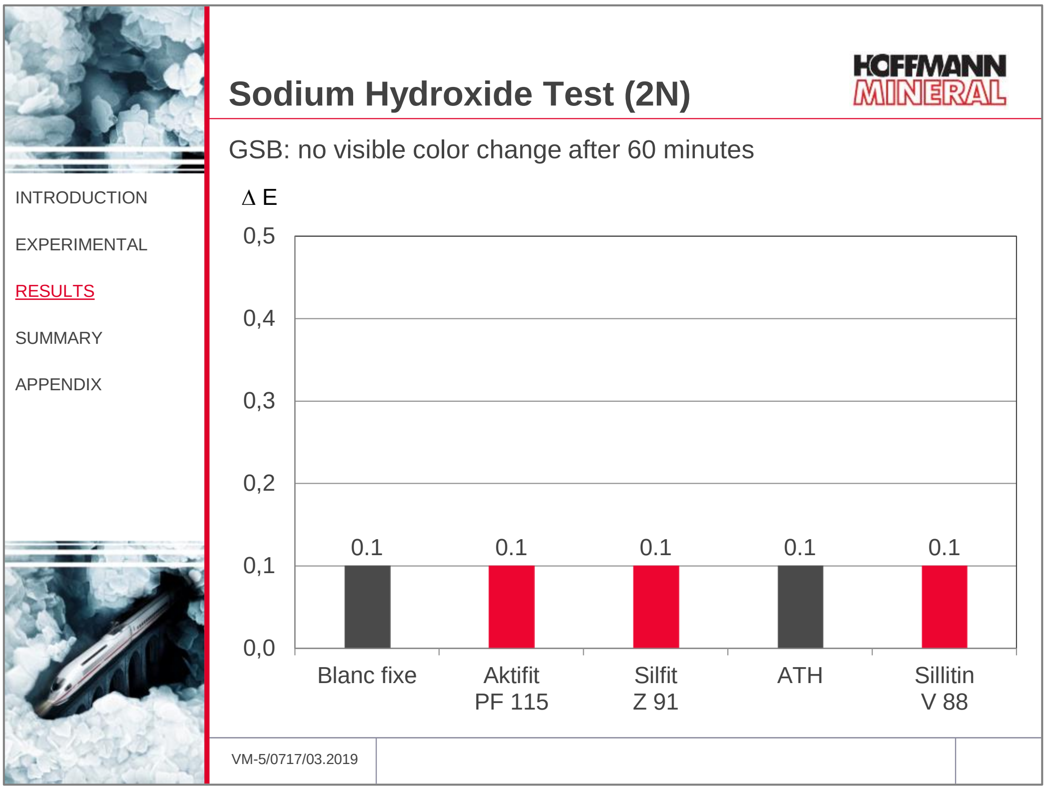

Fig. 17

3.6 Water spot resistance

Water spot resistance was also tested as per GSB regulations. The coating with Blanc fixe suffered significant damage as a result of the humidity impact, with a visible difference in color. The measured Delta L* was at 2.7. All other fillers passed this test without visible color change with a Delta L* of 0.1 *(Fig. 18 and 19).*

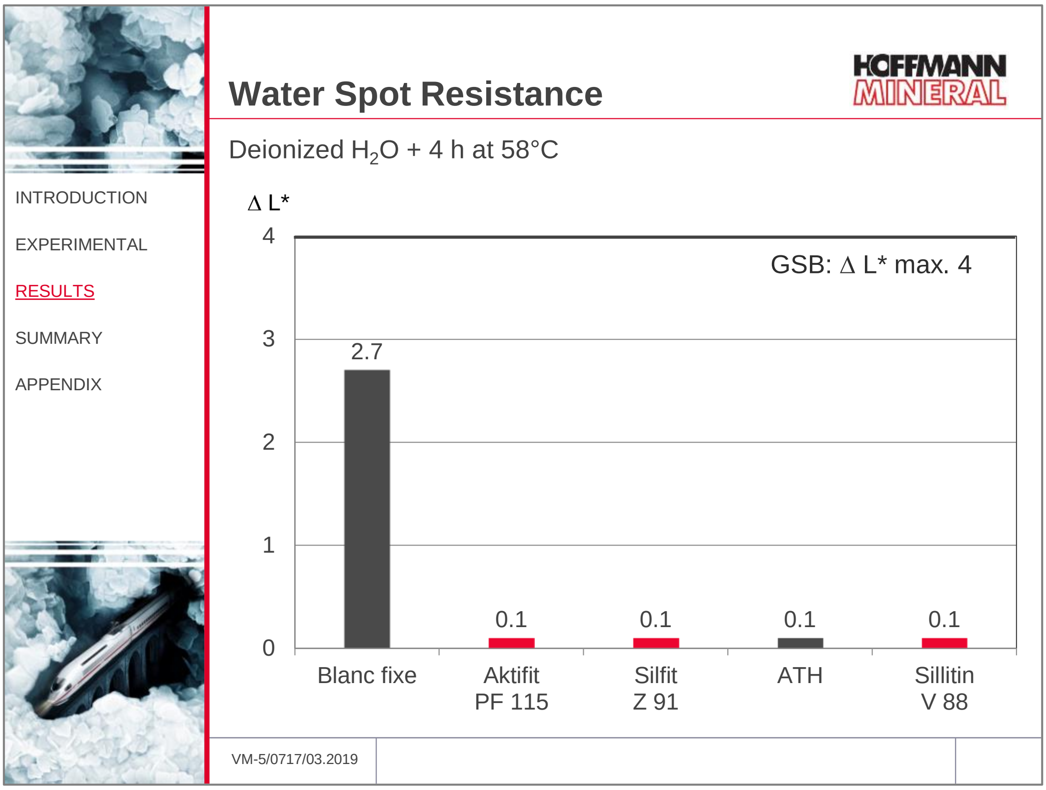

Fig. 18

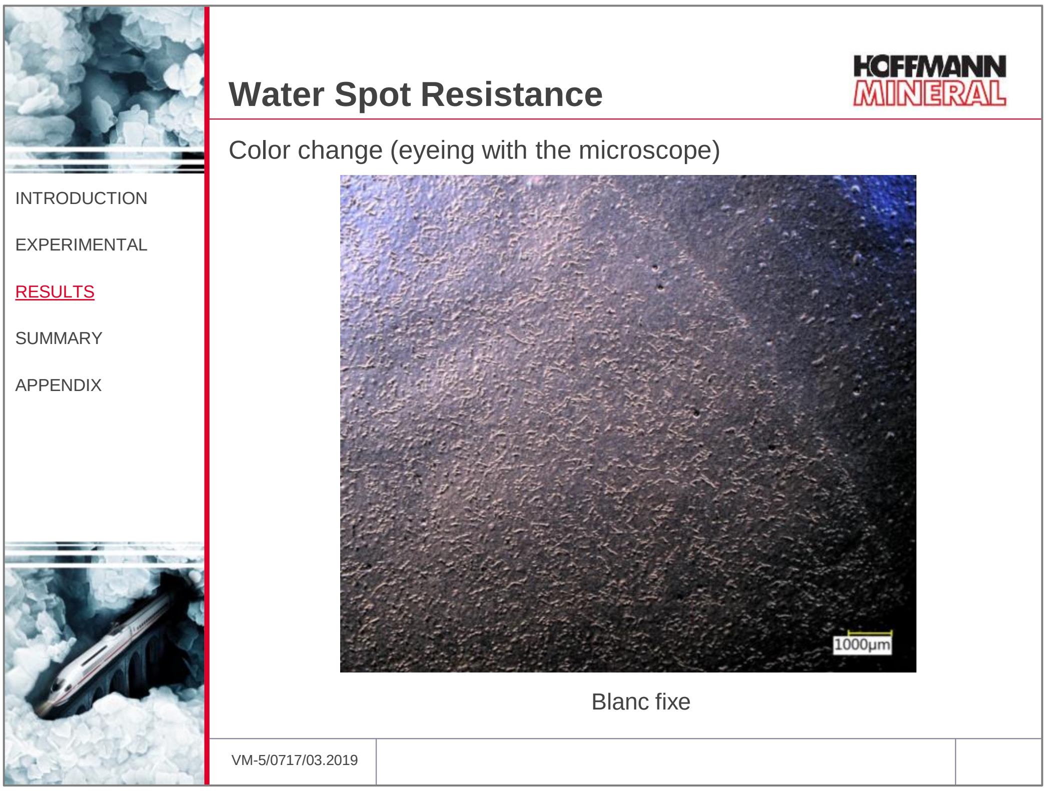

Fig. 19

3.7 Humidity test

The humidity test lasted 1000 hours as specified in DIN EN ISO 6270-2 CH. As with the water spot resistance test in 3.6, the coating using Blanc fixe demonstrated a clearly visible shift in color in terms of Delta E measurement. All other fillers showed no visible change in the color of the coating, with Delta E of less than 0.5 (*Fig. 20*).

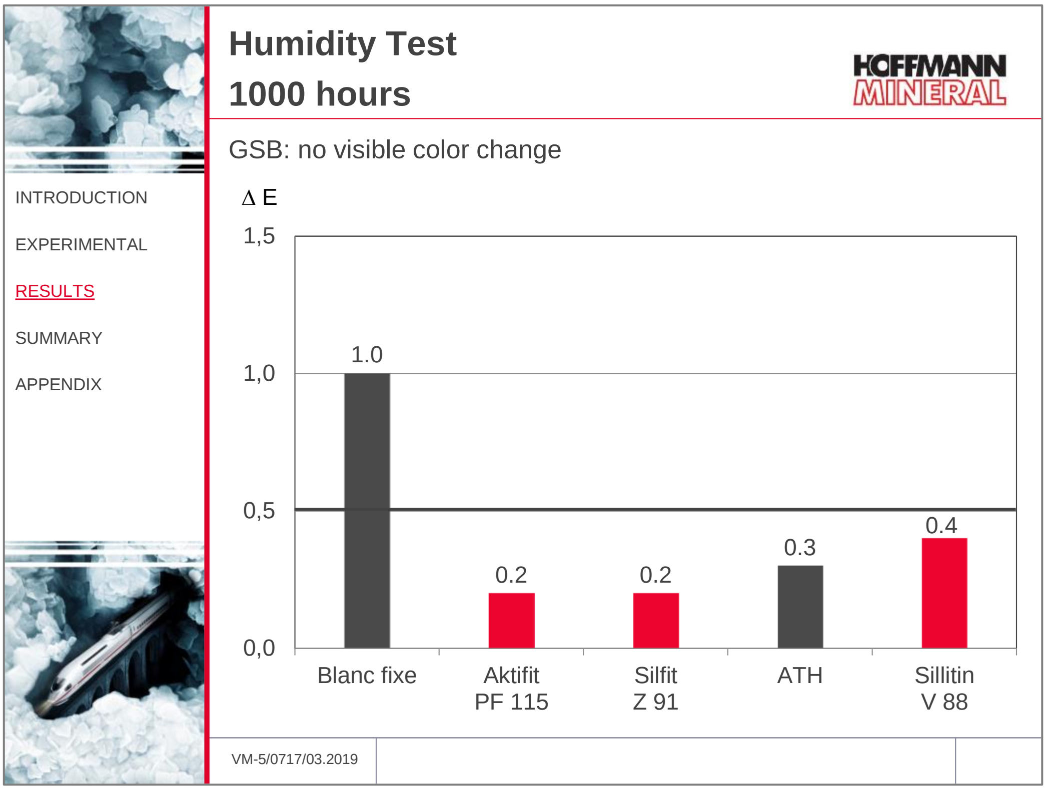

Fig. 20

3.8 Acetic salt spray test

The acetic salt spray test lasted 1000 hours as specified in DIN EN ISO 9227 (AASS). Prior to testing, a scratching tool according to Sikkens was used to cut a diagonal cross (Saint Andrews Cross) into the specimens. After testing, delamination at scribe was evaluated as per DIN EN ISO 4628-8 and blistering according to DIN EN ISO 4628-2. The coatings containing the two reference fillers Blanc fixe and ATH had delaminated completely. By contrast, the Neuburg Siliceous Earth derivate performed much better, with delamination values of 1.5 to 2.3 mm.

After 1000 hours of acetic salt spray testing, none of the formulations showed blisters, the degree of blistering according to the standard was 0 (S0) (*Fig. 21).*

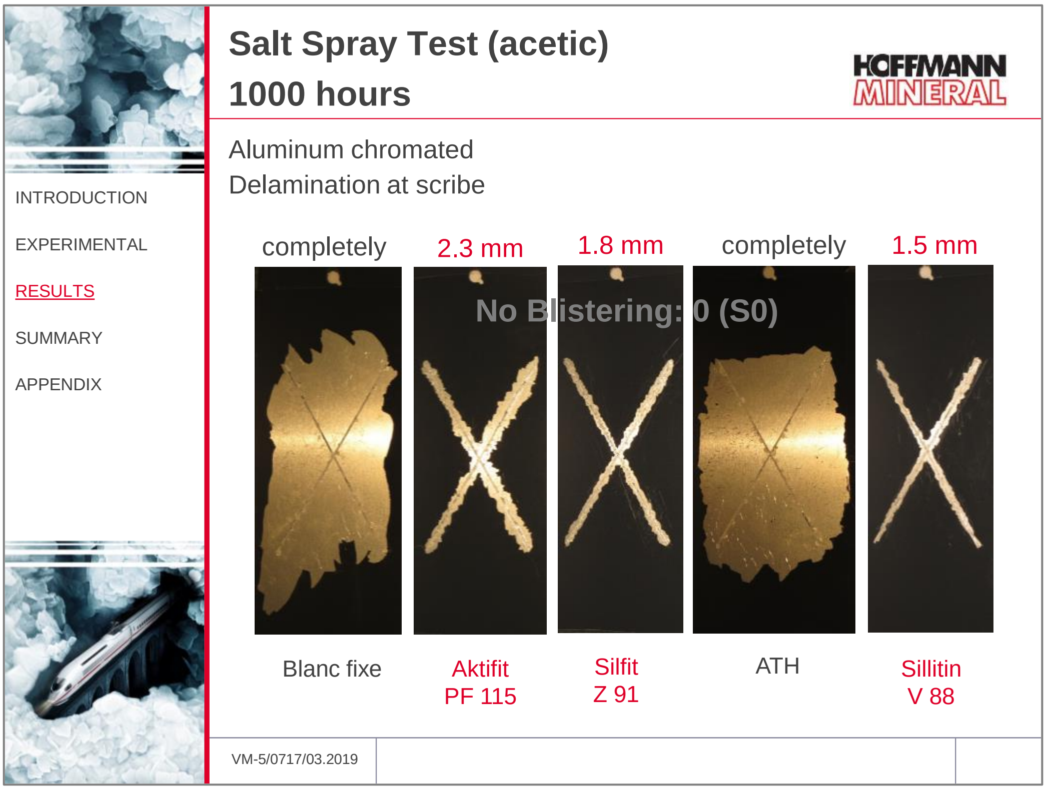

Fig. 21

3.9 Artificial weathering

The coatings were exposed to artificial weathering over a period of 300 hours in alternating cycles of 4 hours each of a condensation phase at 40°C and 4 hours of UVB-313 light at 50°C and a radiation intensity of 0.75 W/m² @ 310 nm. The residual gloss was then measured at 60° angle. The remaining percentage result is shown in *Fig. 22*. All variants satisfied the GSB requirement of > 50% residual gloss. In the high-gloss category, Aktifit PF 115 and Silfit Z 91 performed better than Blanc fixe, with a remaining gloss of more than 70%. In the medium-gloss category, the remaining gloss of Sillitin V 88 was nearly the reference ATH.

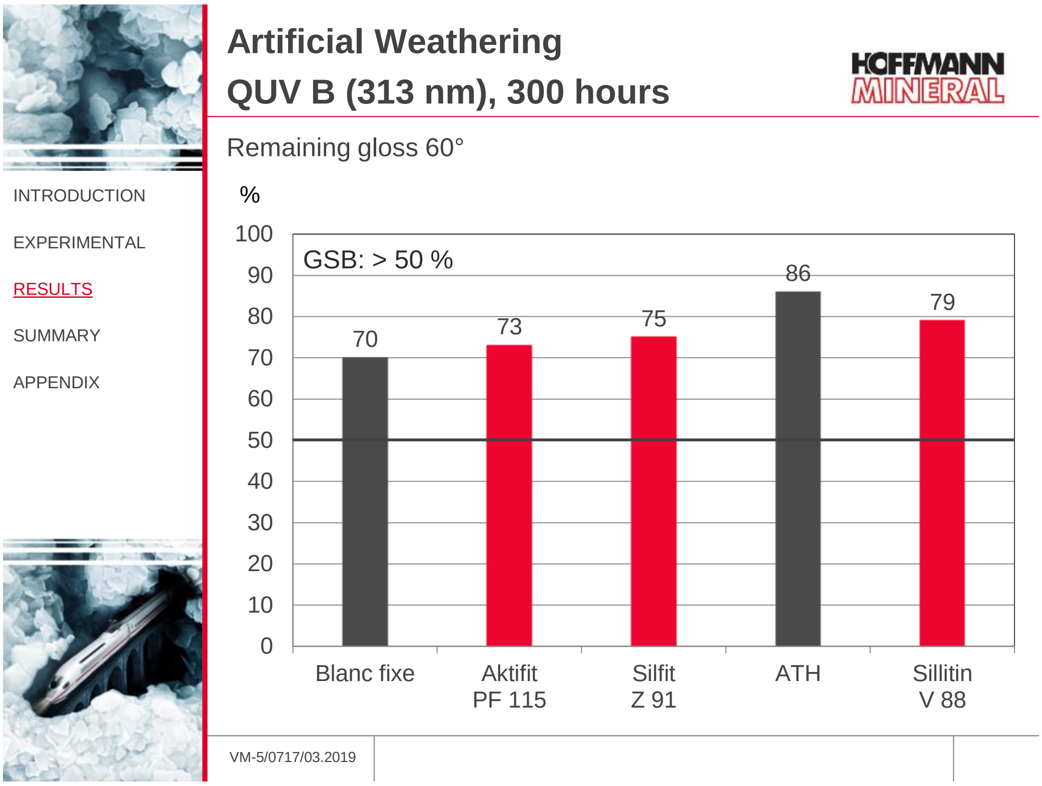

Fig. 22

3.10 Outdoor Exposure Florida

In Florida, the coated panels were exposed South towards direct sun radiation at an angle of 45°. After irradiation of 140 MJ/m² (corresponding to a five-month exposure in Florida under above-mentioned conditions), 280 MJ/m² (11 months) and 420 MJ/m² (16 months), color and gloss (20° and 60°) were measured.
The color change Delta E after 420 MJ/m² was in the range of 0.7 to 1.0, hence it was not possible to show significant differences between the fillers used.

Fig. 23 shows the reduction of the 20° gloss over the time for the high gloss range. In the beginning, the reference showed a gloss higher than both siliceous earth formulas. However, the exposure to the sun for 280 MJ/m² brought the high gloss of the Blanc fixe specimen down to the level of Aktifit PF 115 and close to the level of Silfit Z 91. Therefore, both siliceous earth products show significantly less reduction of gloss than the Blanc fixe comparison.

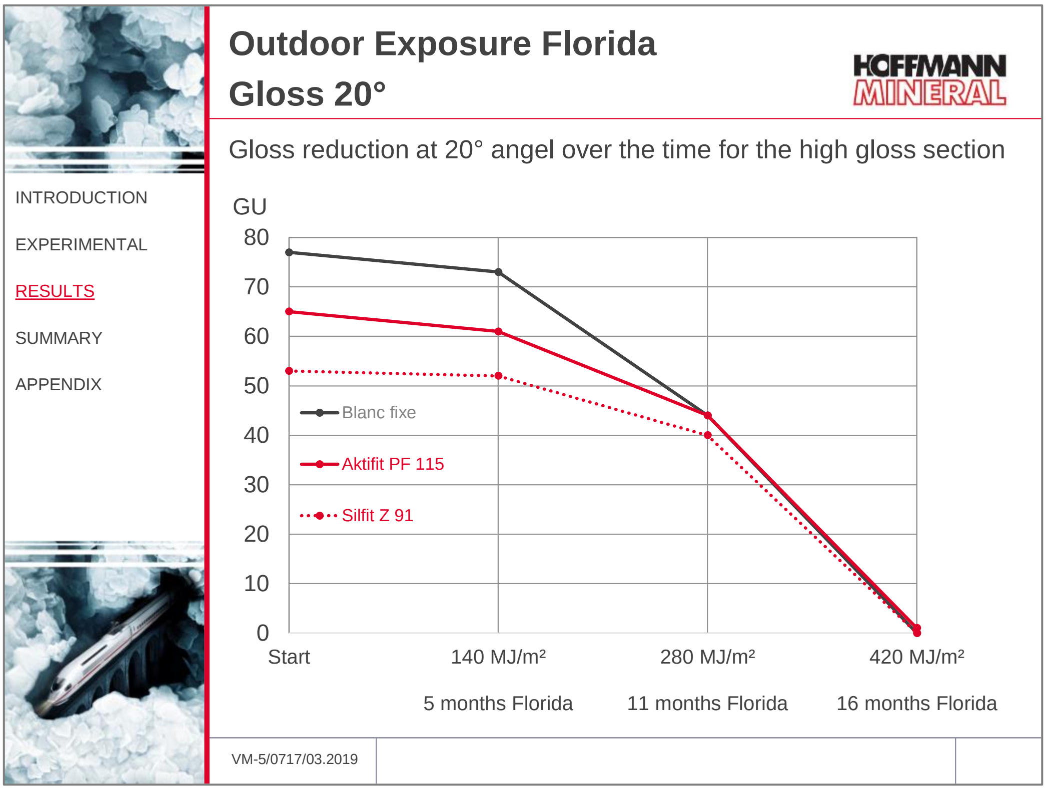

Fig. 23

The reduction of gloss at 60° is shown in *Fig. 24*. Both fillers leading to a medium gloss, ATH and Sillitin V 88 in particular, show only minor reduction of gloss up to an irradiation intensity of 280 MJ/m². Beyond this time of exposure, Sillitin V 88 even shows the highest gloss with 40 GU after 420 MJ/m².

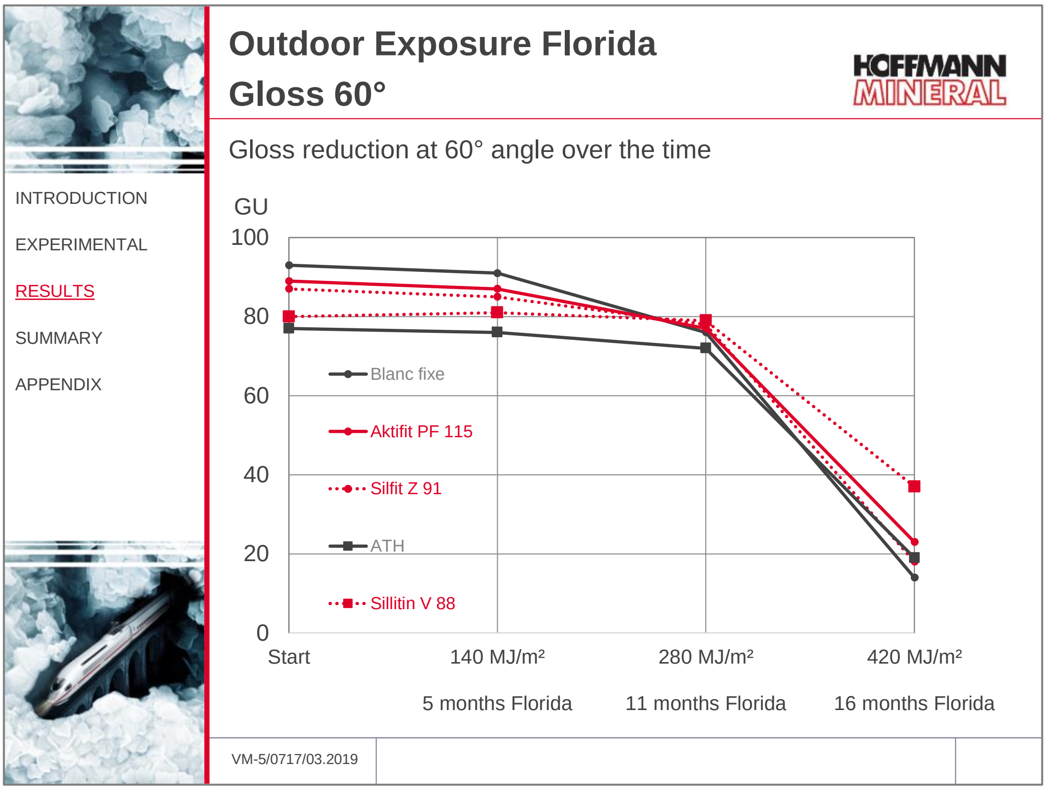

Fig. 24

Fig. 25 depicts the relative remaining gloss at 60° in percent after 280 MJ/m². All varieties reach the level of >= 50% remaining gloss required by GSB. Blanc fixe keeps a remaining gloss of 83% in the high gloss range, surpassed by both Aktifit PF 115 and Silfit Z 91. Sillitin V 88 can keep the medium gloss as well as the reference ATH.

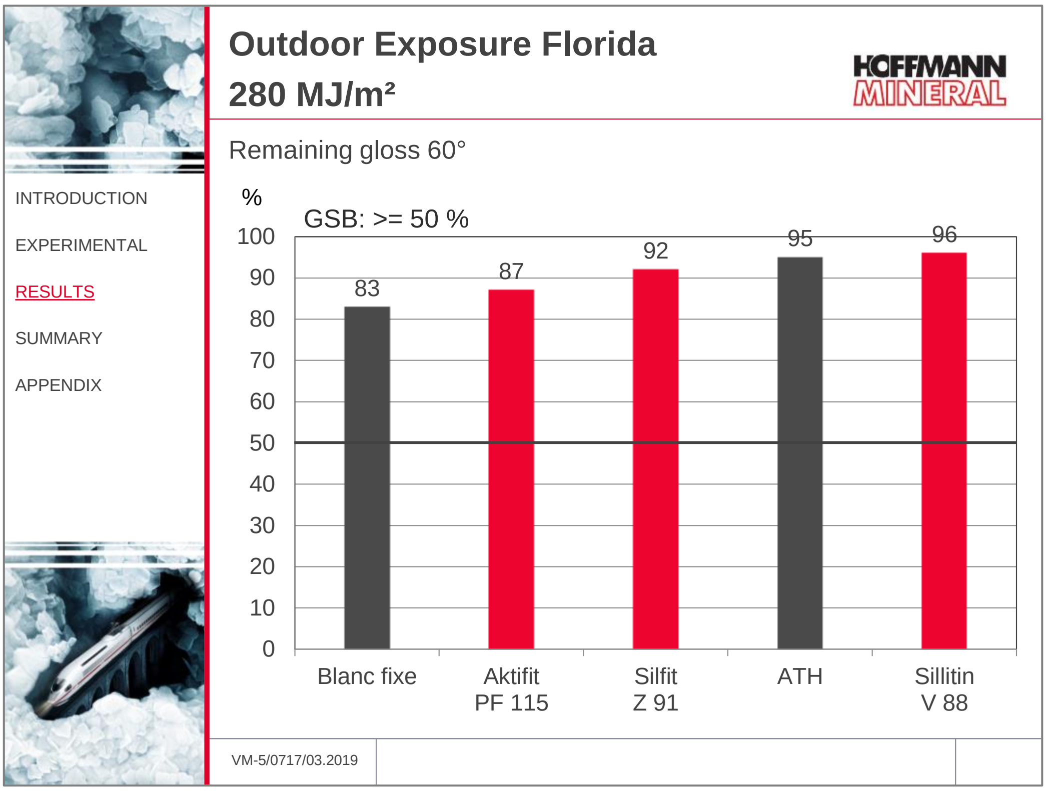

Fig. 25

Fig. 26 displays the relative remaining gloss in percent measured at an angle of 60° after 420 MJ/m². Here, (calcined) Neuburg Siliceous Earth shows an advantage: compared with Blanc fixe, 7-12% higher gloss is preserved in the high gloss range. In the medium gloss range, the remaining gloss achieved through the use of Sillitin V 88 was almost as double high as the one obtained with ATH.

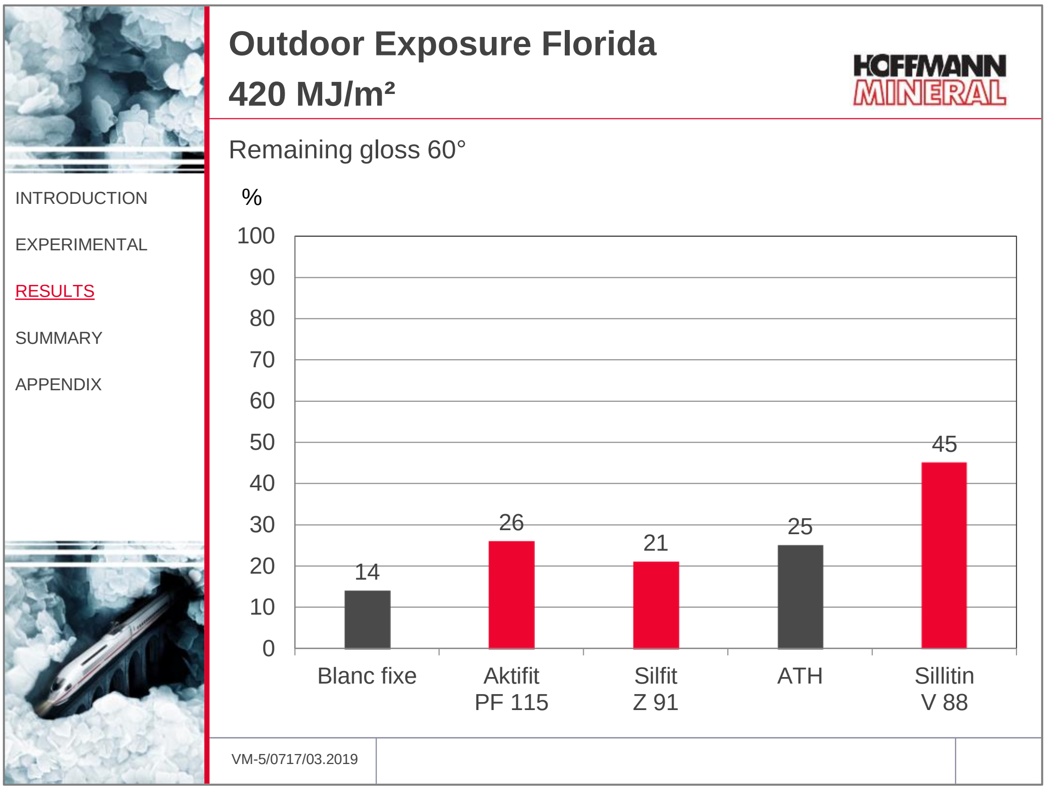

Fig. 26

4. Results

The following effects, depending on the required gloss level, can be achieved by using Neuburg Siliceous Earth in powder coatings (PE/HAA) for outdoor use:

High gloss 60°: ~ 90 GU:

If Aktifit PF 115 or Silfit Z 91 is used instead of the commonly used Blanc fixe:

- the glossy appearance with low haze is largely retained by using Aktifit PF 115

+ the surface appearance / texture is notably improved with the Aktifit PF 115
+ the flexibility of the coating is distinctly improved, especially using Silfit Z 91
+ the anthracite gray color is preserved in full after the water spot resistance test, blushing is prevented
+ delamination following acetic salt spray testing can be significantly reduced
+ the weatherability is slightly improved

Medium gloss 60°: ~ 75 GU:

If Sillitin V 88 is used instead of the common used aluminum hydroxide:

- the good water spot resistance remains comparable
- the desired matting efficiency can be maintained in combination with outstanding film characteristics and optimum surface appearance / texture

+ the flexibility of the coating is improved (direct impact, after 1 hour)
+ delamination following acetic salt spray testing can be significantly reduced
+ the weatherability is slightly improved

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