REEgain Milestone 3, TP3

Benchmark study of NdFeB magnets

REEgain Innovation Consortium (2012-2016)

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1 Introduction to report

The report contains a detailed review and benchmark of different available coatings against each other resulting in guidelines for the coating strategy relating to the operation conditions. The target of the review has primarily been to identify the corrosion resistance of the analyzed coatings.

Part of the project has been development of a new coating type for the permanent magnet segment. This report contains the result of this development work and the benchmark results for this coating.

Based on the outcome of this report a coating catalogue has been created stating selected values for the tested coating types. The aim of the catalogue is to provide the reader with a simple overview of the advantages and disadvantages of the tested coating types.

2 Sol-Gel Coating development

Organic-inorganic hybrid coatings prepared by sol-gel process

Sol-gel technology has in recent years revolutionized the possibilities for changing the surface properties of a large number of materials including almost all metals and alloys, glass, wood etc. This technology can be described as a method for the synthesis of glass ceramic like coatings from liquid reagents. Sol-gel refers to two subsequent process steps of first preparing a colloidal suspension (sol) starting from soluble precursors and then in a second step using the sol to prepare an immobile and cross-linked material (gel), see fig 1. In short the unique properties of the sol-gel coatings are due to a strongly connected inorganic ceramic structure combined with functional organic side chains. The sol-gel technology is based on polymerization of small inorganic molecules, typically silicon coordinated by alkoxy groups.

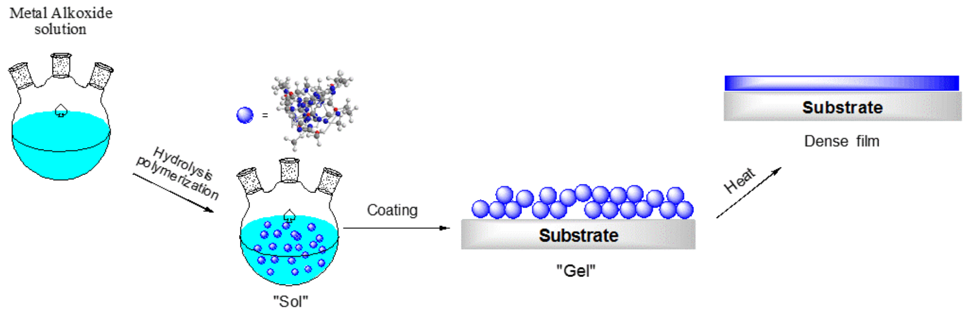

Figure 1:Overview of the coating synthesis procedure. Blue spheres; metal-oxo particles. After heating a dense coating is formed

In an early stage of the coating synthesis, the silanes molecules undergo hydrolysis and condensation, hence become interlinked to form metal-oxo polymer silica particles, the "sol". These particles constitute the building blocks in the subsequent application and curing, which provide a thin ceramic coating with a layer thickness between 5 and 15 μm. Within the last couple of years, research and development in organic functionalization of silicon alkoxides, have resulted in a large number of commercially available chemicals, which facilitates the introduction of a multitude of functional organic groups in the inorganic network. By including one or more of such organic modified metal alkoxide in the sol-gel formulation, it is possible to modify the physical, chemical, optical and mechanical properties of the coated surface. For example, coatings with very low organic content generally show excellent wear qualities but only a low tolerance against deformation. By the mere addition of organic content, the deformation tolerance may increase, but typically at the cost of wear qualities. However, with the inclusion of silanes with carefully designed functional side chains, both of the required qualities may be achieved.

The application process of sol-gel based coating is similar to other types of coatings, see fig 2.

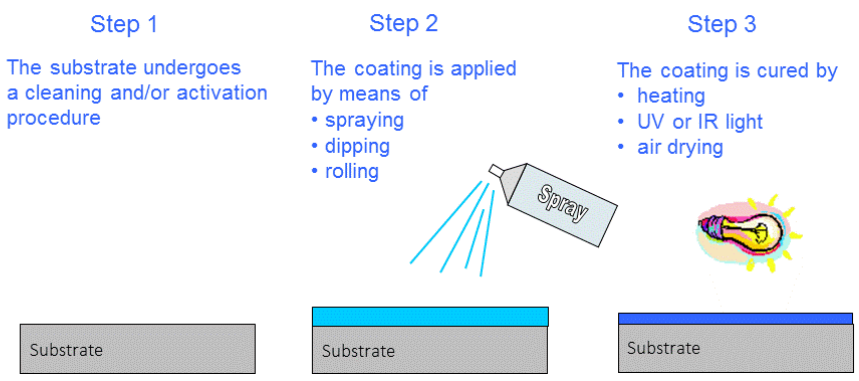

Figure 2: Overview of the coating process step by step

Typically, after cleaning with e.g. ethanol or acetone, the substrates are ready for coating. Some substrates need additional treatments to activate the substrate surface for better accommodating the coating. The coating can be applied by standard application methods like spraying, rolling and paintbrush, see step 2. The curing process is essential in many sol-gel based coating. Typically, these types of coatings are cured at elevated temperature. Sol-gel derived glass ceramic coatings do not, as is the case for conventional glass ceramics, require sintering at highly elevated temperatures. Instead, sol-gel coatings are typically cured at temperatures ranging from 140 °C to 200 °C for a period of one to two hours. If the required curing temperature and time is not achieved the performance of the final coating well be reduced or non-existent.

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The applied test coating

The neodymium magnets were coated with an organic-inorganic hybrid coating. This coating has demonstrated excellent repellent properties towards organic and inorganic deposits. The coating forms a 5µm thick and flexible coating, and have a relatively good mechanical stability. All the chemical coating components become either part of a cross-linked coating film or evaporate during curing. The inorganic part of the coating is constituted of a sol-gel derived siloxane (Si-O) network, forming a glass like structure. A large part of the backbone of the coating is thus constituted of a benign inorganic matrix. This inorganic matrix is stabilized through the inclusion of silicon-bound organic moieties, which provides essential properties, such as flexibility and chemical stability, to the coating. Furthermore, the coating includes a polydimethylsiloxane component to provide the coating with repellent surface characteristics. The coating on aluminum has passed the 1000 hours corrosion salt spray test ISO9227 under neutral conditions and the cross hatch adhesion test ISO2409 after 72 hours in demineralized water.

Application of the coating

Pre-treatment:

The test coating has not only showed good adhesion to **phosphatized** metal substrates but also an increased corrosion protection when used together with the **phosphatized** surfaces. The **phosphatized** neodymium magnets was first cleaned with ethanol and then acetone to remove surface oils and other residues.

Coating application:

To adjust the base coating for brush application, 100g of base coating was diluted with 28g of ethanol and 28g of methyl isobutyl ketone. To avoid contact points on the coated surface each magnet had to be coated in two rounds. All magnets were first coated on five sides leaving one side uncoated. After the first coating application the magnet was cured in an oven. After the magnets have cooled, the remaining uncoated sides of the magnets were coated and finally cured.

Curing procedure:

Before the coated magnets were heat treated they were left to flash off for 5 min., which allows excess solvent to evaporate. The coated magnets were cured for one hour at 200 °C. Due to localized reduced wetting of the surfaces, minor coating defects were observed on some magnets.

3 Benchmark coatings

The following coatings types have been examined:

1. NiCuNi -
2. Zn
3. CZn
4. Black Epoxy
5. Teflon -
6. Phosphate + Sol-gel
7. JL mag epoxy

The magnets 1-6 has been supplied by Technoflex, however the Sol-Gel coating was supplied and applied by The Danish Technological Institute.
The 7. Magnet (JL mag epoxy) was supplied by JL Mag Europe.

Coatings 1-5 and 7 are coatings that are commercially available. The JL mag coating is only supplied by JL mag, while versions the other coatings can be supplied by several suppliers.
Due to availability of samples and samples sizes the JL mag epoxy has not been tested in all tests.

3.1 Coating description

NiCuNi
The NiCuNi is as the name indicates a coating consisting of 3 layers, first and third layer consisting of nickel (Ni) and the middle layer consisting of copper (Cu).
The coating is an electro plated coating and can deposited as a high levelling coating, smoothing any surface roughness.
An advantage of the electroplated coatings is the fact that the coating can be deposited evenly on the complete surface of the sample. However as nickel is more noble than iron the effectiveness of this type of coating relies on the coating being 100% pore and pinhole free
As the upper layer consists of nickel there is a risk of release of nickel from the surface and therefor also a risk of nickel allergy when in contact with this coating.

Zn & CZn
As the NiCuNi coating the Zn coatings are electroplated coatings.
An advantage of the electroplated coatings is the fact that the coating can be deposited evenly on the complete surface of the sample.
As zinc is less noble than iron it will act as a sacrificial anode if the coating is damaged and still offer protection of the underlying iron.

Epoxy
Epoxy is a chemically curing paint, which means that the curing process is irreversible, and these types of paint are commonly known for their good water, chemical and heat resistance.

Teflon
Teflon is mainly known by its “non-stick” properties, but also exhibits excellent chemical resistance properties.
The coating cures by heating, it is possible to apply more than on coat, however careful attention must be put to prober baking of the intermediate layers.

Sol-Gel
The nature and properties of the Sol-Gel coating is addressed in section 2, Sol-Gel coating development.

JL Mag
The precise nature of the JL Mag coating is not known.

4 Procedure

A wide verity of tests may be applied to access mechanical strength and corrosion resistance of coatings.
The report “REEgain Milestone 1, TP3: Development of test protocol for magnet system functional testing”, has provided the project with a background for choosing the following test:

- Fluids exposure (Immersion in Water and Oil)
- Corrosion analysis (Salt Spray Test)
- Adhesive test (Pull-Off Adhesion Test)
- Microstructure examination (Light Optical Microscopy, LOM)
- Layer analysis (Scanning Electron Microscopy, SEM)

5 Results

5.1 Immersion tests

The immersion test is a simple test, where the magnets are left in a beaker and immersed in either tap water or oil over a given period and the interaction between the media and the magnet is then registered.

The water immersion tests ran for approximately 2200hours
The oil, which has been used, is an engine oil (Classicway 10W-40, Statoil, 4.5-5 pH).
The JL mag epoxy has not been tested in immersion testing.
The oil immersion test ran for approximately 2100h.

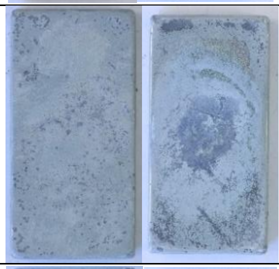
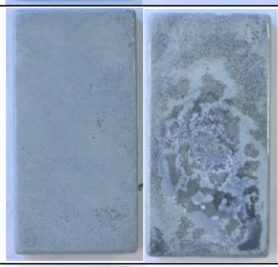
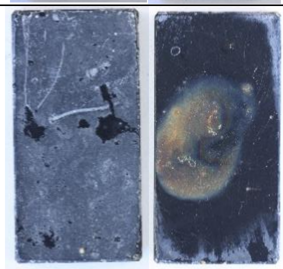
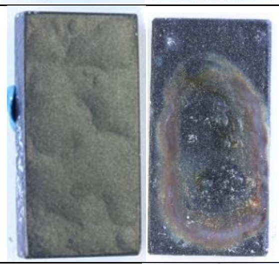
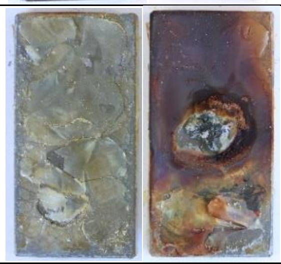
	After 7 days water immersion	After water immersion Front - Back	After oil immersion
NiCuNi			
Zn			
CZn			
Black Epoxy			
Teflon			
Sol-gel			

Table 1: Immersion test

NiCuNi, Zn and CZn do not show any corrosion after being exposed to water for roughly four months. They all lost their shine, which must be due to an oxidised layer forming on top of the coating. The back of the magnets, which faces the beaker, changed the surface as well, but not as homogeneously as the front and no sign of corrosion effects can be spotted either.
The Black Epoxy-coating changed its front, but did not show any signs of corrosion. Since epoxy is a polymer, it cannot corrode or form an oxidised film as metals, hence the changes might be caused by the specific epoxy used in the coating, and its lack of being waterproof – epoxies are often not favoured for long-term submersion in water. The back did not change the surface shine, but starting corrosion have been spotted from the centre.
The Teflon-coating's front is swollen due to uptake of water, the back of the magnet got small blisters and shows a starting corrosion.
The Sol-gel-coating showed same trends as the Teflon-coating, since the coating swallow water and breaks as well as starting corrosion in the magnet.

None of the samples exposed to immersion in oil showed any degradation of the coating after an exposure of approximately 2100hours.

5.2 Light Optical Microscopy(LOM)

LOM is used to examine the coating thickness and the connection between coating and the magnet. The magnets are moulded into an acrylic resin, and polished in order to handle it. The layer thickness of all coatings are measured with a software called analysis.
The NiCuNi-coating consist of three layers, as seen in figure 3, whereas the others are only one layer, hence 1st layer for NiCuNi is the Ni-layer closest to the magnet.

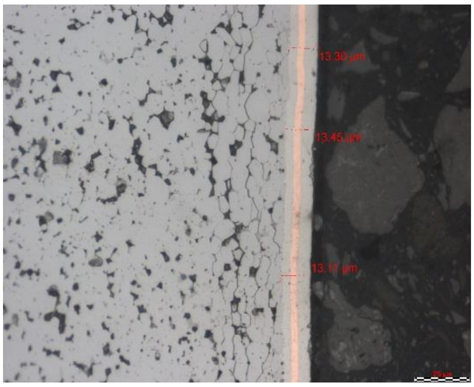


Figure 3: LOM picture of the surface of the Ni-CuNi coating

	Avg. coating thickness [µm]	Avg. corner thickness [µm]
NiCuNi	12.95±0.45 (4.03;2.95;5.91)	22.29±1.24
Zn	43.07±0.66	16.26±1.42
CZn	66.46±1.97	16.60±0.87
Black Epoxy	14.13±0.44	20.32±1.01
Teflon	20.04±0.41	21.37±1.79
Sol-gel	9,24±0,93	0
JL mag	28,19±3,7	38±5,08

Table 2 – coating thickness measured at the surface and at the corner via LOM.

Total layer thicknesses are given with a standard deviation and the results are based on the measurements seen in appendix 1.
The coatings are added in different thicknesses, but the most important thing is to keep an eye on the variation in thickness on the corners compared to the flat surfaces. It might cause problems for Sol-gel, Zn and CZn where the corner thickness is much thinner than the flat surface if these are used in services, where the coating works as protection against forces or harsh environments. It is however interesting that the JL mag samples exhibit higher coating thickness on corners than on sides.

5.3 Salt Spray Test

Salt spray testing is developed to measure the resistance of a coating in neutral salt environment. The degree of rusting is evaluated according to ISO 4628-3:2003, and the grading can be found in table 2. Few exceptions have been made according to the standard, in order to give a more detailed examination of the samples, data can be found in appendix 2.

Degree of rusting	Rusted area %
Ri 0	0
Ri 1	0.05
Ri 2	0.5
Ri 3	1
Ri 4	8
Ri 5	40-50

Table 3: ISO 4629 :2003 Marking scale

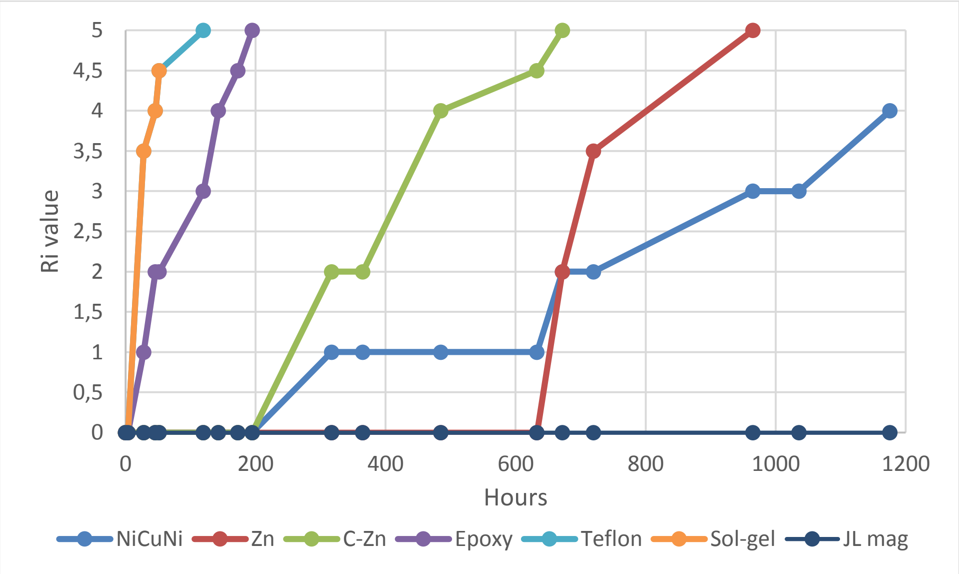


Figure 4 – Salt spray test of the samples graded according to ISO 4628-3:2003.

Epoxy-, Teflon- and Sol-gel-coating did not show great resistances and rusted within 200 hours under this environment. NiCuNi-coating shows a good resistance against corrosion, and even when it starts to corrode it does so slowly. Zn-coating also exhibits good resistance against the environment, roughly 650 hours before rust was observed. However, when the rusting begins, it starts to accelerate. The JL mag epoxy samples exhibited excellent resistance in saltspray. Hence for services where no rust at all can be tolerated, the JL mag epoxy could be a good solution. However also the Zn coating showed good resistance, the NiCuNi-coating should be picked where small rust spots can be tolerated in order to benefit from a longer service time. Keeping in mind that Ri 3 corresponds to 1% of the area is rusted after 1000 hours.

Figure 5 – Salt Spray Test

5.4 Pull-Off Adhesion Test

The Pull-Off Test is done in order to give a measurement of how well the coating adheres to the magnet. The glue, which has been used, is a two-component epoxy glue and it is cured at 70 °C for 45 min.
Due to sample size Pull-Off test was not performed on the JL mag epoxy samples

Figure 6 - Detachment between coating and magnet.

	1st [MPa]	2nd [MPa]	3rd [MPa]	4th [MPa]	Result [MPa]
NiCuNi	20.84	8.88*	19.16	17.40	19.1±1.7
Zn	19.02	22.25	18.80	13.67	18.4±3.5
CZn	23.22	17.48	21.34	14.58	19.2±3.9
Black Epoxy	22.39	18.34	19.02	24.07	21.0±2.7
Teflon	17.59*	18.20*	14.34*	16.65*	>16.7±1.7
Solgel	15.38*	N/A	10.78*	2.37*	>9.5±6.6

*Table 4 - Pull-Off Adhesion results. *the detachment is between coating and glue.*

Detachment occurs between the coating and the magnet, if nothing else is stated. This gives a measurement of how-well the coating adheres. For coatings like Teflon, it is only possible to conclude, that the coating adheres better to the surface than the connection between glue and coating.

5.5 *Scanning Electron Microscopy*

SEM is used to examine the structure of the coating so inhomogeneous parts or defects may be revealed together with a composition analysis. The samples used, are the same moulded pieces as used in LOM and they are looked upon at 2000x, 5000x, 10000x and 15000x zoom. Furthermore, an energy dispersive x-ray spectroscopy (EDX) mapping is performed at 10000x and an EDX mapping and point analysis is done at 15000x zoom.The SEM mapping technique is used to identify layers in the coating not visible by just SEM picture. Mapping is also used to see how well the applied coating technique follows the surface and fills cracks.
The analysis is rather comprehensive, so the following section is only showing the NiCuNi-coating. A summary of all the coatings' analysis can be found last in this section and the SEM pictures can be found in appendix 3.

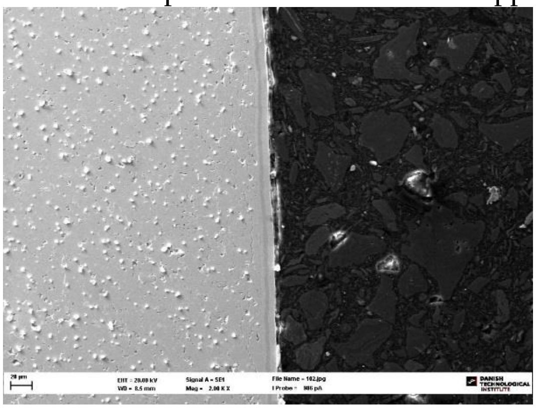


Figure 7 – NiCuNi-coating in SEM at 2000x zoom.

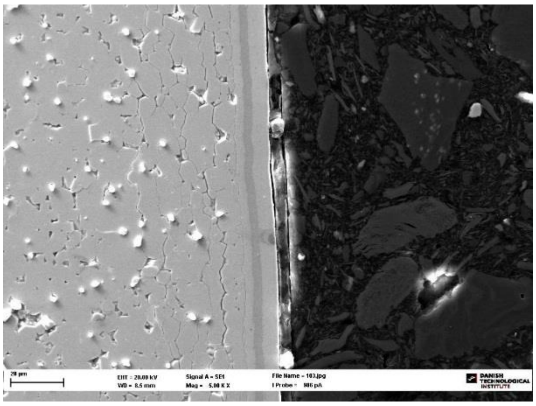


Figure 8 – NiCuNi-coating in SEM at 5000x zoom.

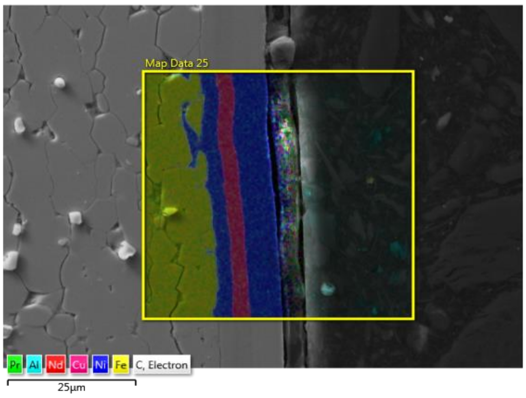


Figure 9 – NiCuNi-coating in SEM at 10000x zoom with EDX.

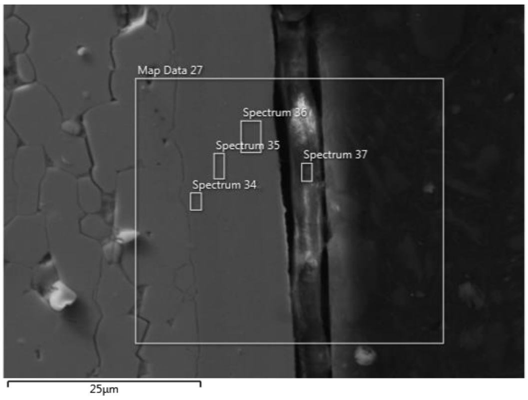


Figure 10 – NiCuNi-coating in SEM at 15000x zoom with EDX.

Figure 7-10 does not show any large variations in layer thickness. The inner layer of Ni and the layer of Cu are comparable in thickness, whereas the outer Ni-layer is a bit thicker.

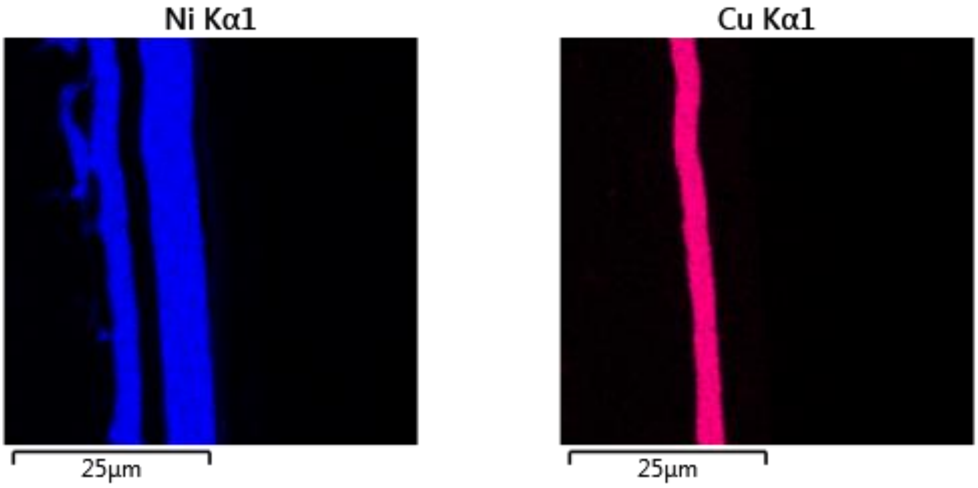


Figure 11 – EDX mapping of the NiCuNi-coating, showing the presence of different atoms at 10000x zoom.

It can be seen, that the inner layer of Ni is great at filling gabs, which else could lead to trapping of air in between the magnet and coating.

Spectrum 34

	Wt%	σ
Ni	93.1	0.4
C	4.1	0.4
Fe	2.8	0.1

Spectrum 35

	Wt%	σ
Cu	91.7	0.5
C	5.3	0.4
Ni	2.2	0.1
Fe	0.8	0.1

Spectrum 36

	Wt%	σ
Ni	94.9	0.4
C	4.5	0.4
Fe	0.6	0.1

Spectrum 37

	Wt%	σ
C	57.4	0.3
Ni	20.3	0.2
Zn	18.2	0.2
Fe	3.6	0.1
Ca	0.2	0.0
Si	0.1	0.0
Al	0.1	0.0
S	0.0	0.0

Figure 12 – Point analysis and mapping summary of EDX at 15000x, areas can be found in Figure 109.

Figure 1212 gives the chemical composition of specified regions as can be seen in Figure 109. Nothing unexpected or concerning percentages of elements have been found.

Zn: This coating shows some inhomogeneities in the coating, but the layer thickness is kept the same. The point analysis reveals that the different areas varies in composition of zinc from 10-30 wt%.

CZn: Shows some inhomogeneous characteristics with some variations in the thickness of the coating. Zinc varies from20-43 wt% depending on the area analysed at. Some areas appears as corns in which the zinc content is the highest.

Black Epoxy: This coating is rather well distributed and homogeneous. It contains roughly 3-4 wt% Al and Si.

Teflon: The coating is equal in thickness, but contains rather large dense variations through the coating. It contains aluminum rod fillers (approx. 3.5 wt%), which, to the best of our knowledge, works as a reinforcement of the coating added to the other benefits of Teflon as well as retaining the structure. Sulfur is added in 4.4 wt% and is homogeneously distributed throughout the coating, one of the downside effects of adding sulfur is, that it lowers the mechanical strength, which then leads to another reason for adding rod-shaped aluminum to the coating in order to compensate for this effect.

Sol-Gel: The coating is uniform and consists mainly of silicium (Si) and Oxygen (O)

JL Mag: Due to confidentiality, the SEM/EDX analysis is limited to pictures. The coating is uniform and contains flake like metallic particles.

6 Conclusion

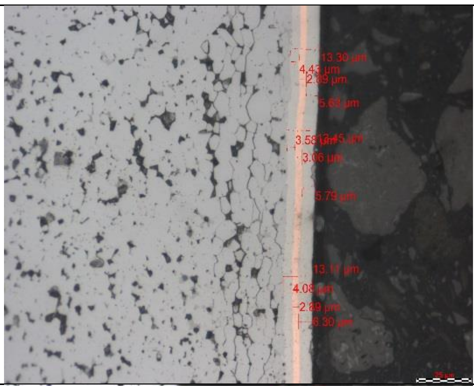
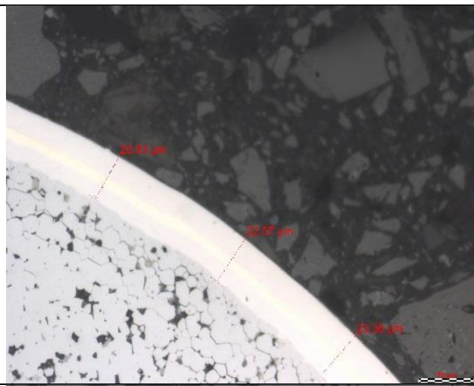
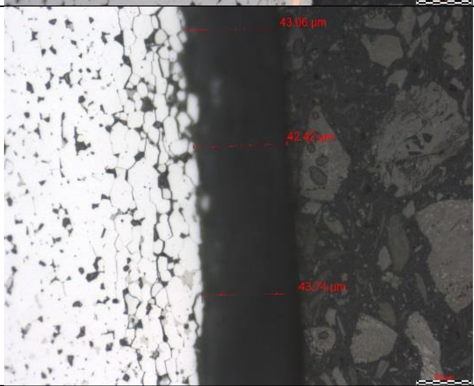
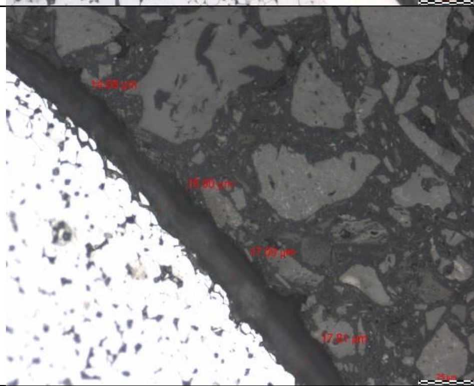
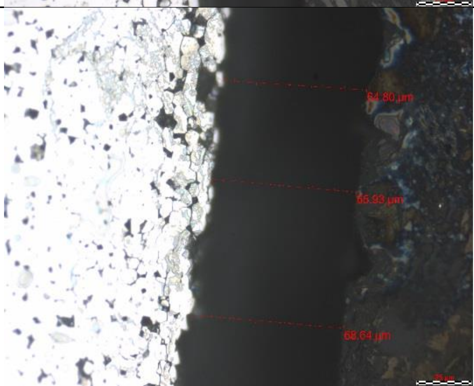
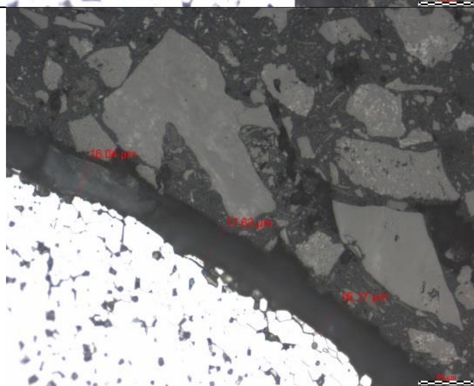
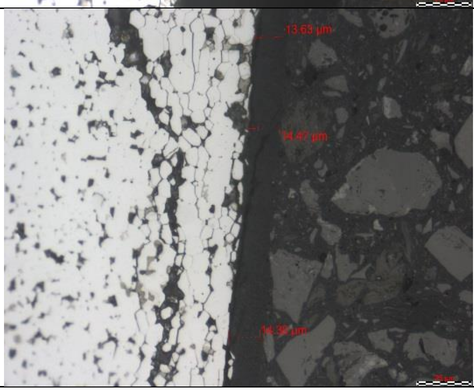
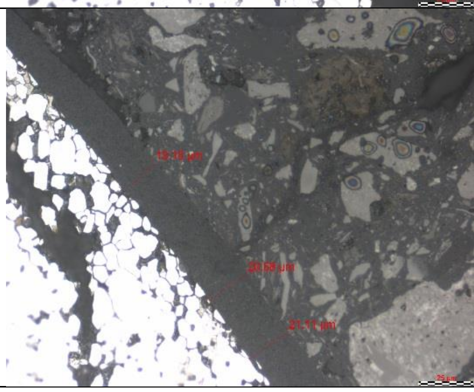
The tested coatings all have different strengths, however the coatings appears to perform in three different levels.
Poor: Black Epoxy, Teflon, Sol-Gel
Good: NiCuNi, Zn, CZn
Excellent: JL Mag
The primary reason for the placement in these groups is the performance in water immersion and salt spray testing.

At the NdFeB magnet itself is very prone to corrosion it must rely on the applied coating for corrosion protection. Therefore coatings without pores and a self healing ability will have the highest efficiency.
It should however be noted that the coatings tested in this report might not be representative for all coating of the same kind.

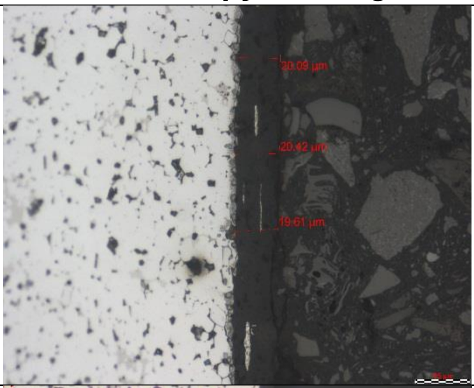
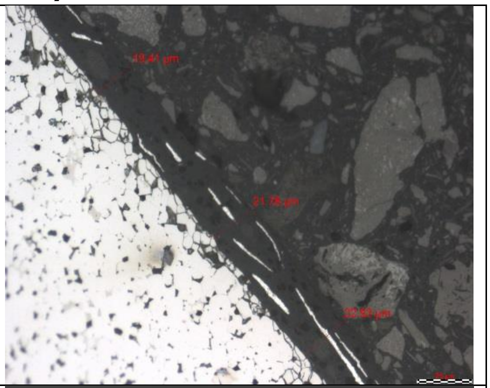
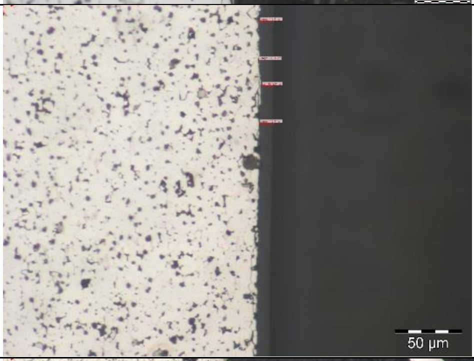
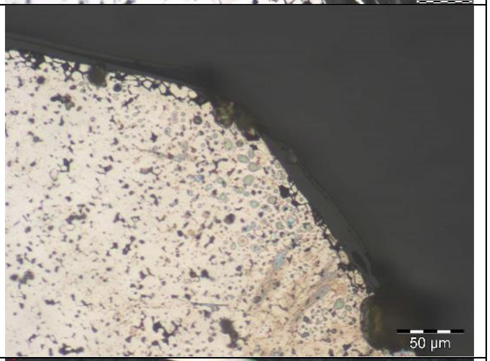
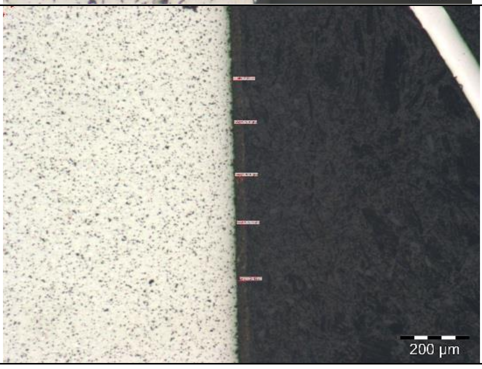
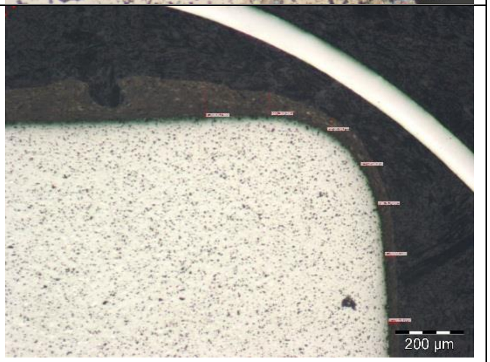
	NiCuNi	Zn	CZn	Black Epoxy	Teflon	Sol-Gel	JL Mag
Coating colour	Silver	Silver	Silver	Black	Black	Trans-parent	Silver
Immersion in water	Good	Good	Good	Average	Average	Poor	-
Immersion in oil	Good	Good	Good	Good	Good	Good	-
Salt spray [h]	Good	Good	Good	Poor	Poor	Poor	Excel-lent
Adhesion [MPa]	19.1±1.7	18.4±3.5	19.2±3.9	21.0±2.7	>16.7±1.7 *		-
Layer thickness [μm]	12.95 ±0.45	43.07 ±0.66	66.46 ±1.97	14.13 ±0.44	20.04 ±0.41	9,24 ±0,93	28,19 ±3,7
Corner thickness [μm]	22.29±1.24	16.26±1.42	16.60±0.87	20.32±1.01	21.37±1.79		38,00 ±5,08

*Table 5 – Summary of coating performances. *detachment between glue and coating.*

Light Optical Microscopy investigation (LOM)

	Surface	Corner
Ni-CuNi		
Zn		
CZn		
Black Epoxy		

Light Optical Microscopy investigation (LOM)

Tef-lon		
Sol-Gel		
JL Mag		

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Appendix 2.1

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SALT SPRAY RESULTS

Samples 1.x: NiCiNi, Samples 2.x: Zn, Sampels 3.x:CZn, Sampels 4.x: Black Epoxy, Samples 5.x: Teflon, Samples 6.x Phosphate + Sol-Gel, Samples 7.x: JL Mag epoxy

Hours	4	28	46	52	120	143	173	195	317	365	485	633	672	720	965	1036	1176
1.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1-2
1.2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3	0	0	0	0	0	0	0	0	1	1	1	1-2	2	2	3	3	3-4
1.4	0	0	0	0	0	0	0	0	0	0	0	0	1	1	2	2	2-3
1.5	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1-2	3
2.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	5		
2.2	0	0	0	0	0	0	0	0	0	0	0	0	2-3	3-4	5		
2.3	0	0	0	0	0	0	0	0	0	0	0	0	2-3	3-4	5		
2.4	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0		
2.5	0	0	0	0	0	0	0	0	0	0	0	0	2-3	3-4	5		
3.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	3-4		
3.2	0	0	0	0	0	0	0	0	0	0	0	0	0	1	4		
3.3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0		
3.4	0	0	0	0	0	0	0	0	0	0	0	0	0	0-1	4		
3.5	0	0	0	0	0	0	0	0	2	2-3	4	4-5					
4.1	0	1	1	1	1-2	1-2	2	2	3	4							
4.2	0	1	1-2	1-2	3	3-4	4	4-5	5								
4.3	0	1-2	3	3	4	4-5	5										
4.4	0	1	1-2	1-2	3-4	4-5	4-5	4-5	5								
4.5	0	1	1-2	1-2	3	3-4	4	4-5	5								
5.1	0	2	3-4	3-4	5												
5.2	0	2	3-4	3-4	5												
5.3	0	4	4-5	5													
5.4	0	4	4-5	5													
5.5	0	4	4-5	5													
6.1	0	3-4	4	4-5													
6.2	0	3-4	4	4-5													
6.3	0	3-4	4	4-5													
7.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
7.2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
7.3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

JL Mag epoxy samples were tested in saltspray for more than 2000hours without sign of corrosion.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Zn

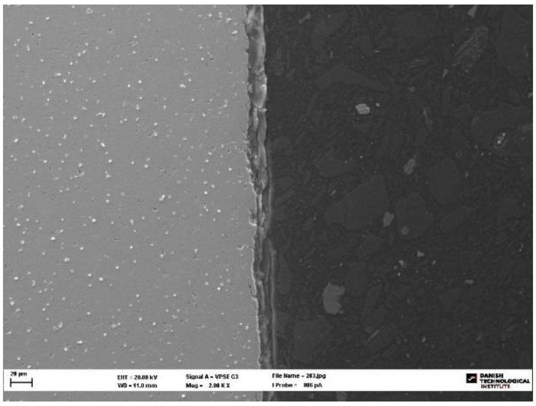

Figure 13 – Zn-coating in SEM at 2000x zoom.

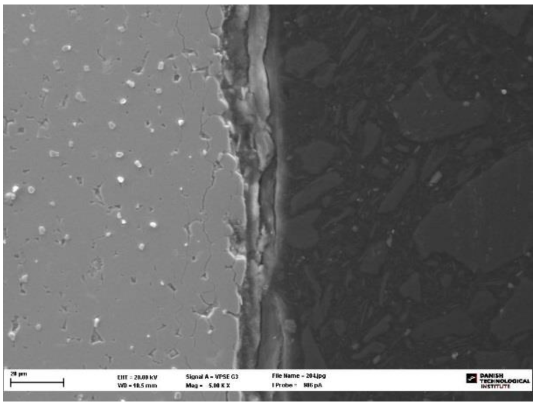

Figure 14 – Zn-coating in SEM at 5000x zoom.

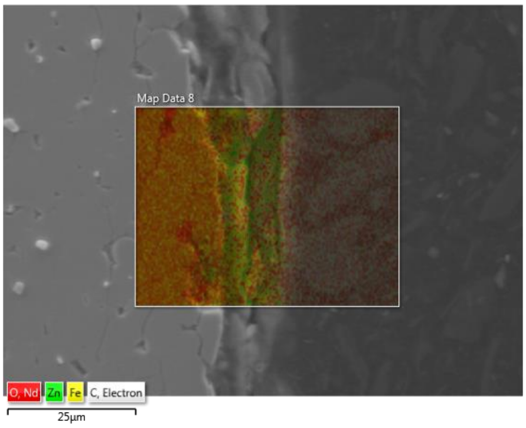

Figure 15 – Zn-coating in SEM at 10000x zoom with EDX

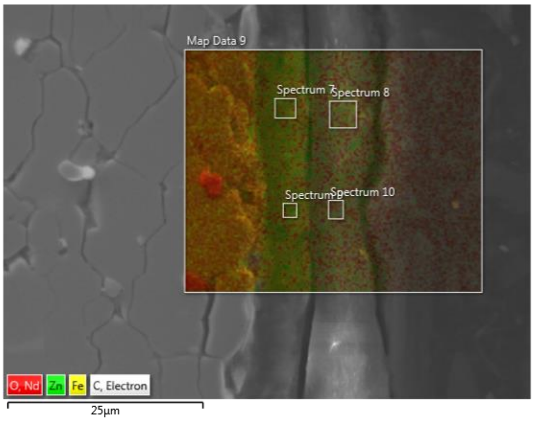

Figure 16 – Zn-coating in SEM at 15000x zoom with EDX.

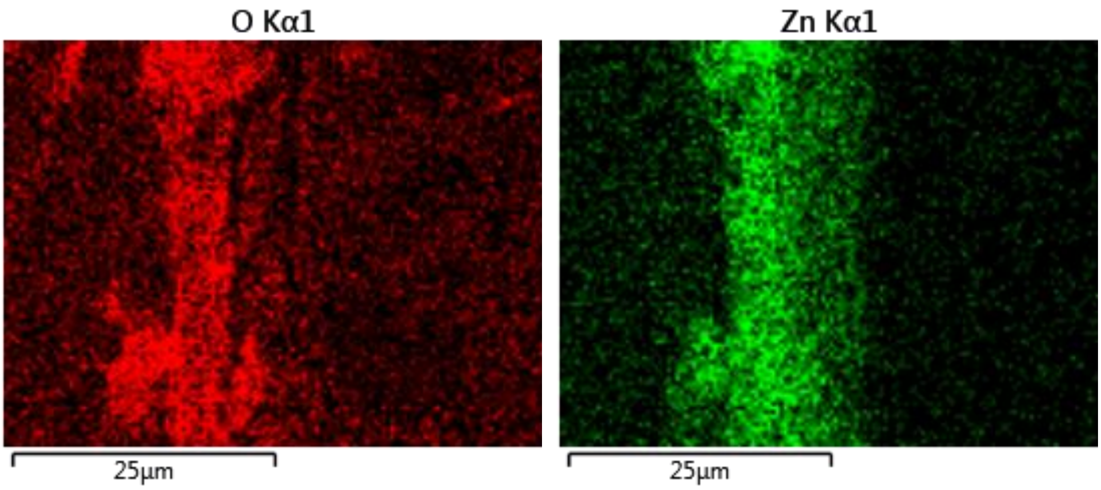

Figure 17 - EDX mapping of the Zn-coating, showing the presence of different atoms at 10000x zoom.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Spectrum 10

	Wt%	σ
C	65.1	0.3
Zn	10.8	0.2
Fe	10.8	0.1
O	6.8	0.2
Nd	3.8	0.1
Pr	1.1	0.1
Na	0.9	0.1
Al	0.3	0.0
Si	0.2	0.0
Cl	0.1	0.0

Spectrum 7

	Wt%	σ
C	39.7	0.4
Zn	30.7	0.3
O	13.9	0.2
Fe	9.3	0.1
Nd	3.1	0.1
Pr	1.1	0.1
Cl	0.9	0.0
S	0.6	0.0
Al	0.4	0.0
Si	0.2	0.0

Spectrum 8

	Wt%	σ
C	63.5	0.3
Zn	11.0	0.2
Fe	10.9	0.1
O	7.7	0.2
Nd	3.8	0.1
Pr	1.2	0.1
Na	1.0	0.1
Al	0.4	0.0
Si	0.3	0.0
Ca	0.1	0.0
S	0.1	0.0
Cl	0.1	0.0

Spectrum 9

	Wt%	σ
C	40.6	0.4
Zn	28.7	0.3
O	14.3	0.2
Fe	10.0	0.1
Nd	3.2	0.1
Pr	1.0	0.1
Cl	0.9	0.0
S	0.7	0.0
Al	0.5	0.0
Si	0.2	0.0

Figure 18 – Point analysis and mapping summary of EDX at 15000x, areas can be found in Figure 166.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

CZn

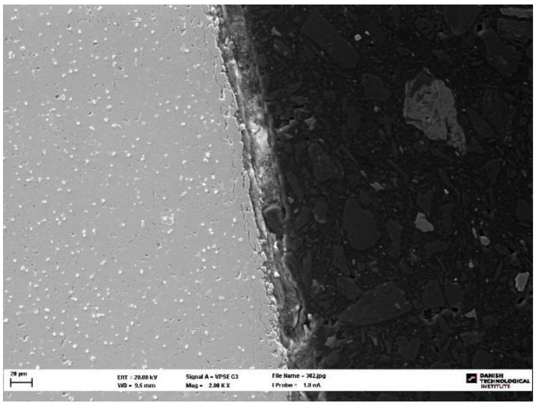

Figure 19 – CZn-coating in SEM at 2000x zoom.

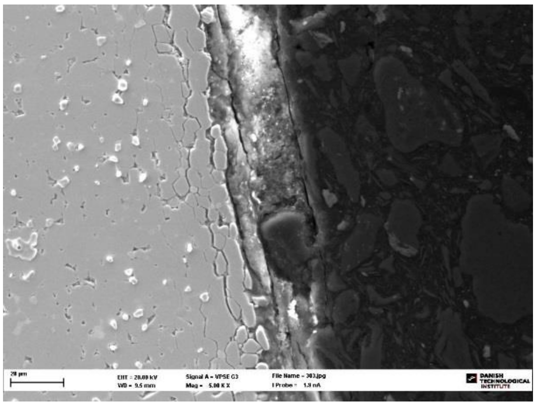

Figure 20 – CZn-coating in SEM at 5000x zoom.

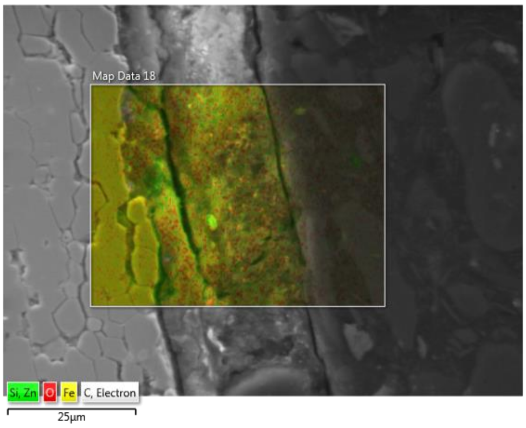

Figure 21 – CZn-coating in SEM at 10000x zoom with EDX.

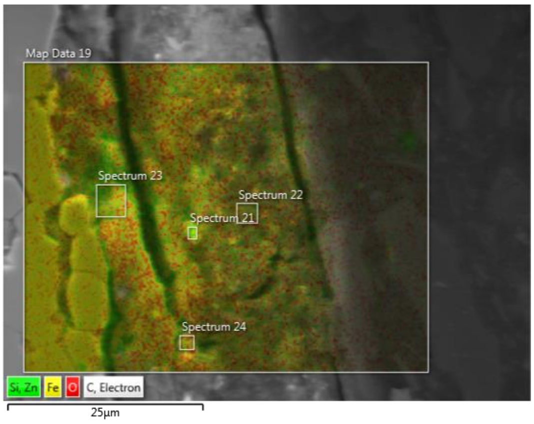

Figure 22 – CZn-coating in SEM at 15000x zoom with EDX.

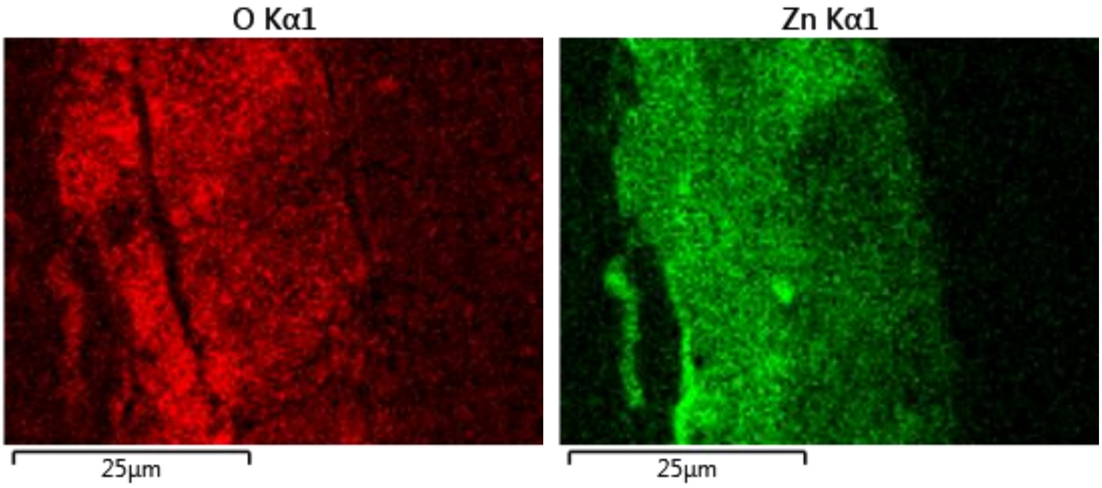

Figure 23 – EDS mapping of the CZn-coating, showing the presence of different atoms at 10000x zoom.

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Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Spectrum 21

	Wt%	σ
Zn	42.9	0.4
C	34.8	0.4
O	10.9	0.2
Fe	7.3	0.1
Nd	2.4	0.1
Pr	0.8	0.1
Al	0.3	0.0
S	0.2	0.0
Si	0.2	0.0
Cl	0.1	0.0
Ca	0.1	0.0

Spectrum 22

	Wt%	σ
C	47.4	0.3
Zn	20.1	0.2
O	14.0	0.2
Fe	11.5	0.1
Nd	3.3	0.1
Na	1.4	0.2
Pr	0.9	0.1
Al	0.4	0.0
S	0.3	0.0
Si	0.3	0.0
Cl	0.3	0.0

Spectrum 23

	Wt%	σ
C	35.9	0.4
Zn	32.0	0.3
O	12.8	0.2
Fe	11.3	0.1
Nd	4.0	0.1
Na	1.4	0.2
Pr	1.2	0.1
Al	0.5	0.0
S	0.3	0.0
Cr	0.3	0.1
Si	0.2	0.0
Cl	0.2	0.0

Spectrum 24

	Wt%	σ
C	38.5	0.4
Zn	26.7	0.3
O	16.4	0.2
Fe	10.2	0.1
Nd	3.2	0.1
Na	1.8	0.2
Pr	1.1	0.1
Cr	0.9	0.1
Al	0.4	0.0
S	0.3	0.0
Cl	0.3	0.0
Si	0.3	0.0

Figure 24 – Point analysis and mapping summary of EDX at 15000x, areas can be found in Figure 2223.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Black Epoxy

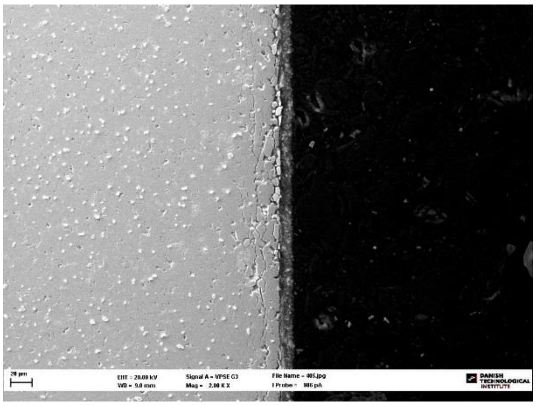

Figure 25 – Black Epoxy-coating in SEM at 2000x zoom.

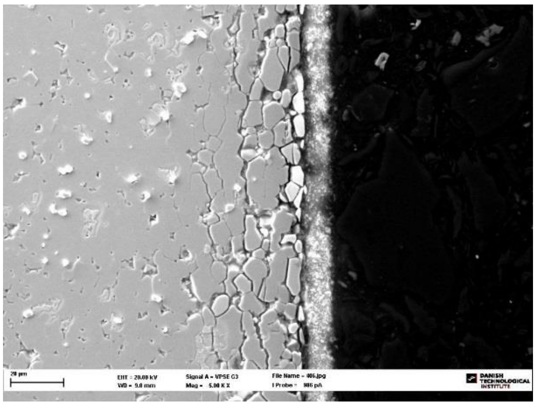

Figure 26 – Black Epoxy-coating in SEM at 5000x zoom.

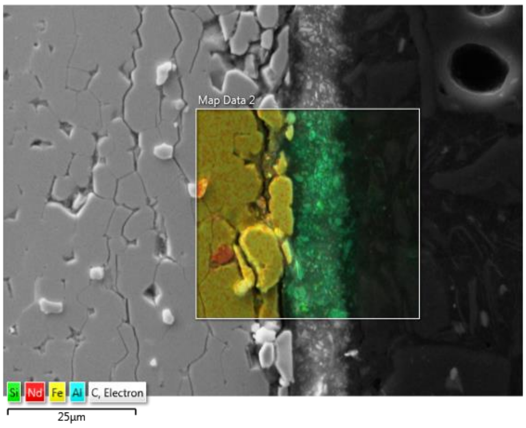

Figure 27 – Black Epoxy-coating in SEM at 10000x zoom with EDX.

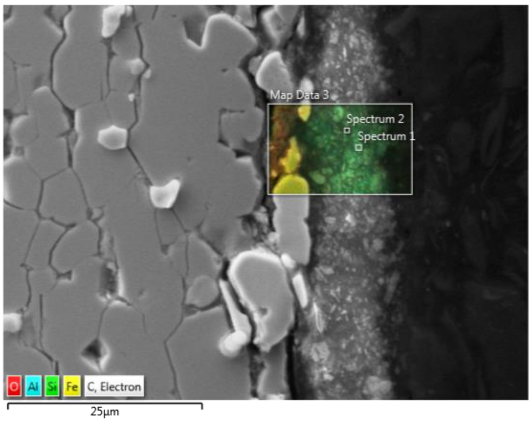

Figure 28 – Black Epoxy-coating in SEM at 15000x zoom with EDX.

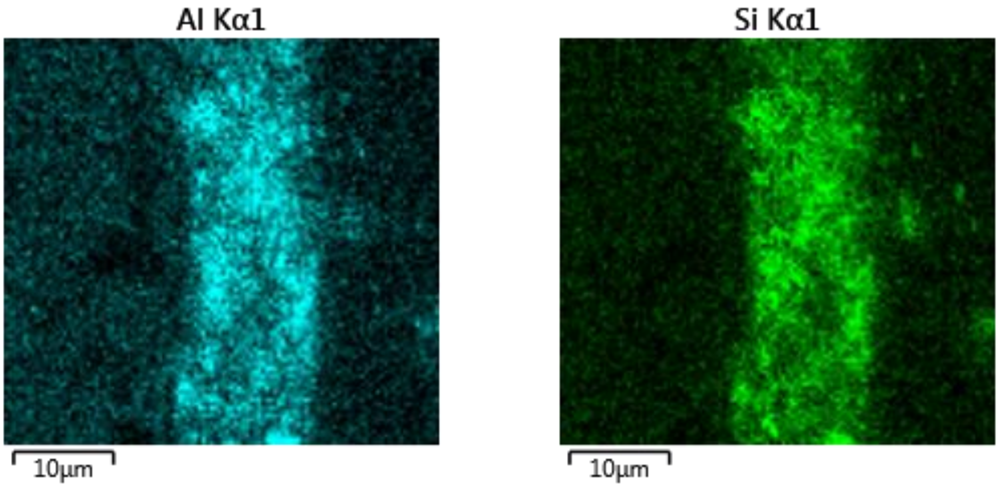

Figure 29 – EDS mapping of the Black Epoxy-coating, showing the presence of different atoms at 10000x zoom.

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Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Spectrum 1

	Wt%	σ
C	60.2	0.3
O	14.6	0.3
Fe	8.5	0.1
Al	6.1	0.1
Si	5.8	0.1
Nd	3.2	0.1
Pr	1.0	0.1
Sn	0.2	0.1
Ti	0.2	0.0
Ca	0.1	0.0
P	0.1	0.0
S	0.1	0.0

Spectrum 2

	Wt%	σ
C	63.9	0.3
O	14.8	0.2
Fe	8.1	0.1
Al	4.6	0.1
Si	4.4	0.1
Nd	3.1	0.1
Pr	1.0	0.1
Ca	0.1	0.0
Ti	0.1	0.0

Figure 30 – Point analysis and mapping summary of EDX at 15000x, areas can be found in Figure 288.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Teflon

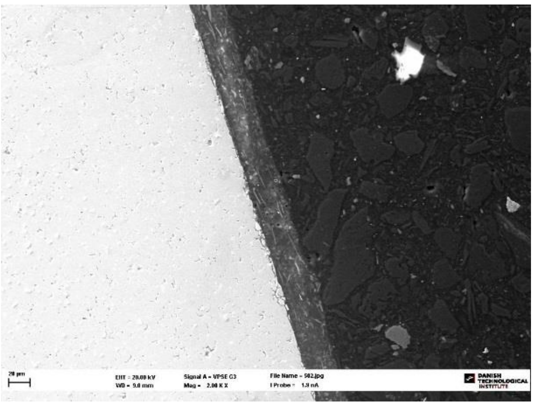


Figure 31 – Teflon-coating in SEM at 2000x zoom.

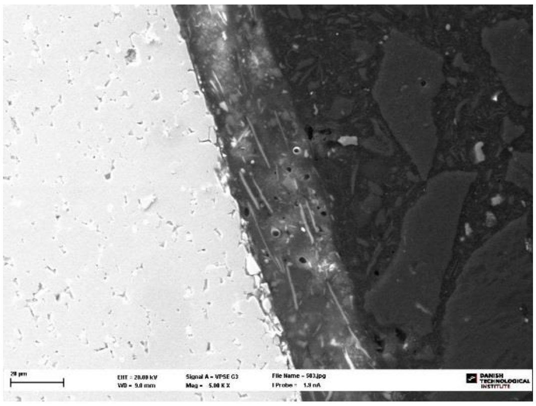


Figure 32 – Teflon-coating in SEM at 5000x zoom.

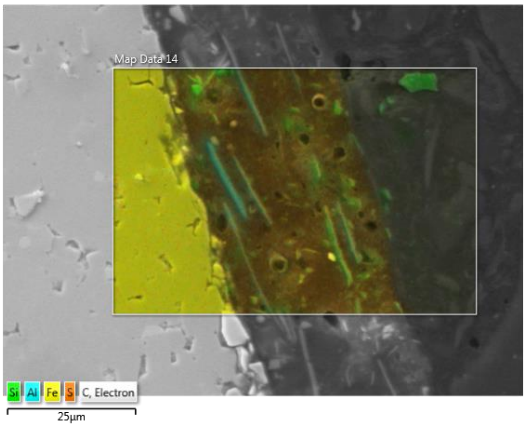


Figure 33 – Teflon-coating in SEM at 10000x zoom with EDX.

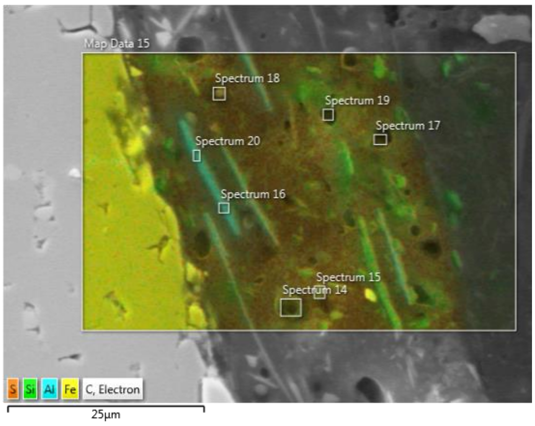
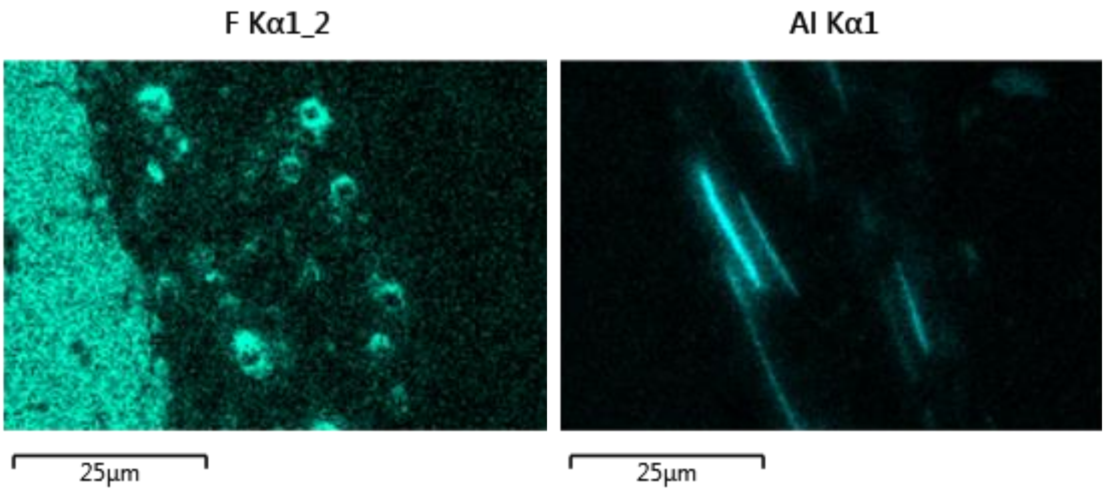


Figure 34 – Teflon-coating in SEM at 15000x zoom with EDX.



Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

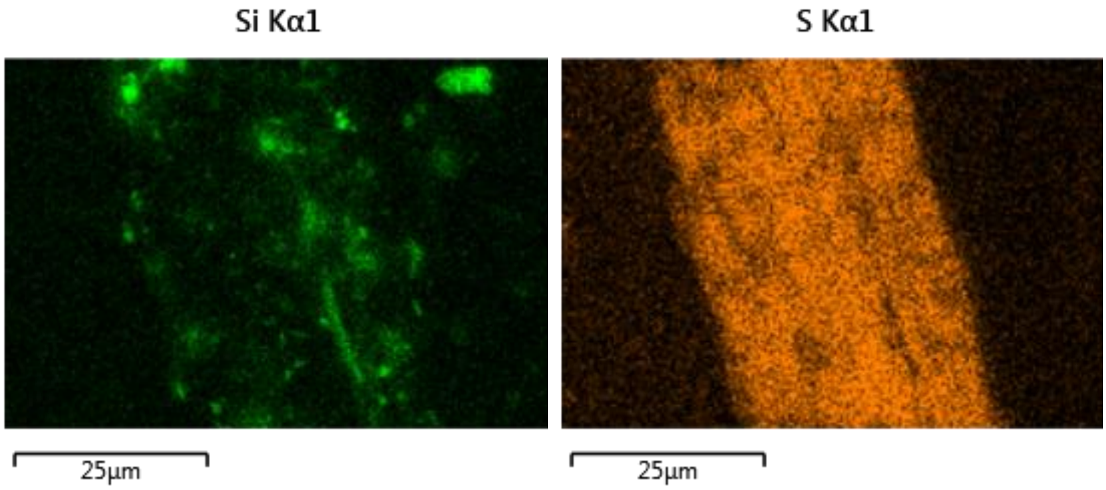


Figure 35 – EDX mapping of the Teflon-coating, showing the presence of different atoms at 10000x zoom.

Spectrum 14

	Wt%	σ
C	49.5	0.7
F	14.4	0.3
Fe	12.2	0.2
S	7.2	0.1
Nd	4.4	0.2
O	4.1	0.2
Si	3.8	0.1
Al	2.1	0.1
Pr	1.3	0.1
K	0.7	0.0
Ca	0.1	0.0

Spectrum 15

	Wt%	σ
C	51.6	0.6
O	14.8	0.3
Fe	9.0	0.2
S	7.7	0.1
F	7.5	0.2
Si	3.6	0.1
Nd	3.3	0.1
Al	1.1	0.0
Pr	1.0	0.1
K	0.3	0.0
Ca	0.1	0.0

Spectrum 16

	Wt%	σ
C	43.7	0.9
Al	29.4	0.5
Fe	8.3	0.2
O	5.2	0.2
F	4.3	0.2
S	4.2	0.1
Nd	3.0	0.1
Pr	0.9	0.1
Si	0.8	0.0
K	0.2	0.0
Ca	0.1	0.0

Spectrum 17

	Wt%	σ
C	56.3	0.6
Fe	11.8	0.2
S	8.7	0.1
F	5.2	0.2
O	4.6	0.2
Nd	4.3	0.2
Al	3.3	0.1
Si	3.1	0.1
Pr	1.4	0.1
K	1.0	0.0
Ca	0.3	0.0

Spectrum 18

	Wt%	σ
C	53.3	0.6
F	13.7	0.3
Fe	10.4	0.2
S	7.7	0.1
O	5.9	0.2
Nd	3.7	0.1
Al	2.0	0.0
Si	1.7	0.0
Pr	1.0	0.1
Ca	0.4	0.0
K	0.2	0.0

Spectrum 19

	Wt%	σ
C	53.5	0.6
F	11.3	0.2
Fe	10.3	0.2
S	6.1	0.1
O	5.1	0.2
Si	4.2	0.1
Nd	3.9	0.1
Al	2.0	0.1
Pr	1.2	0.1
K	1.2	0.0
W	0.5	0.2
Na	0.4	0.1
Ca	0.2	0.0
Mg	0.1	0.0

Spectrum 20

	Wt%	σ
C	46.4	0.9
Al	27.2	0.4
Fe	8.9	0.2
O	6.8	0.2
S	5.2	0.1
Nd	3.3	0.1
Si	1.0	0.0
Pr	0.9	0.1
K	0.1	0.0
Ca	0.1	0.0

Figure 36 – Point analysis of EDX at 15000x, areas can be found in Figure 344.

Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

Sol-Gel

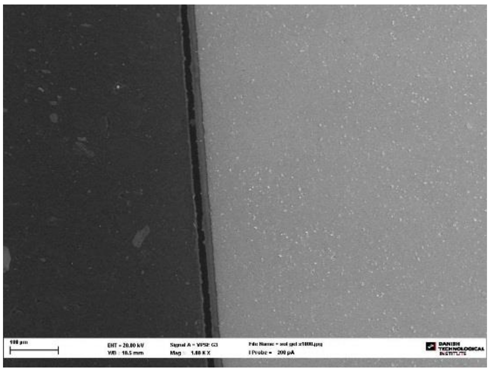


Figure 37 – Sol-Gel-coating in SEM at 1000x zoom

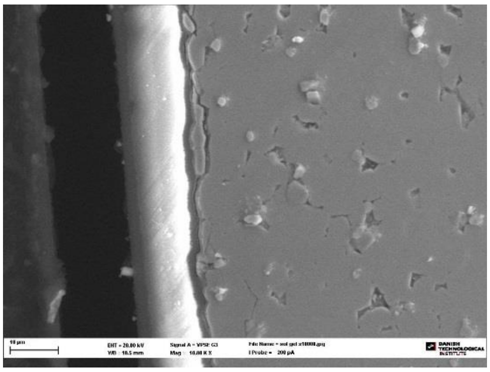


Figure 38 – Sol-Gel-coating in SEM at 10000x zoom

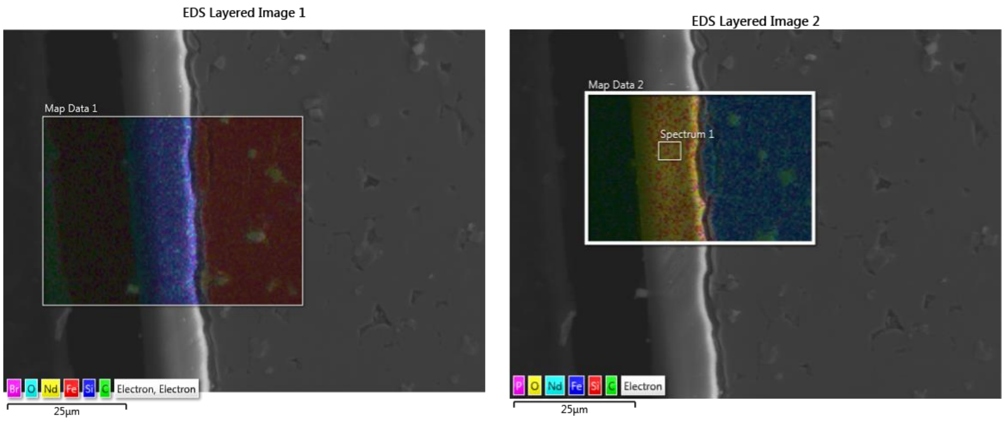
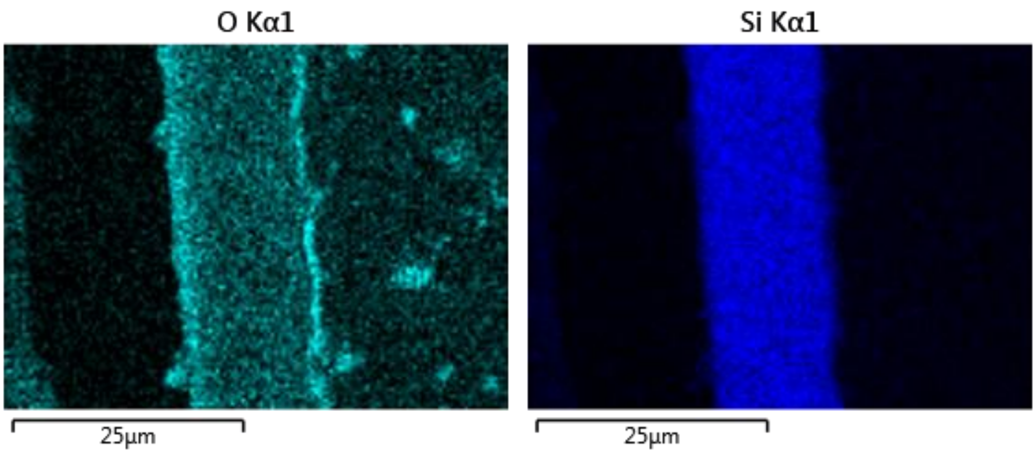


Figure 39 – Sol-Gel coating in SEM at 10000x zoom with EDX.



Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

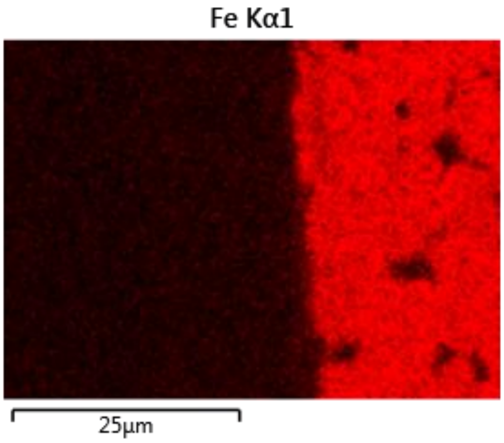


Figure 40 – EDX mapping of the Teflon-coating, showing the presence of different atoms at 10000x zoom.

Spectrum 1

	Wt%	σ
C	53.2	0.3
Si	16.1	0.1
O	13.8	0.2
Fe	10.9	0.1
Nd	4.1	0.1
Pr	1.3	0.1
Al	0.4	0.0
P	0.1	0.0

Figure 41 – Point analysis of EDX at 10000x, areas can be found in Figure 349.

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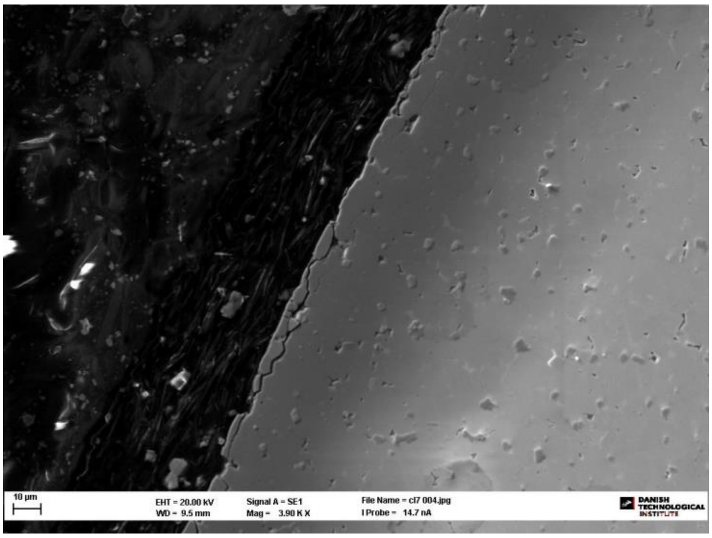
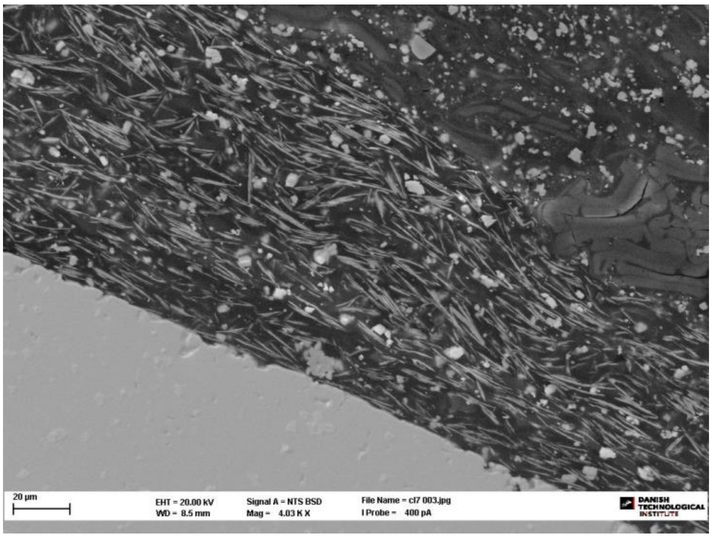
Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)

JL mag epoxy





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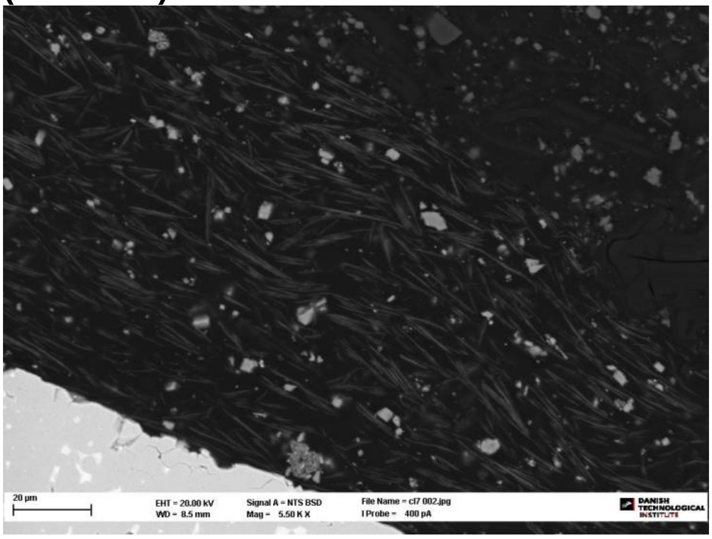
Scanning Electron Microscopy equipped with Energy Dispersive X-ray (SEM/EDX)



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Appendix 3.1

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