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Unternehmensgruppe Hoffmann

Neuburg Siliceous Earth

in medium solid epoxy

anti-corrosion coatings

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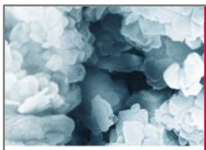
1 Introduction and objectives

Anti-corrosion paints make an important contribution to the conservation of the value of buildings and economical goods. Such coatings often contain zinc phosphate as the active anti-corrosion pigment as well as fillers which are used to further improve technical properties.
Stricter legislations more and more put limits on the use of high amounts of zinc phosphate. The lower the anti-corrosion pigment content in a formulation, the more important becomes the suitable selection of the fillers. Differences in the roughness of the substrate surface to be coated represent a further challenge.
With this background, the question appeared of interest if high performance functional fillers based on Neuburg Siliceous Earth are able to compensate the reduced efficiency of the lower anti-corrosion pigment concentration.
Therefore, formulations with different zinc phosphate contents and varied filler combinations were assessed in classical salt spray and humidity (condensation water) test. Furthermore, the additional use of aminosilane as an adhesion promoter was evaluated.

2 Experimental

2.1 Base formulation

The starting point of the present work was a commercial two-component anti-corrosion primer based on a standard epoxy resin and a polyamide resin as hardener component. The solids content of 68 % and a volatile organic matter content (VOC) of 430 g/l indicate the «medium solids» character of the formulation. *Fig. 1* lists the recipe of the base formulation (control) which aside from zinc phosphate as active anti-corrosion pigment contained a classical filler combination made up from talc and barium sulfate.



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Base Formulation

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	Anti-Corrosion Primer, 2P Epoxy-Polyamide, medium-solid		parts by weight
A	Epikote 1001 x 75	Solid epoxy resin based on Bisphenol A, 75% in xylene, EEW 633	23.8
	Bentone 34 paste	Rheological additive, 10% in xylene / ethanol 87:3	4.3
	Xylene	Solvent	6.5
	Ethylglycol	Solvent	4.7
	MIBK	Solvent, Methylisobutylketone	6.6
	Nusa 57	Wetting and dispersing additive	0.4
	BYK-354	Leveling additive	0.8
	Sachtleben RD3	Pigment, Titanium dioxide	5.9
	Zinc phosphate	Anti-corrosion pigment	7.5
	Talc	Filler, hydrated magnesium silicate	19.0
	Blanc fixe	Filler, barium sulfate ppt	7.8
B	Versamid 115 x70	Polyamide resin, 70% in xylene, HEW 283	12.7
Total			100.0
Solids content w/w		[%]	68.0
Pigment volume concentration (PVC)		[%]	33.8
Volatile organic compounds (VOC)		[g/l]	430

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Fig. 1

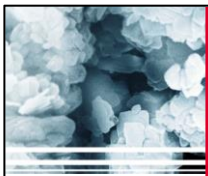
2.2 Fillers

Fig. 2 summarizes the characteristical properties of the fillers included in the study. The results were obtained in our laboratory, and therefore are comparable.

The talc gave a medium oil number and is characterized by a fairly coarse lamellar grain shape. As a further typical filler for anti-corrosion applications from competition, a natural mixture of quartz, mica and chlorite was included. Morphologically, it shows a mixed structure of corpuscular and lamellar constituents. The grain size again is rather coarse; oil number and specific surface area are in the area of talc.

In comparison with these fillers, the performance of Neuburg Siliceous Earth was evaluated. This is a natural combination of corpuscular, crypto-crystalline and amorphous silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural formation, the silica portion exhibits a round grain shape and consists of aggregated, crypto-crystalline primary particles of about 200 nm diameter, which are covered by amorphous silica opal-like. The particular structure and the fine grain size explain the relatively high specific surface area.

The test program involved two modified grades whose interaction with the polymer matrix has been suitably adjusted via functionalization of the filler surface. Aktisil PF 777 is a version of Neuburg Siliceous Earth which has been hydrophobically modified with an alkyl functional group, while Aktisil AM is distinguished by a surface treatment with an amino functional group.



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Filler Characteristics

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		Talc	Natural Mixture of Quartz, Mica, Chlorite	Neuburg Siliceous Earth: Aktisil PF 777	Neuburg Siliceous Earth: Aktisil AM
Morphology		lamellar	corpuscular / lamellar		
Density	[g/cm³]	2.8	2.8	2.6	2.6
Particle size d_{50}	[µm]	6.8	8.0	2.2	2.2
Particle size d_{97}	[µm]	18	27	10	10
Oil absorption	[g/100g]	45	43	35	45
Specific surface area BET	[m²/g]	5	5	8	9
Surface treatment		---	---	alkyl functionalized hydrophobic	amino functionalized

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Fig. 2

2.3 Formulation variations

As shown in *Fig. 3,* the zinc phosphate portion was partially or totally replaced by talc at equal volume. A second step aimed at varying the type of the filler. In order to maintain the same PVC, in view of the different filler densities slightly different loadings had to be applied. The amount of precipitated barium sulfate was kept constant in all formulation variations. If used, the aminosilane was introduced after mixing it with the hardener.

Variation Formulation

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Substitution of anti-corrosion pigment by filler, PVC constant

	Control [pbw]	Reduced [pbw]	Without [pbw]
Zinc phosphate	7.5	2.5	0
Talc	19.0	22.8	24.7
		Substitution of filler type, PVC constant	
Natural mixture of quartz, mica, chlorite		22.8	24.7
Neuburg Siliceous Earth grades		21.3	23.1

Addition of 1 pbw of (3-Aminopropyl)triethoxysilane to part B (Versamid) as indicated

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Fig. 3

2.4 Preparation

The batches were prepared in a dissolver equipped with adapted bead mill. After mixing the components in the sequence of the formulation, dispersion was achieved by grinding for 15 minutes with 2 mm glass beads at a peripheral speed of the grinding disk of 7.8 m/s. After adding and mixing the hardener component, the complete formulation was applied by air spraying (4 bar, nozzle 1.2 mm) with a resulting dry film thickness for all formulations of about 80 µm. The chosen single-layer application generated effectively critical starting conditions for the subsequent corrosion tests, as every smallest film defect becomes visible as a rusty point.
After this, the test samples were dried and conditioned during 14 days at 23 °C and 50 % relative humidity.
Immediately before testing part of the coatings were scratched according to van Laar with a scribe (half-rounded metal tip).
The substrates were blasted sheets made up of unalloyed, cold-rolled steel 150 x 100 x 2 mm. In order to simulate the situation for immovable steel buildings, a blasted surface according DIN EN ISO 12944 Part 6 with a degree of surface treatment Sa 2½ at a surface roughness «medium (G)» was selected.
In addition, resistance tests were run on non-blasted steel (Q Panel type R-48, 200 x 100 x 0.8 mm). This substrate, because of its relatively smooth surface, proved very critical compared to blasted steel.

2.5 Assessment criteria for corrosion protection

For the assessment of the corrosion protection efficiency, the humidity test according to DIN EN ISO 6270-2 CH and the classical neutral salt spray test according to DIN EN ISO 9227 were carried out. The assessment of the damage was made on the surface as well as at the scribe which was applied in a defined way, with the individual specific criteria as listed in *Fig. 4*.

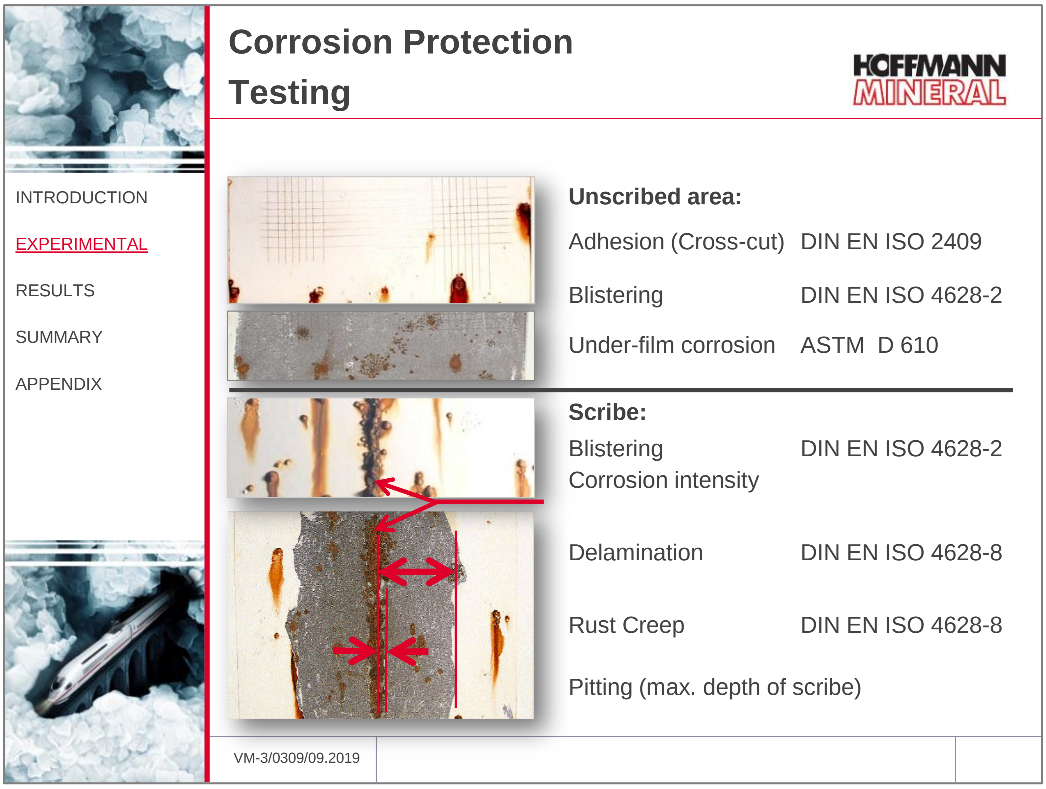
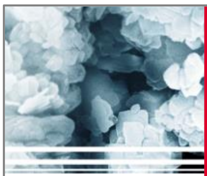


Fig. 4

While the standardized assessment criteria can be fully measured in a quantitative way, the amount of rust at the scribe and the depth of corrosion via eliminated substrate material give approximative information about the intensity of the corrosion process.
For the total assessment the level of the individual test result was translated into a numerical rating between 0 and 10, as shown in *Fig. 5*. With equal weighting of the individual characteristics, the amount of the resulting cumulative figure allows to obtain a direct definition of the performance level of the coating system.



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Corrosion Protection Performance Evaluation

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	Criterion	rating number	10	9	8	7	6	5	4	3	2	1	0
	Unscribed Area												
1	Adhesion Cross-cut	[GT]	0	0-1	1	1-2	2	2-3	3	3-4	4	4-5	5
2	Blistering	Quantity Size	- -	1 1	2 2	2-3 2-3	3 3	3-4 3-4	4 4	4-5 4-5	5 5	>5 >5	compl. Delam.
3	Under-film corrosion	[%]	0	0,03	0,1	0,3	1	3	10	16	33	50	100
	Scribed												
4	Blistering	Quantity Size	- -	1 1	2 2	2-3 2-3	3 3	3-4 3-4	4 4	4-5 4-5	5 5	>5 >5	compl. Delam.
5	Delamination	[mm]	0	2	4	7	10	13	16	20	25	30	40
6	Rust creep	[mm]	0	0,5	1	2	3	5	7	9	12	15	20
7	Pitting	[mm]	0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9	1,0
8	Corrosion intensity	-	very low		low		moderate			high		very high	
	1 - 8 cumulative rating 80 max.		80	72	64	56	48	40	32	24	16	8	0
			very good										very bad

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Fig. 5

3 Results

3.1 General properties

3.1.1 Producibility

The filler combinations in their individual loadings are generally distinguished by good incorporation properties. Minor advantages are observed for Aktisil AM and the blend of quartz, mica and chlorite. The latter filler after grinding shows somewhat coarser particles according to Grindometer test (*Fig. 6*).

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Producibility

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Part A

		Control 7.5	2.5 pbw respectively 0 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Ease of filler incorporation		☺	☺	☺☺	☺	☺☺
Fineness of grind	[µm]	5	5	10	5	5

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Fig. 6

3.1.2 Storage stability

The storage stability after 60 days at 23 °C can be rated as very good for all formulations. The base batch is free from sediment; serum and filler containing phases can be easily homogenized.

3.1.3 Rheology and leveling

The rheological properties were determined in a Rheometer MCR 300 (Paar) with a cylinder system under rotation conditions. In *Fig. 7*, the representative viscosity plots of the formulations with reduced anti-corrosion pigment content are illustrated for comparative purposes.

The higher viscosity in the low shear region for all formulations gives an indication of structure viscosity which, however, comes out differently depending on the individual fillers. In comparison with the quartz/mica/chlorite blend, the diagram for talc shows a distinctly higher level. By contrast, the viscosity curve of Aktisil AM is very similar to the last-mentioned filler blend, and therefore is situated in the lower region.

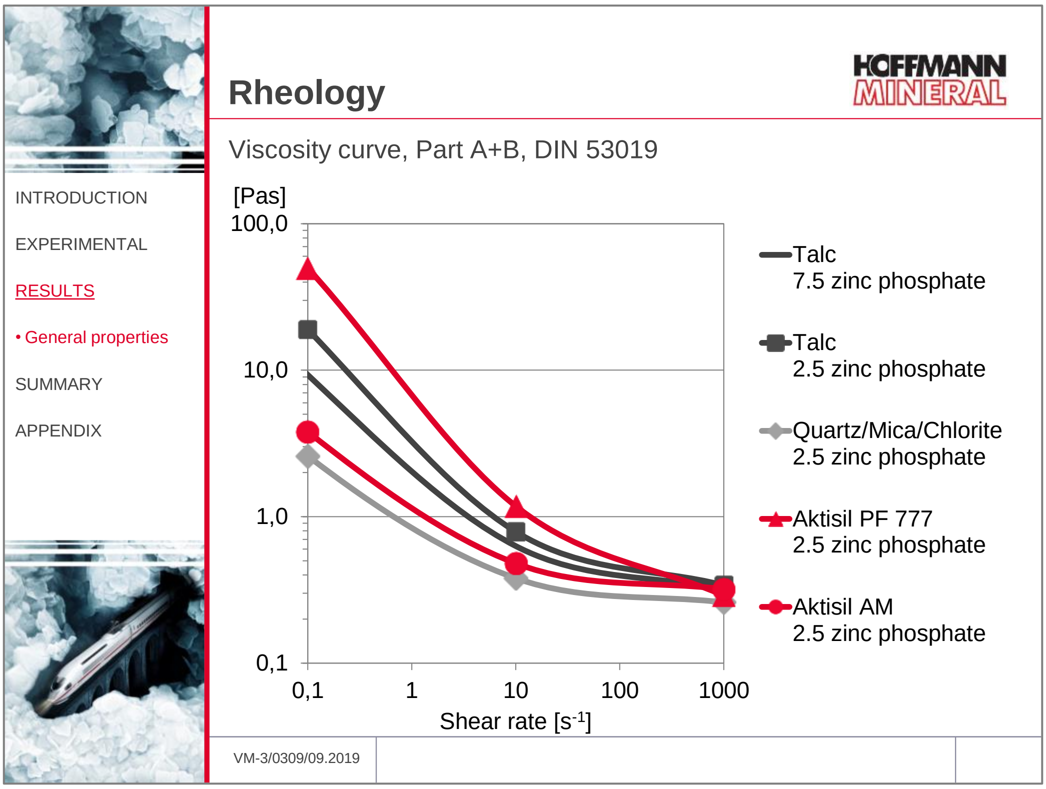
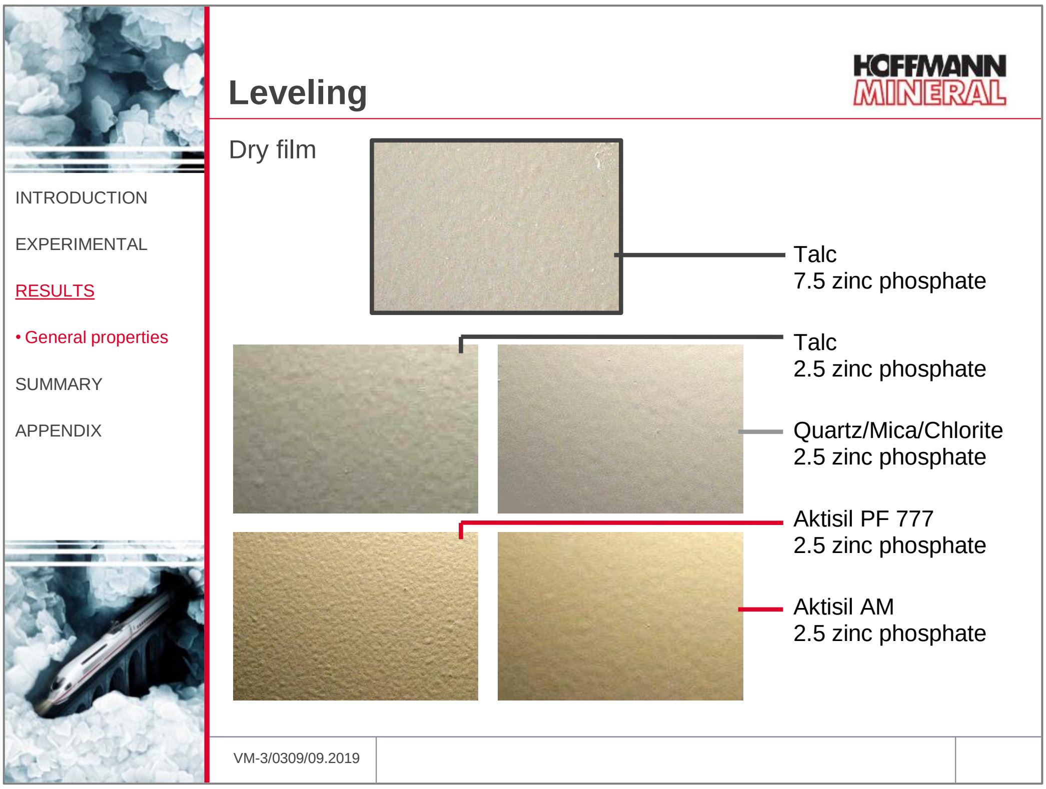


Fig. 7

The rheological activity of Aktisil PF 777 results in strongly shear-thinning, thixotropic properties with the heaviest structure increase in the low shear region. Parallel to this, a yield point appears. As a result of this characteristic, the sag resistance on vertical application areas, even at higher layer thicknesses, is improved. With rather lower film thicknesses, a more pronounced structured surface compared with talc can be observed (*Fig. 8*).

Moreover, this rheological effect of Aktisil PF 777 offers the possibility to leave out the bentonite paste usually added as rheological additive.

For higher leveling requirements on the applied films, Aktisil AM would be preferred, which offers the same good flow properties as the quartz/mica/chlorite mixture.


Leveling
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Dry film
Talc
7.5 zinc phosphate
Talc
2.5 zinc phosphate
Quartz/Mica/Chlorite
2.5 zinc phosphate
Aktisil PF 777
2.5 zinc phosphate
Aktisil AM
2.5 zinc phosphate
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Fig. 8

3.1.4 Coating hardness

The development of the hardness of the coatings was followed with the aid of a König pendulum over 14 days. The hardness of the control and of the formulation with reduced zinc phosphate content with talc is situated on a low level (*Fig. 9*). The batches with Aktisil AM or the quartz/mica/chlorite blend give evidence of a largely similar trend with slight advantages for the amino functional treated filler, while the final hardness comes out markedly higher than for the talc.

Of interest is the unusually rapid hardness increase when using Aktisil PF 777, which already attains a pendulum hardness of up to 27 seconds higher after two days. Also the final hardness with Aktisil PF 777 comes out highest. This means freshly coated objects should offer a better resistance to mechanical loads.

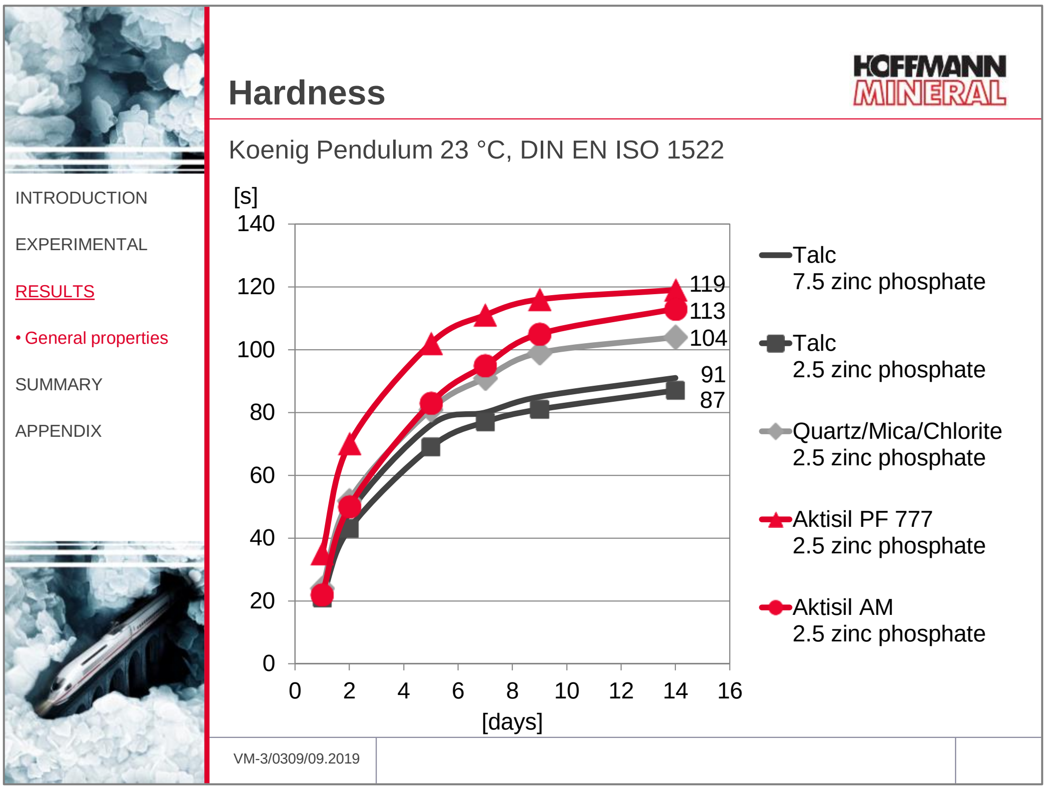

Fig. 9

3.2 Corrosion protection on blasted steel

3.2.1 Reduced addition of zinc phosphate

Prior to the exposure tests, all formulation variants without exception are distinguished by outstanding substrate adhesion with cross-cut results of GT 0.

The very good resistance of the formulations after 1000 h condensation water test is illustrated in *Fig. 10* with the example of a scribed sample sheet. Deterioration of the excellent adhesion or of other properties cannot be observed.

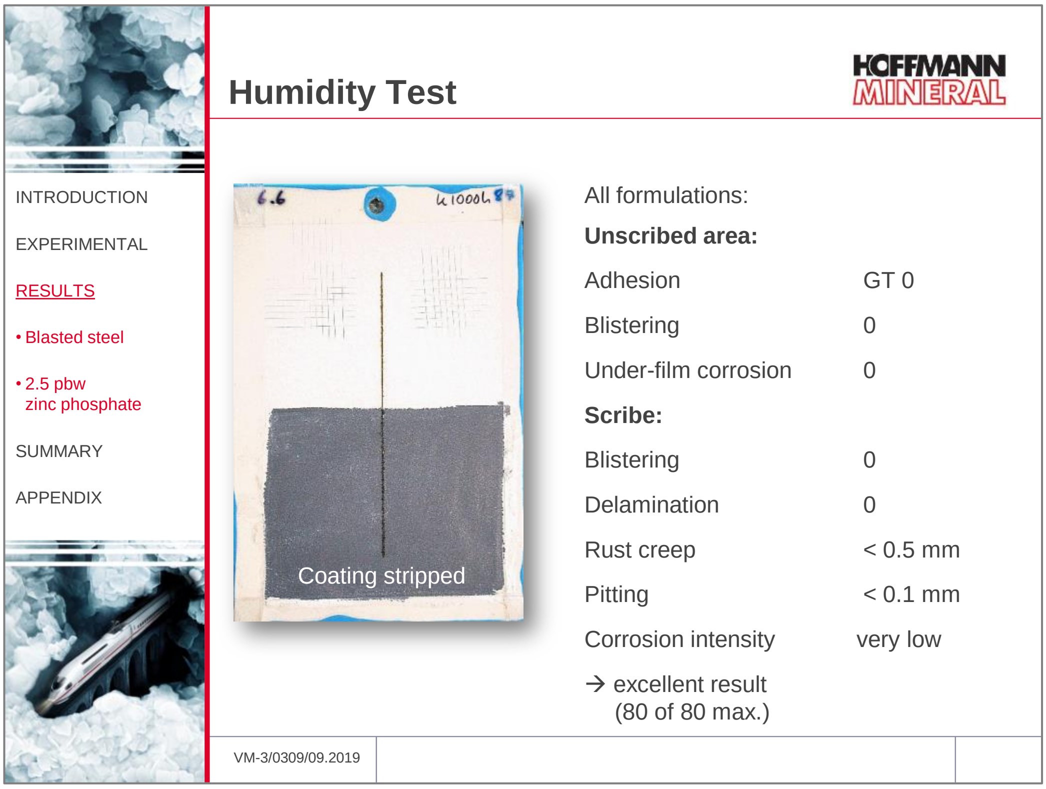

Fig. 10

Heavily corrosive environmental conditions, such as simulated in the salt spray test, let come out differences in the performance of the formulations much more distinctly.

Fig. 11 shows representative results of the salt spray test for the formulations with 2.5 pbw zinc phosphate after an exposure time of 1000 hours. The control formulation with the original portion of anti-corrosion pigment is included for comparison. The reduction of the zinc phosphate addition in the formulation with talc gives unfavorable results with respect to the barrier properties on the surface. Despite only some individually visible blisters many weak areas lead to locally changed adhesion properties, as becomes evident after stripping the coating. The optically darker regions without remaining coating residues point to a deteriorated adhesive strength. In the cross cut test, however, this situation is not yet observed.

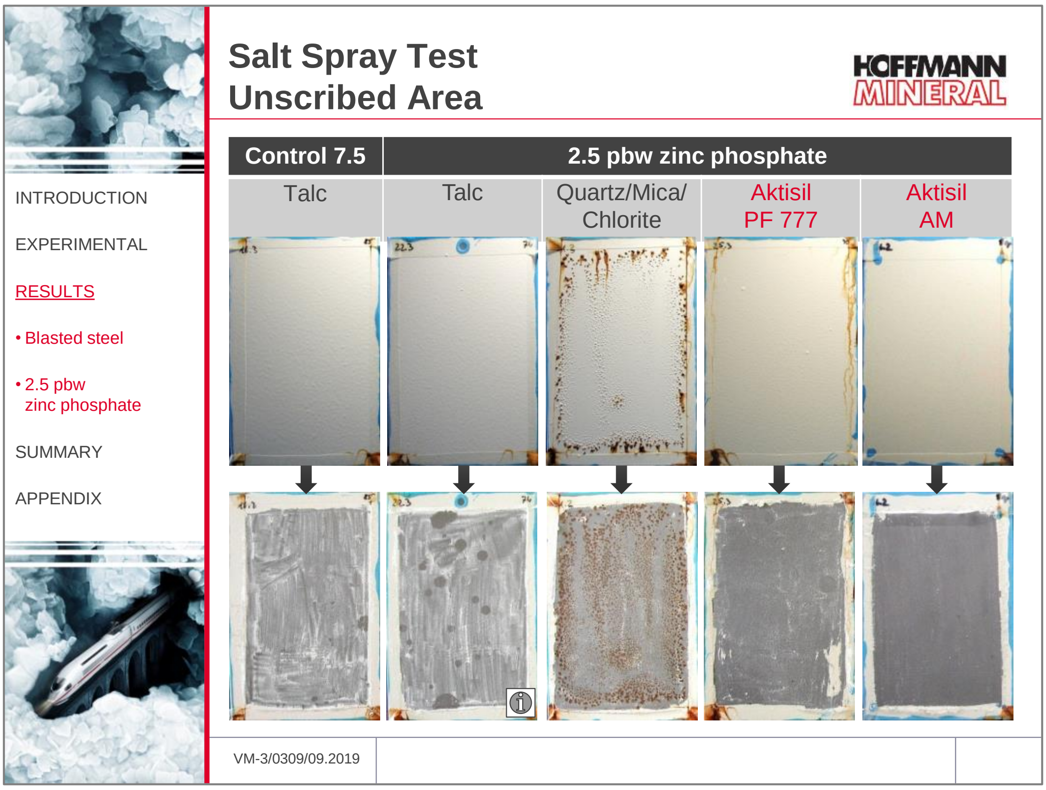

Fig. 11

The blend of quartz, mica and chlorite gives rise to intense blistering. Despite the maintained zinc phosphate content numerous rust perforations are found in the coating. The corrosive attack onto the underlying substrate is an indication of the complete loss of the protective function.

The Neuburg Siliceous Earth grades, by contrast, come up with an excellent result over the surface. Aktisil PF 777 and Aktisil AM not only counteract a breakthrough of rust, but minimize also the appearance of blisters compared with the similar formulation with talc. In particular Aktisil AM impresses by a blister free coating surface, and offers similarly good protection of the surface as the control formulation.

At the scribe, the reduced zinc phosphate content of the formulation with talc leads to an increased formation of corrosion products. The pronounced dissolving of the metal in the immediate damage area is illustrated by a large depth of pitting. With regard to the width of delamination (*Fig. 12*, lower line) and to corrosion the formulation comes out similar to the control with full zinc phosphate addition. First rust perforations away from the damage area, however, indicate a beginning loss of the basic protection efficiency over the surface.

The natural mixture of quartz, mica and chlorite in total has to be judged unsatisfactory.

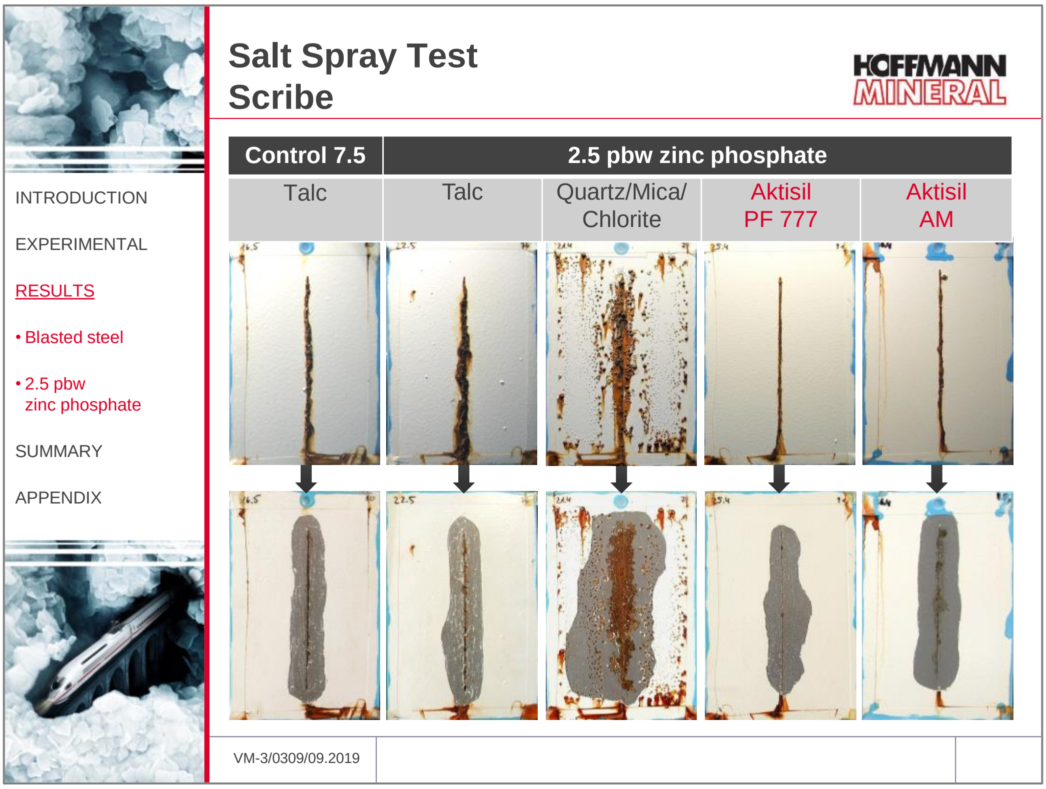

Fig. 12

Aktisil PF 777 and Aktisil AM with respect to the width of delamination give almost the same results as the control and the comparative formulation with talc. Of particular advantage, however, Neuburg Siliceous Earth has proven for the rust formation at the scribe. Both grades show markedly reduced corrosion intensity at the scribe in form of hardly detectable rust volume (*Fig. 12*, upper line). Obviously, the higher resistance to anodic iron solution counteracts the corrosion at the scribe. This shows off also after the stripping of the coating through the markedly reduced width of underrusting and the reduced pitting (*Fig. 12*, lower line). In particular Aktisil PF 777 imparts a very high resistance so that despite the scribe damage a nearly rust-free surface is obtained. In combination with a reduced addition of zinc phosphate, therefore, Aktisil PF 777 acts synergistically and for the protective function at the scribe even surpasses the performance of the control formulation with the high zinc phosphate content.

The assessment data are summarized in *Fig. 13*. The cross-cut test confirms the very good adhesion of all formulations 24 hours after the exposure.

Salt Spray Test
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• 2.5 pbw zinc phosphate
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		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0	0	0	0
Blistering	Quantity Size	0 -	1 S3	4-5 S3	1 S2	0 -
Under-film corrosion	[%]	0	0	25	0	0
Scribe						
Blistering	Quantity Size	1 S4	1 S3	5 S3	0 -	1 S4
Delamination	[mm]	11	12	23	12	15
Rust creep	[mm]	0.5	0.7	5.5	0.1	0.4
Pitting max.	[mm]	0.3	0.8	0.2	0.2	0.3
Corrosion intensity	-	moderate	high	very high	very low	low

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Fig. 13

Fig. 14 graphically summarizes the individual results for the performance of the formulations tested. For the conventional fillers, the afore-mentioned performance deficiencies are clearly evident, in particular very pronounced through the increasing corrosion tendency at the scribe and the blistering on the surface.

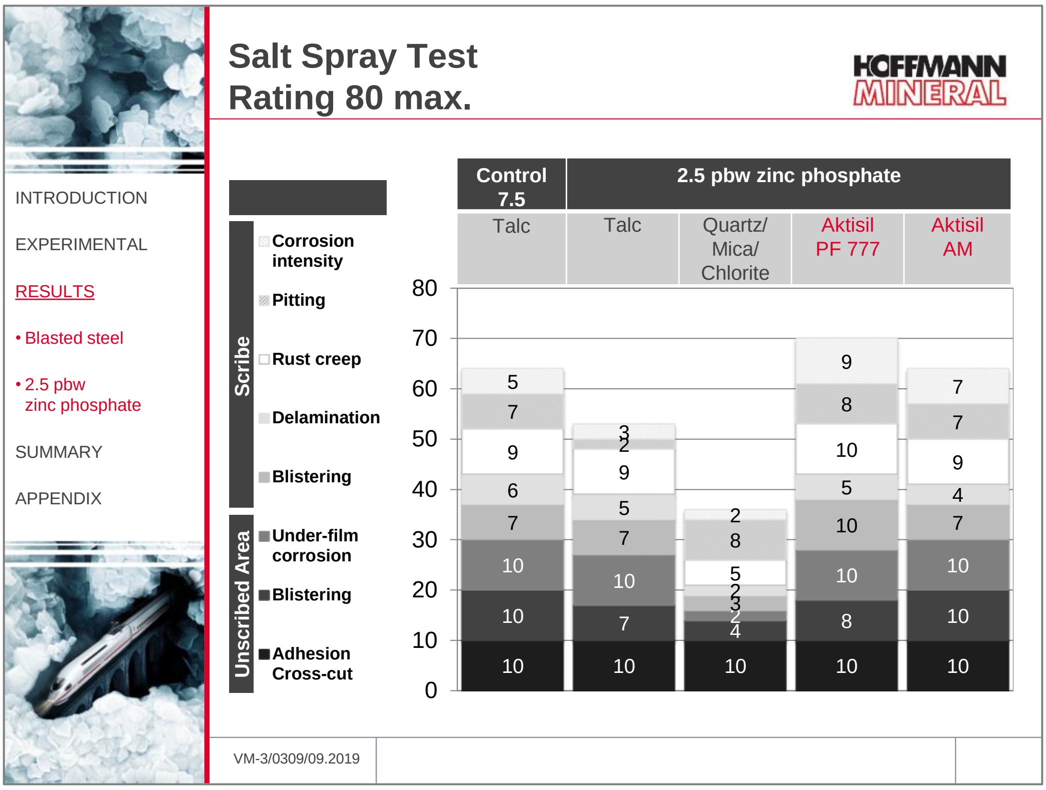

Fig. 14

Neuburg Siliceous Earth grades are able to ensure a markedly better property profile. Along with the very good results in the humidity test, when using Neuburg Siliceous Earth grades, over the whole the corrosion protection level of the control formulation can be attained, and even improved with Aktisil PF 777.

3.2.2 Without zinc phosphate

In order to verify the efficiency of Neuburg Siliceous Earth, tests were also run with totally replacing the anti-corrosion pigment by the filler.

Even without zinc phosphate, without exception very good adhesion on blasted steel and excellent resistance against condensation water are obtained.

Marked differences in the performance of the fillers are evident after the salt spray test. With talc or with the blend of quartz, mica and chlorite a heavy blister formation can be observed already in the undamaged surface (*Fig. 15*). The rust perforations in priority at the blisters confirm a shortage of active pigment, and lead to heavy corrosion of the substrate. While this damage intensely proceeds with the quartz/mica/chlorite mixture, talc shows still an intermediate stage with a move from local delamination to selectively active corrosion cells.

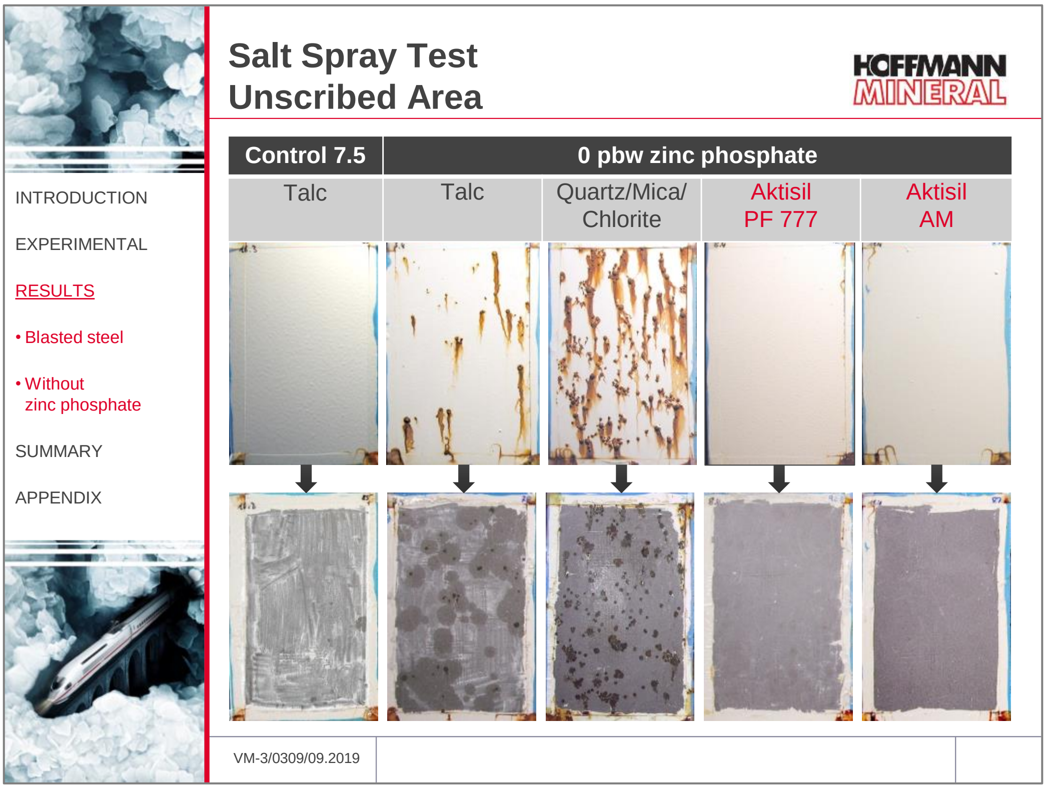

Fig. 15

By contrast, Aktisil PF 777 and Aktisil AM impart excellent corrosion protection. Apart from isolated blisters, both Aktisil grades deliver defect free results, and this in the coating film as well as below in a corrosion free steel surface.

Such a result largely matches the performance of the control formulation with talc and full zinc phosphate concentration.

Total replacement of the zinc phosphate with filler increases the corrosion tendency at the scribe damage (*Fig. 16*). The effect is more or less pronounced, depending on the nature of the filler.

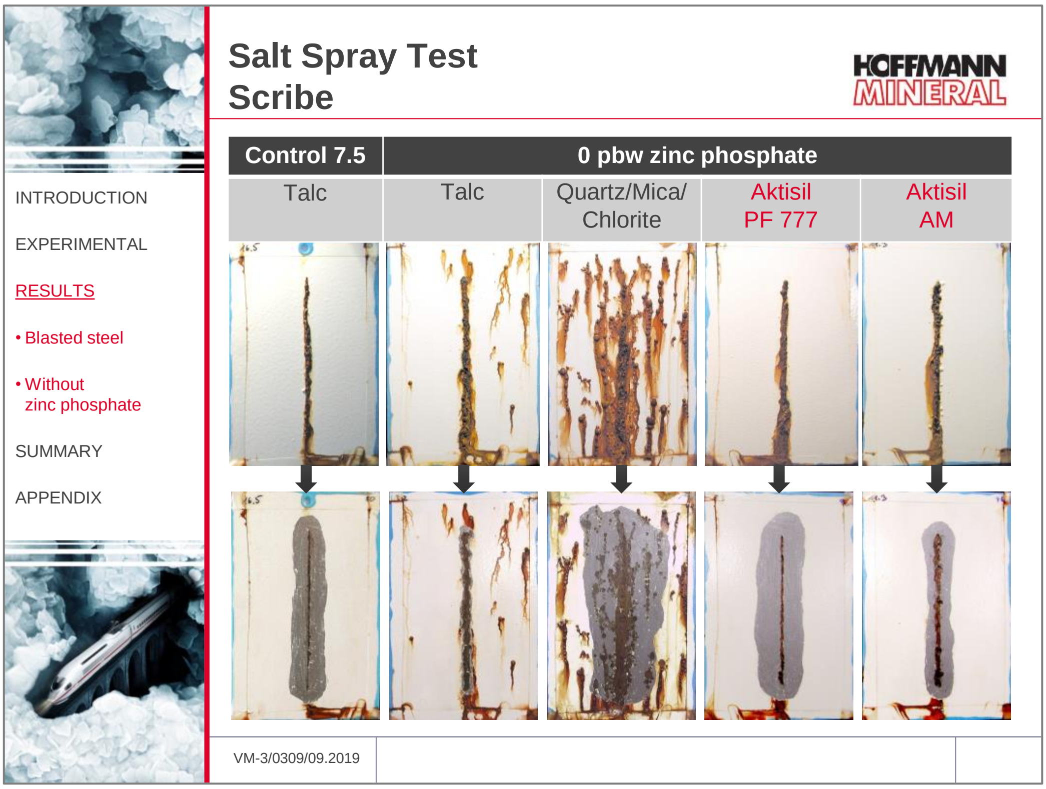

Fig. 16

A replacement with talc gives rise to markedly lower delamination along with overproportionally heavy corrosion at the scribe. With the blend of quartz, mica and chlorite, the performance is dramatically deteriorated. Both grades of Neuburg Siliceous Earth at first sight (upper line) seem to tend towards more pronounced rust formation at the scribe. But after stripping the coating, the effects of the different surface treatments become obvious: Aktisil PF 777 even without an active anti-corrosion pigment at the scribe shows less corrosion than the competitive fillers and thus by far the best result among the formulations without zinc phosphate. Aktisil AM with a lower trend towards delamination and heavier corrosion has to be ranked with regard to the effectiveness at the scribe between the two formulations with talc.

The adhesion strength of the coatings after the exposure comes out basically very good, but will be affected by increasing surface corrosion with the formulations containing talc or the quartz-mica-chlorite mixture.

The corresponding results are summarized in *Fig. 17*.

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		Control 7.5	0 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0/5	0/5	0	0
Blistering	Quantity Size	0 -	2 S3-4	3 S4	1 S2	1 S3
Under-film corrosion	[%]	0	2	15	0	0
Scribe						
Blistering	Quantity Size	1 S4	1 S4	4 S4	1 S4	4 S4
Delamination	[mm]	11	3	21	16	8
Rust creep	[mm]	0.5	2.0	5.5	0.7	1.7
Pitting max.	[mm]	0.3	0.4	0.4	0.5	0.4
Corrosion intensity	-	moderate	high	very high	moderate	moderate

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Fig. 17

The higher ratings in *Fig. 18* are an indication of the markedly improved properties of the formulations with Neuburg Siliceous Earth.

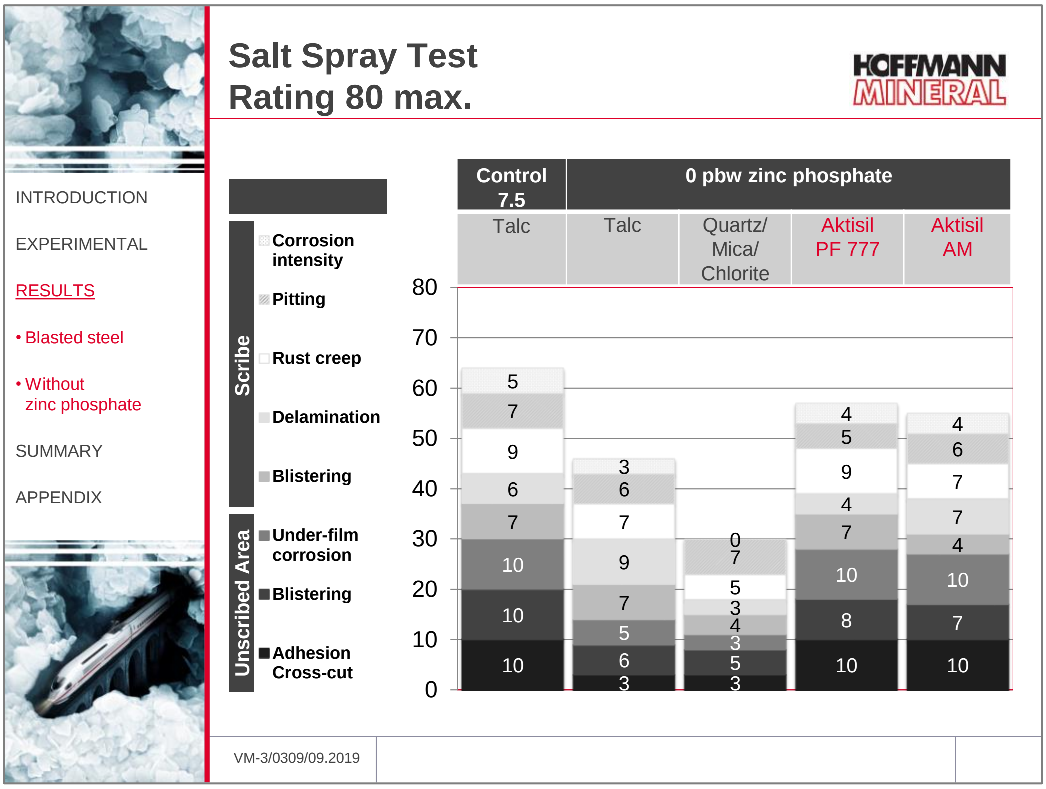


Fig. 18

3.3 Corrosion protection on non-blasted steel

3.3.1 Reduced addition of zinc phosphate

The second round of trials of the present work concerned the properties on non-blasted steel. This substrate because of its fairly smooth surface has proven very critical in comparison with the blasted steel. The anti-corrosion pigment again was partly replaced by fillers.

All formulations showed very good adhesion with cross-cut results of GT 0 to GT 1 (the Aktisil AM batch).

During the condensation water exposure a noticeable deterioration of the adhesion strength of all formulations with a reduced zinc phosphate addition is observed (*Fig. 19*). The formulations tend to develop blisters, in particular when the blend of quartz, mica and chlorite is used.

Just only Aktisil PF 777 offers a sufficient protection on the surface at the level of the control formulation without blistering and without under-film corrosion.

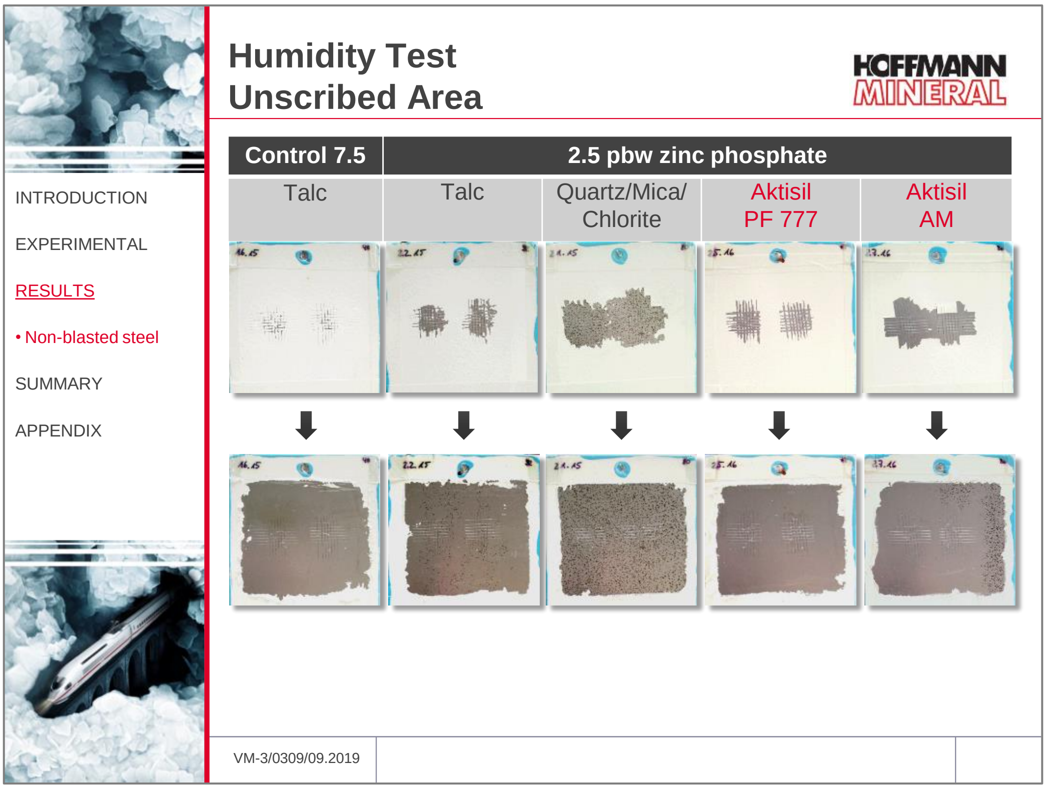


Fig. 19

The decrease of the adhesion strength vs. the control formulation is evident again at the assessment at the scribe (*Fig. 20*). Talc and Aktisil PF 777 give a comparable, just marginal worsening of the delamination. With the quartz/mica/chlorite mixture or with Aktisil AM the coating can be completely removed from the substrate. The formation of corrosion products at the scribe damage in total can be judged as minimal.

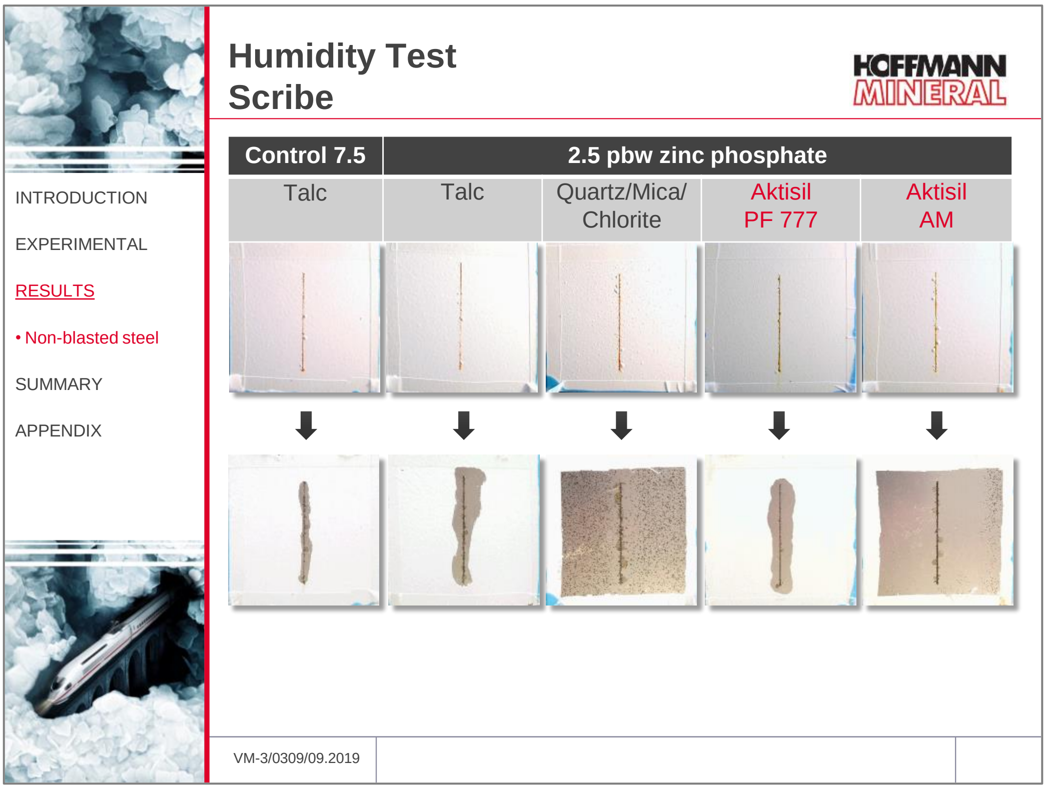
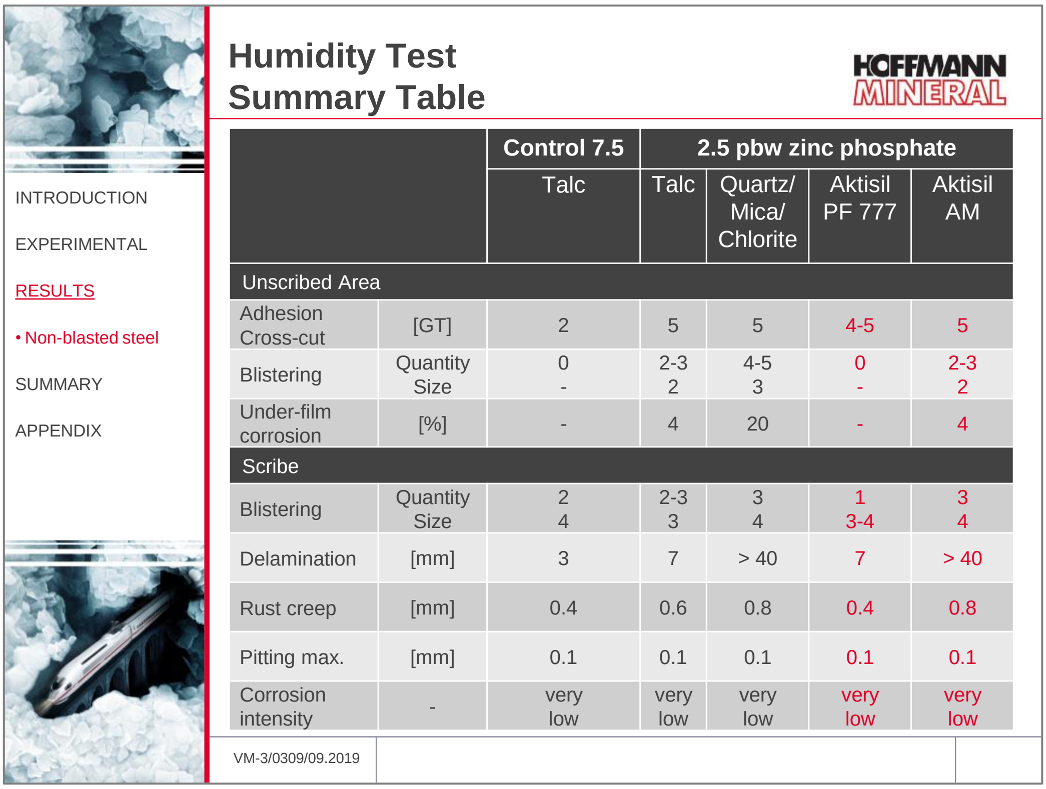


Fig. 20

The results and the assessment of the condensation water resistance can be found in *Fig. 21 and Fig. 22.*



Humidity Test Summary Table

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		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	2	5	5	4-5	5
Blistering	Quantity Size	0 -	2-3 2	4-5 3	0 -	2-3 2
Under-film corrosion	[%]	-	4	20	-	4
Scribe						
Blistering	Quantity Size	2 4	2-3 3	3 4	1 3-4	3 4
Delamination	[mm]	3	7	> 40	7	> 40
Rust creep	[mm]	0.4	0.6	0.8	0.4	0.8
Pitting max.	[mm]	0.1	0.1	0.1	0.1	0.1
Corrosion intensity	-	very low	very low	very low	very low	very low

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Fig. 21

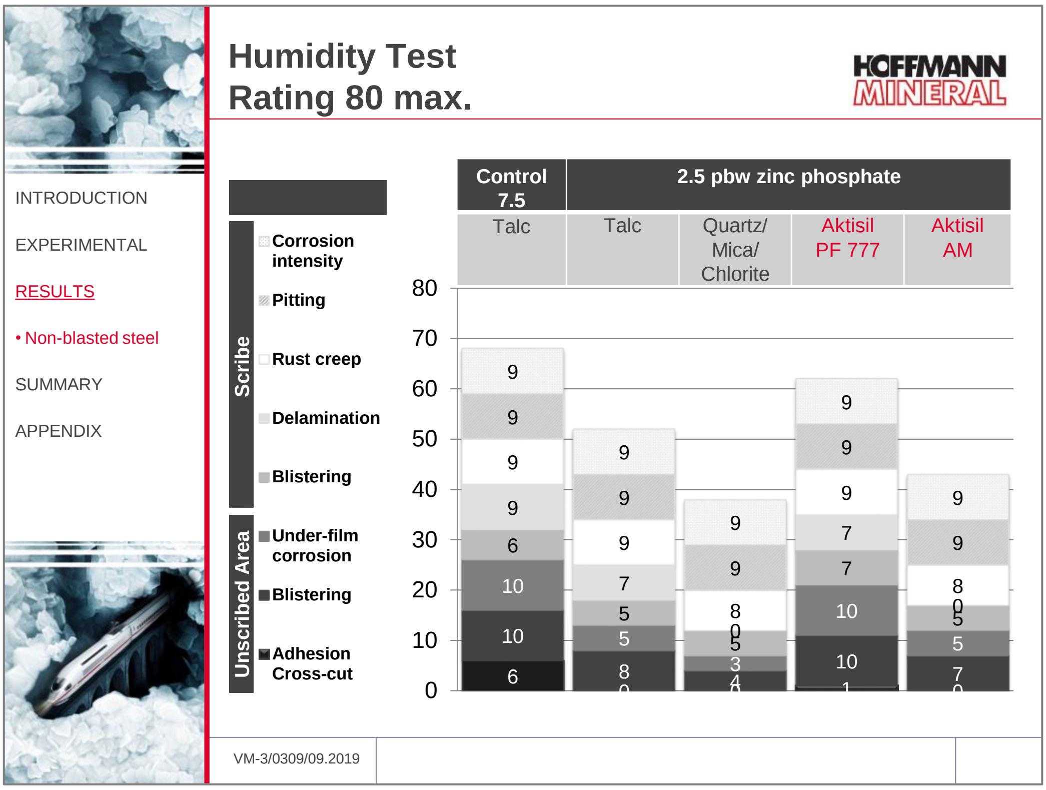

Fig. 22

The reduction of the zinc phosphate addition affects also the protection properties of the undamaged surface after exposure to the salt spray test.

Although no detectable blistering is seen in the coating with talc, the cross-cut test brings out the effects of point-by-point weaknesses in the coating film (*Fig. 23*). Compared to blasted steel, the effect concerns a larger area, and through the complete loss of adhesion very early decreases the mechanical load-bearing capacity of the coating.

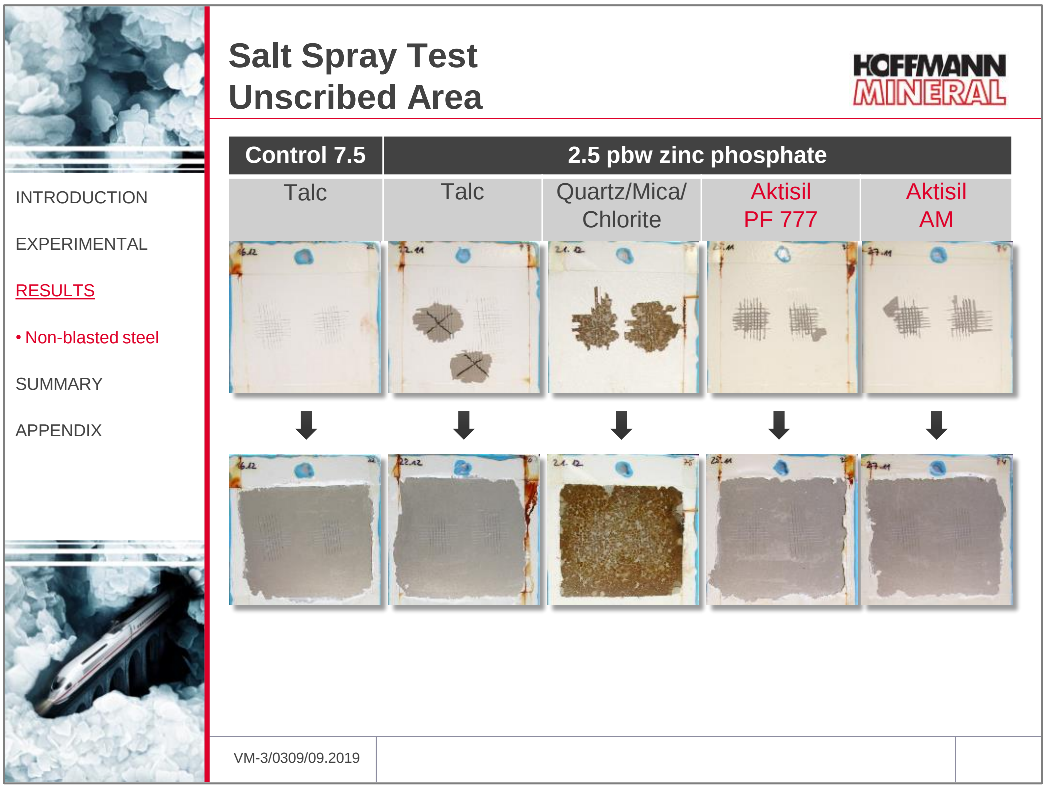

Fig. 23

The mixture of quartz, mica and chlorite leads to heavy blister formation, poor adhesion and heavy corrosion of the substrate. Formulations containing Neuburg Siliceous Earth like the versions with talc in the surface are free from blisters and corrosion. Compared with the control formulation, the adhesion of the coating is somewhat reduced, the direct comparison with the formulation with talc and decreased zinc phosphate content, however, shows a visibly higher resistance against adhesion loss at the surface.

The partial replacement of the anti-corrosion pigment with talc or with the mixture of quartz, mica and chlorite gives rise to heavier blistering in the scribe damage area. Of more advantage are here the Neuburg Siliceous Earth grades (*Fig. 24*). Especially Aktisil PF 777 helps not only to avoid blistering at the scribe, but also the intensity of corrosion is noticeably reduced.

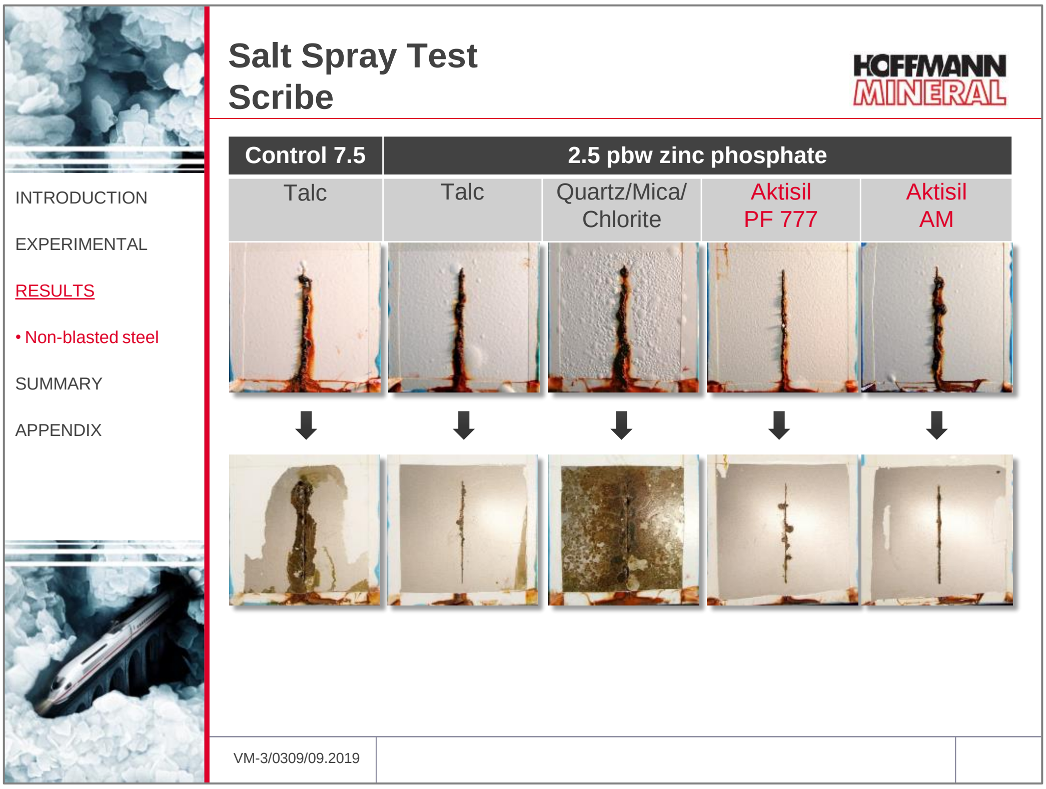

Fig. 24

With the exception of the control formulation and the batch with Aktisil AM, the coatings can be mechanically removed easily over the whole surface up to the edge of the substrate. The pronounced heavy corrosion tendency of the control formulation at the scribe can be reduced by using a lower concentration of anti-corrosion pigment in combination with talc or with Neuburg Siliceous Earth grades.

The formulation containing the natural mixture of quartz, mica and chlorite has obviously negative effects and consequently comes off in the overall rating (*Fig. 25 and Fig. 26*) at the lower end.

Salt Spray Test
Summary Table

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		Control 7.5	2.5 pbw zinc phosphate			
		Talc	Talc	Quartz/ Mica/ Chlorite	Aktisil PF 777	Aktisil AM
Unscribed Area						
Adhesion Cross-cut	[GT]	0	0/5	5	4	4
Blistering	Quantity Size	0 -	0 -	5 3-4	0 -	0 -
Under-film corrosion	[%]	0	0	70	0	0
Scribe						
Blistering	Quantity Size	2 2	3 5	5 5	0 -	1 3
Delamination	[mm]	25	> 40	> 40	> 40	30
Rust creep	[mm]	4	0.5	21	1	0.7
Pitting max.	[mm]	0.4	0.5	0.4	0.3	0.4
Corrosion intensity	-	high	high	high	moderate	high

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Fig. 25

Salt Spray Test
Rating 80 max.

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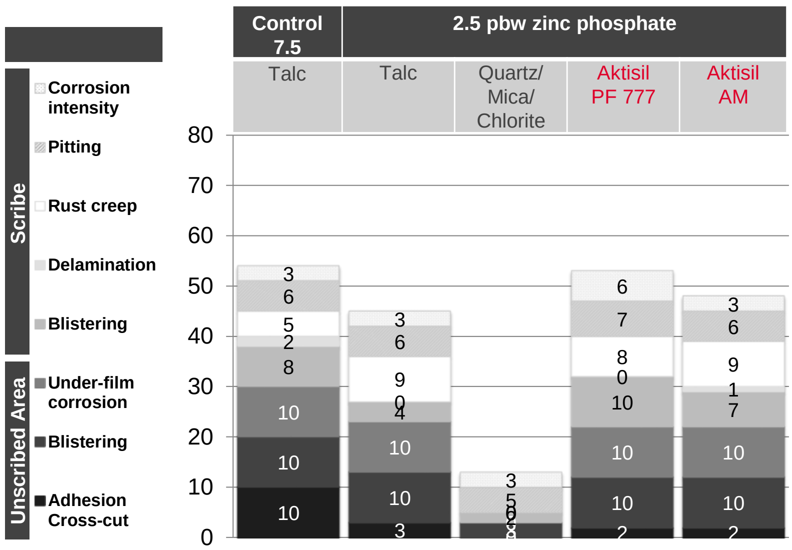

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Fig. 26

Compared with the results on blasted steel, over the whole a markedly poorer performance of the formulations becomes evident on non-blasted steel. This performance loss is clearly illustrated in *Fig. 27 and Fig. 28.* For the control formulation, the change to the non-blasted substrate gives rise to a markedly poorer corrosion protection at the scribe. The formulations with reduced zinc phosphate addition show losses in the adhesion strength as the most prominent effect.

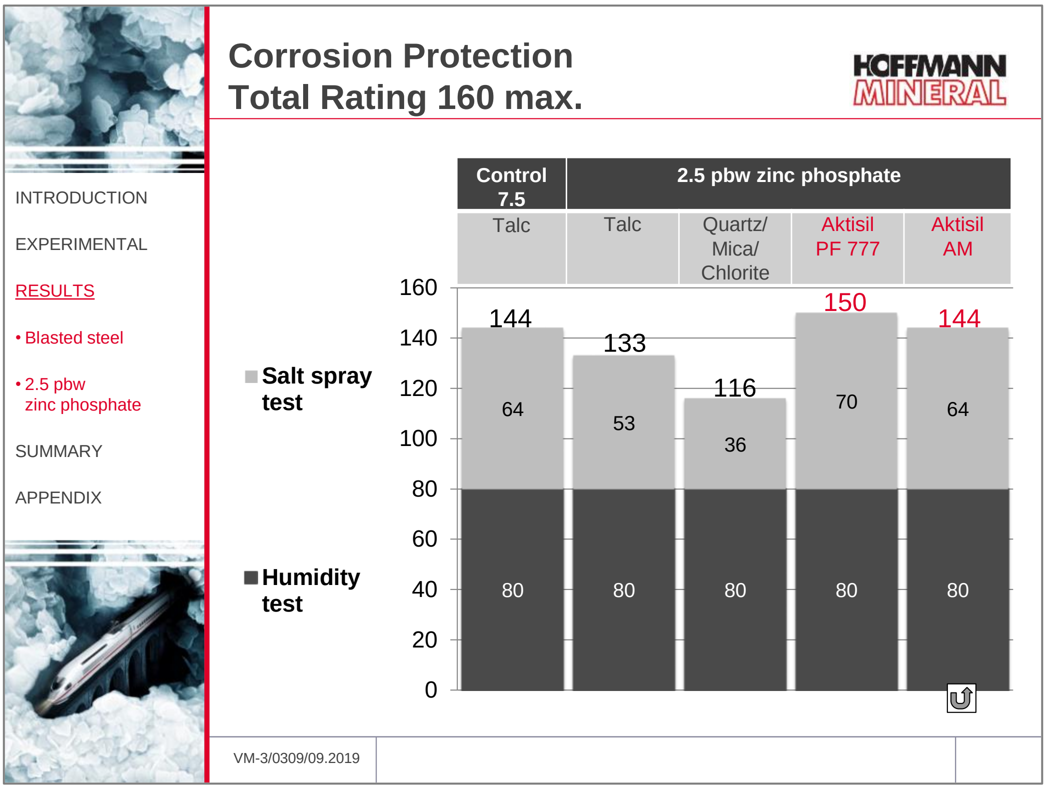


Fig. 27 blasted steel

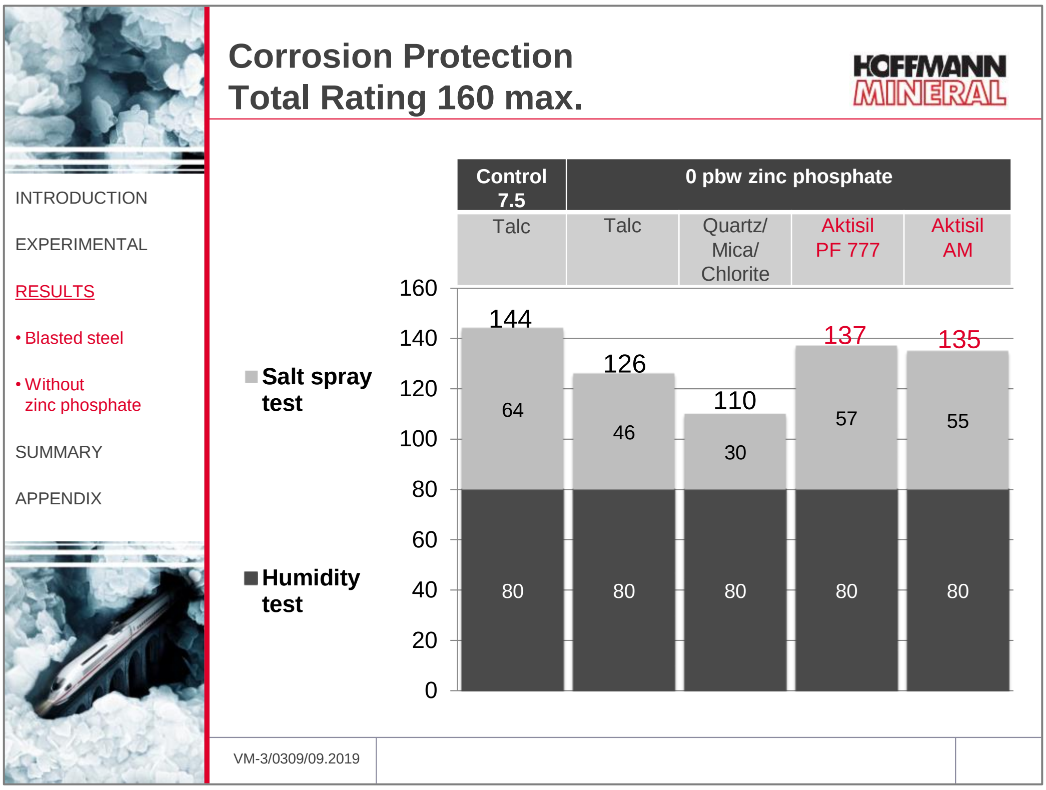


Fig. 28 non-blasted steel

3.3.2 Use of amino silane

The addition of an alkoxy silane with suitable functionality as an adhesion promoter can be very helpful on non-blasted steel. Upon addition of a small amount of amino silane to the hardener component, prior to and after the exposure test always an excellent adhesion is obtained.

The performance of the formulations with amino silane surpasses the satisfactory level of the control in the humidity test, as shown in *Fig. 29 and Fig. 30.* Blistering and surface corrosion remain absent, delamination processes at the scribe are practically undetectable.

The natural filler mixture of quartz, mica and chlorite was not included in this series, as its overall performance in the earlier studies had come out unsatisfactory.

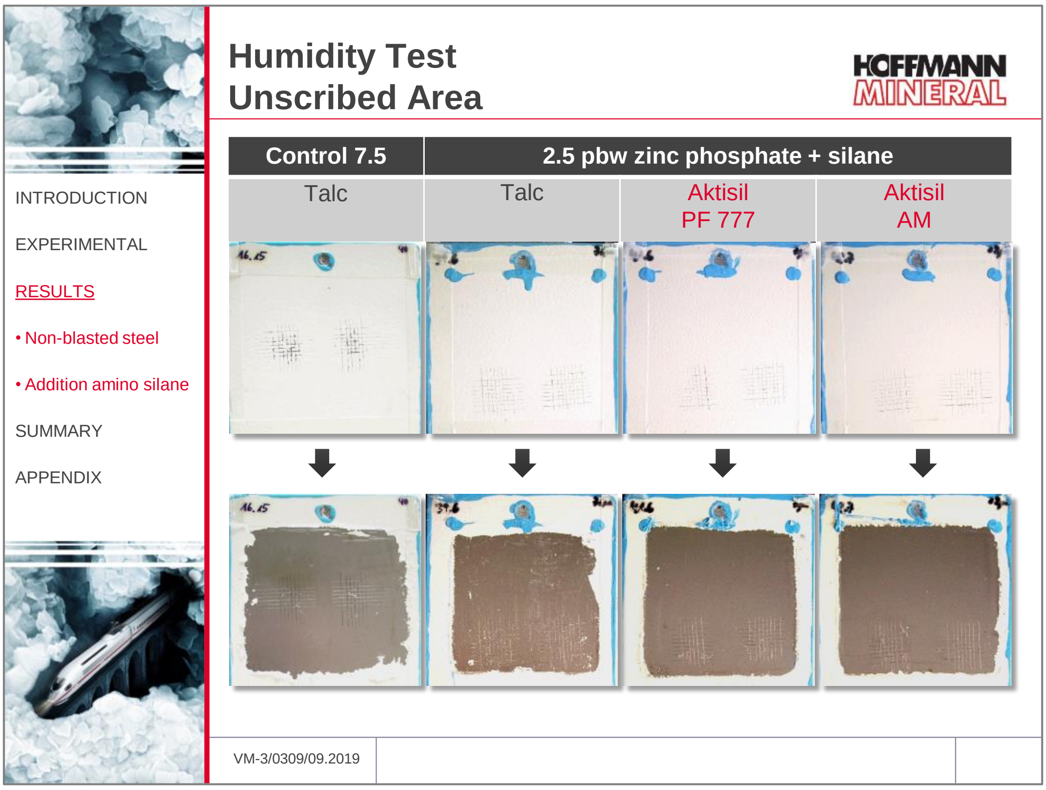
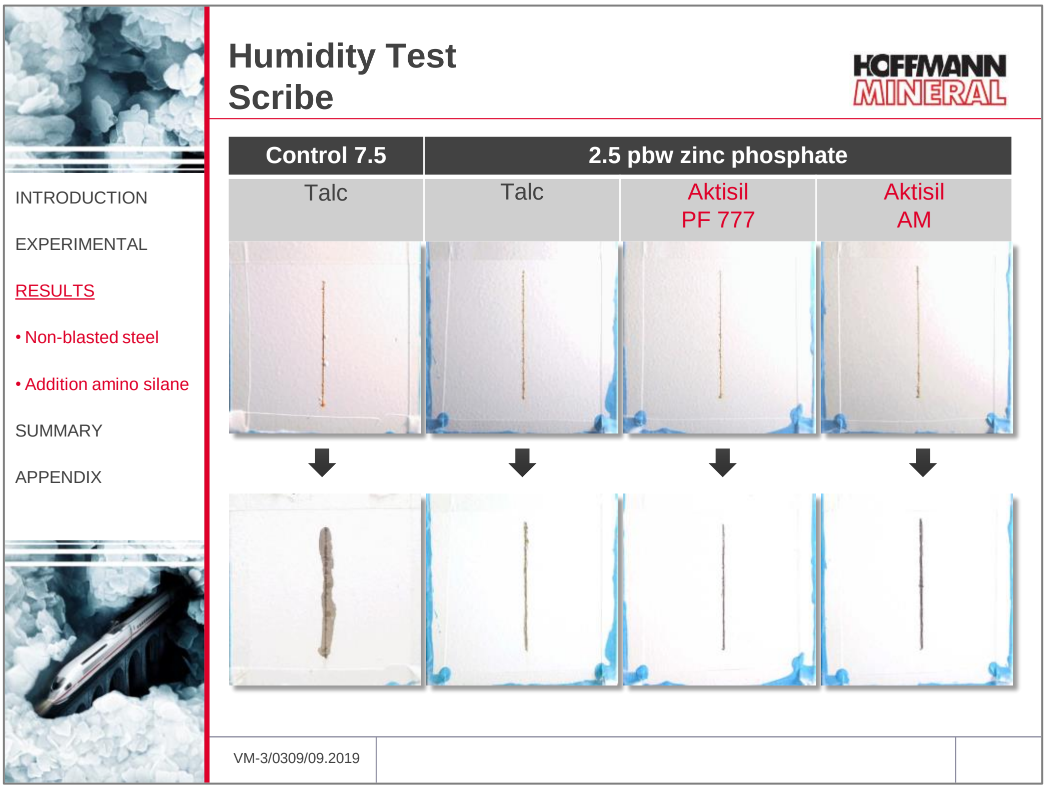


Fig. 29


Humidity Test
Scribe
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Control 7.5
2.5 pbw zinc phosphate
Talc
Talc
Aktisil
PF 777
Aktisil
AM
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Fig. 30

Fig. 31 and Fig. 32 summarize the pertinent data and the assessment profile.

Humidity Test Summary Table

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		Control 7.5	2.5 pbw zinc phosphate + silane		
		Talc	Talc	Aktisil PF 777	Aktisil AM
Unscribed Area					
Adhesion Cross-cut	[GT]	2	0	0	0
Blistering	Quantity Size	0 -	0 -	0 -	0 -
Under-film corrosion	[%]	0	0	0	0
Scribe					
Blistering	Quantity Size	2 4	1 3	0 -	1 2
Delamination	[mm]	3	0.5	0.5	0.8
Rust creep	[mm]	0.4	0.1	0.1	0.4
Pitting max.	[mm]	0.1	0.1	0.1	0.1
Corrosion intensity	-	very low	very low	very low	very low

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Fig. 31

Humidity Test Rating 80 max.

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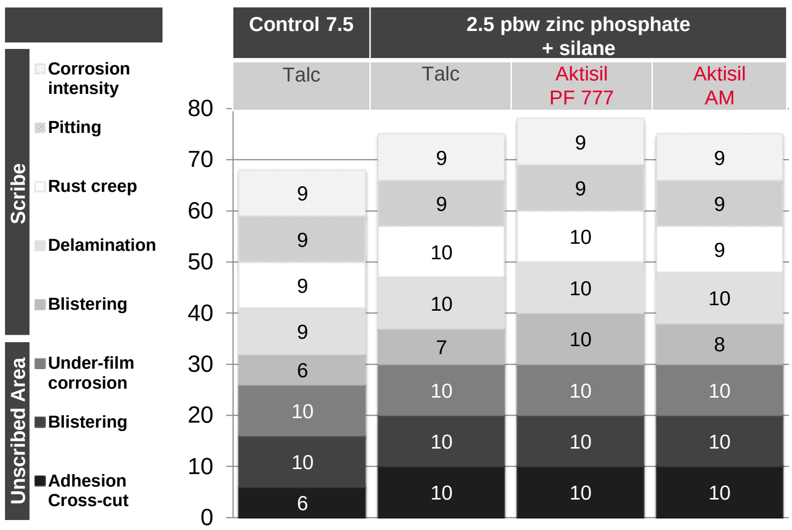

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Fig. 32

The salt spray test with talc leads to isolated larger and flat blisters in the surface (*Fig. 33*). Over longer test periods, they become the starting point of local substrate corrosion plus the formation of multiple smaller blisters in their direct neighborhood.

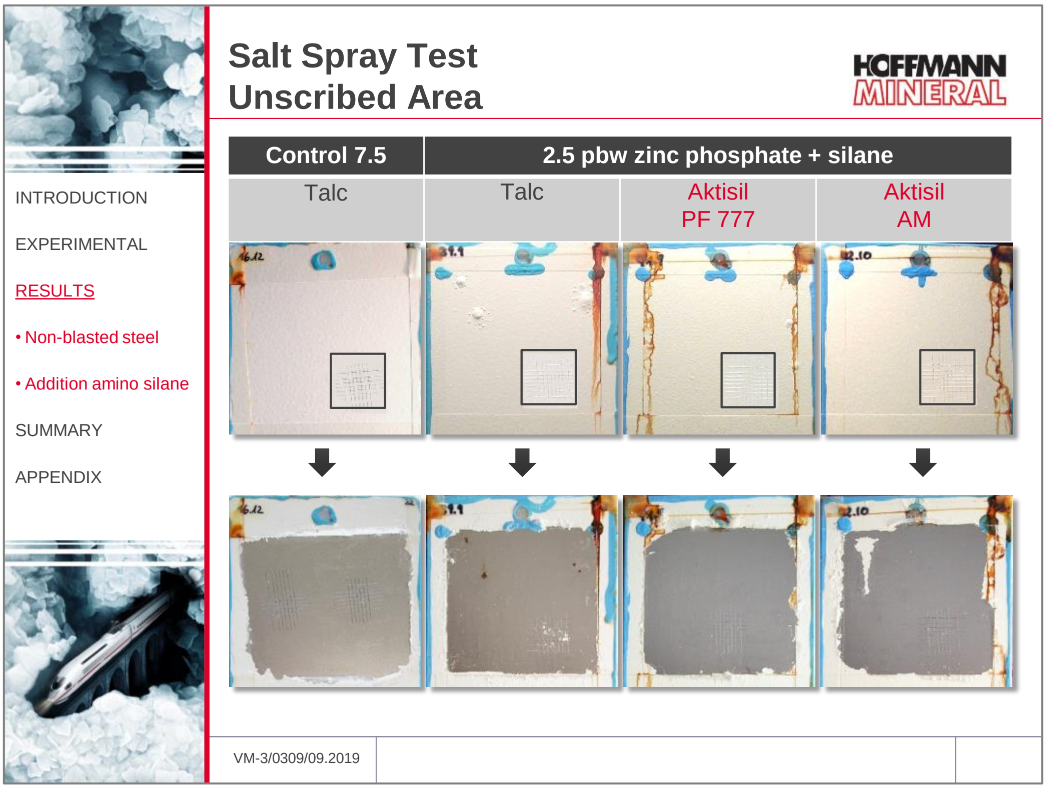


Fig. 33

The use of Neuburg Siliceous Earth helps to avoid the substrate corrosion. Blisters are located only very isolated and with smaller size compared to the talc formulation. Aktisil AM here gives the best results.
The basic effects of the adhesion promoter, similar to the surface, can also be derived at the scribe via markedly less delamination (*Fig. 34*).

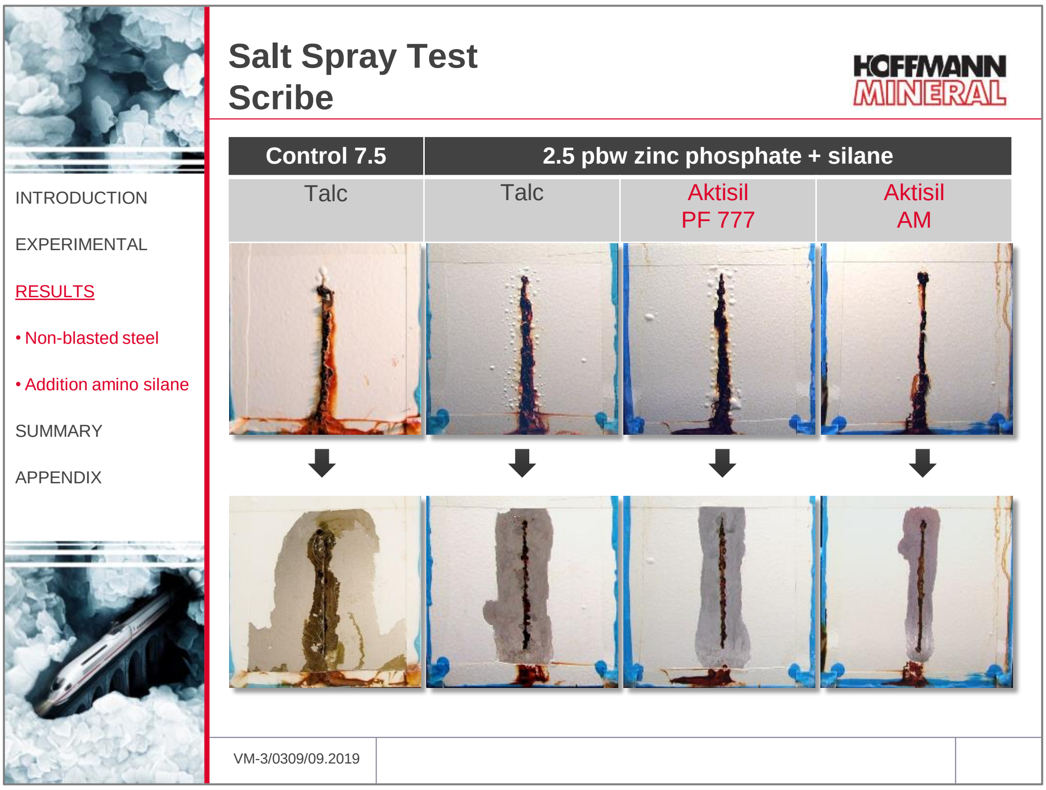
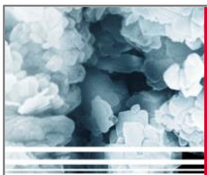


Fig. 34

While compared with talc, Aktisil PF 777 offers a means to reduce the delamination at the scribe, Aktisil AM in combination with aminosilane offers particular benefits. Blistering and degree of corrosion at the scribe come out visibly lower, and the area of delamination is much further reduced in size. *Fig. 35 and Fig. 36* list the individual test results as well as the overall performance rating.



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Salt Spray Test Summary Table

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		Control 7.5	2.5 pbw zinc phosphate + silane		
		Talc	Talc	Aktisil PF 777	Aktisil AM
Unscribed Area					
Adhesion Cross-cut	[GT]	0	0	0	0
Blistering	Quantity Size	0 -	1-2 5	1 4	0-1 2-3
Under-film corrosion	[%]	0	0.2	0	0
Scribe					
Blistering	Quantity Size	2 2	4 4	3 4	1 3
Delamination	[mm]	25	15	12	8
Rust creep	[mm]	4	1.3	1.3	1.3
Pitting max.	[mm]	0.4	0.5	0.3	0.3
Corrosion intensity	-	high	high	high	moderate

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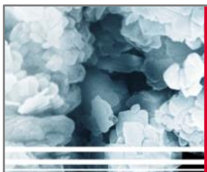
Fig. 35



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Salt Spray Test Rating 80 max.

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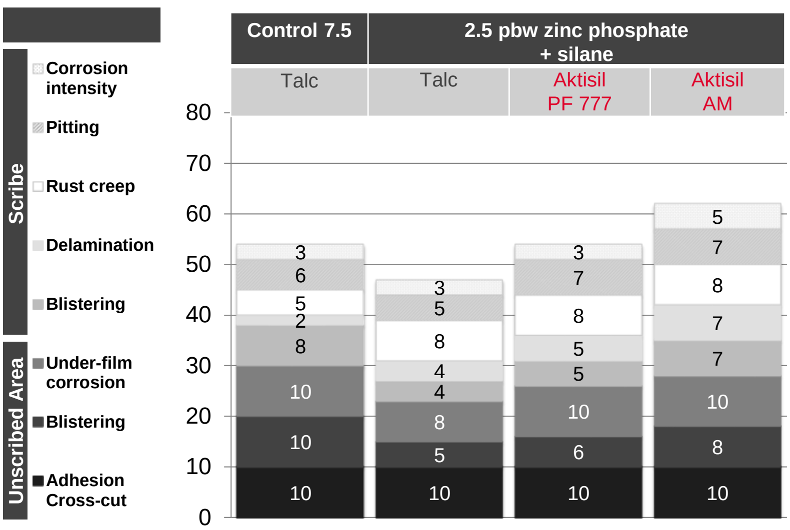

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Fig. 36

The example of the formulation with talc confirms that the addition of an adhesion promoter not necessarily exerts positive effects on all test properties. Increased blister formation and corrosion in the surface may go together with increased adhesion strength. The rather poor performance in the salt spray test, according to *Fig. 37*, in the total assessment only allows reaching a rating similar to the control formulation.

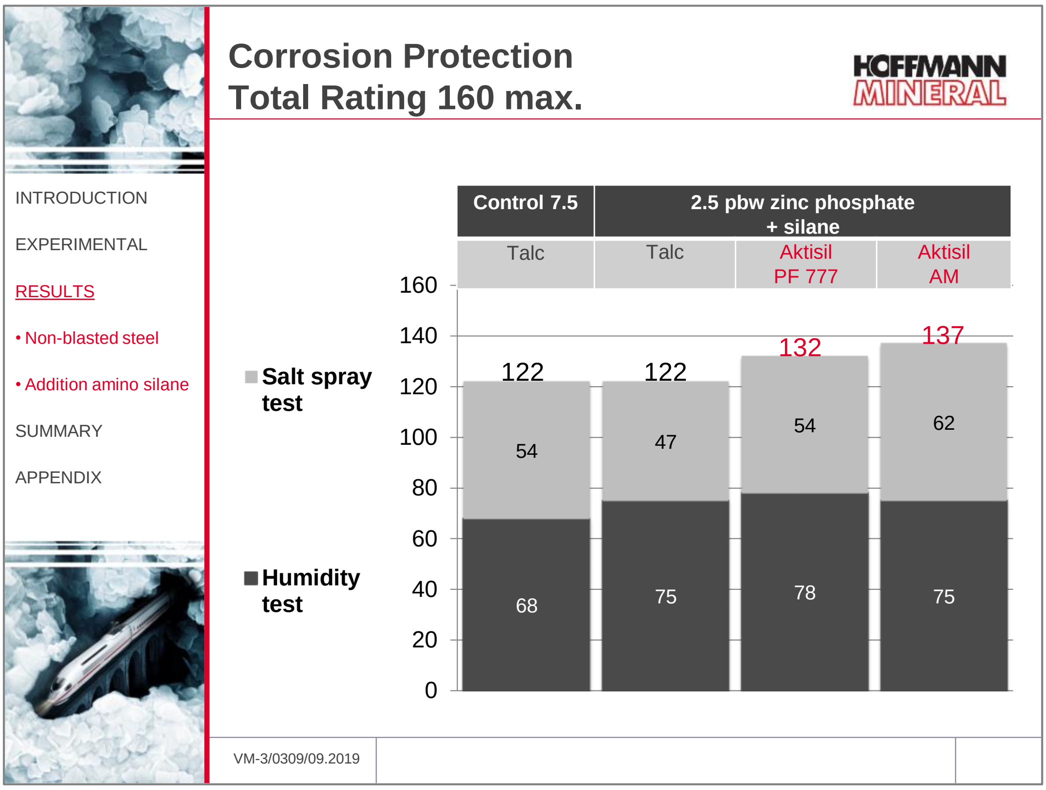


Fig. 37

Useful benefits come out only in the formulations with the Neuburg Siliceous Earth grades. Filler combinations including Aktisil PF 777 or preferably Aktisil AM offer the desired performance improvement beyond the level of the control formulation. Even with reduced zinc phosphate content an outstanding corrosion protection on adhesion critical substrates becomes possible.

For optimum protection efficiency, the concentration of the adhesion promoter has to be adjusted for each coating; for the formulation in question, the optimum can be expected at slightly less than 1 %.

In further tests, the positive effects of Neuburg Siliceous Earth grades on non-blasted steel could be confirmed also in formulations without anti-corrosion pigment. Compared with the competitive fillers, Aktisil PF 777 and Aktisil AM impart outstanding protection in the surface. For applications on non-blasted steel, total elimination of zinc phosphate, however, does not appear practical, as after a mechanical damage the corrosion processes progress overproportionally faster and thus reduce the performance of the coating system.

4 Summary

The reduced protection efficiency caused by a decreased zinc phosphate addition in the coating system tested cannot be compensated by fillers such as talc or the blend of quartz, mica and chlorite.

- on blasted steel the performance loss primarily shown in the salt spray test via heavier corrosion intensity at the scribe and an early loss of protection in the surface.
- on non-blasted steel poorer adhesion must be considered as the cause of poorer corrosion protection properties under exposure to condensation water (humidity test) as well as under strong ionic exposure (salt spray test).

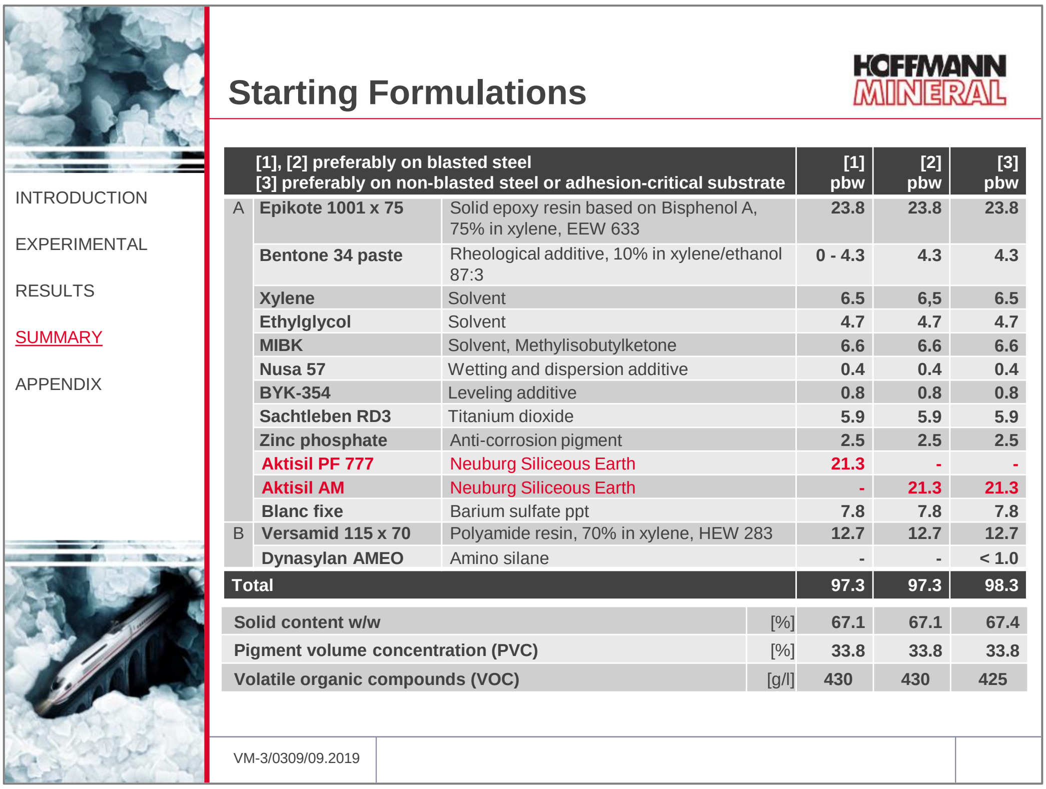
Only surface modified fillers such as Aktisil PF 777 and Aktisil AM offer:

- high anti-corrosion efficiency already in single-layer systems
  - » on blasted steel with zinc phosphate reduced or totally eliminated
  - » on non-blasted steel in combination with aminosilane and a reduced concentration of zinc phosphate
  - » based on
    - excellent adhesion
    - excellent condensation water resistance
    - outstanding resistance against salt spray
- better performance compared with talc or a natural mixture of quartz, mica and chlorite
- along with a small portion of zinc phosphate comparable performance with commercial standard systems containing conventional fillers and a high loading of zinc phosphate

In particular, the following properties of Neuburg Siliceous Earth are of interest:

- Aktisil PF 777
  - » hydrophobic
  - » rheologically active, suitable for thick film applications
  - » rapid development of high hardness
  - » very high protection efficiency against corrosion at the scribe on blasted steel
  - » very high protection efficiency against blistering and corrosion in the surface
- Aktisil AM
  - » very good leveling
  - » high protection efficiency against corrosion at the scribe on blasted steel
  - » high protection efficiency against blistering and corrosion in the surface
  - » in combination with aminosilane synergistic effect leading to excellent adhesion strength and excellent corrosion protection on non-blasted steel.

Suggested starting formulations with Neuburg Siliceous Earth are compiled in Fig. 38.



Starting Formulations

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	[1], [2] preferably on blasted steel [3] preferably on non-blasted steel or adhesion-critical substrate		[1] pbw	[2] pbw	[3] pbw
A	**Epikote 1001 x 75**	Solid epoxy resin based on Bisphenol A, 75% in xylene, EEW 633	23.8	23.8	23.8
	Bentone 34 paste	Rheological additive, 10% in xylene/ethanol 87:3	0 - 4.3	4.3	4.3
	Xylene	Solvent	6.5	6,5	6.5
	Ethylglycol	Solvent	4.7	4.7	4.7
	MIBK	Solvent, Methylisobutylketone	6.6	6.6	6.6
	Nusa 57	Wetting and dispersion additive	0.4	0.4	0.4
	BYK-354	Leveling additive	0.8	0.8	0.8
	Sachtleben RD3	Titanium dioxide	5.9	5.9	5.9
	Zinc phosphate	Anti-corrosion pigment	2.5	2.5	2.5
	Aktisil PF 777	Neuburg Siliceous Earth	21.3	-	-
	Aktisil AM	Neuburg Siliceous Earth	-	21.3	21.3
	Blanc fixe	Barium sulfate ppt	7.8	7.8	7.8
B	**Versamid 115 x 70**	Polyamide resin, 70% in xylene, HEW 283	12.7	12.7	12.7
	Dynasylan AMEO	Amino silane	-	-	< 1.0
Total			**97.3**	**97.3**	**98.3**

Solid content w/w	[%]	67.1	67.1	67.4
Pigment volume concentration (PVC)	[%]	33.8	33.8	33.8
Volatile organic compounds (VOC)	[g/l]	430	430	425

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Fig. 38

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