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Mitigation of Risks of Corrosion and Delamination by Surface Pre-Treatment

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The corrosion and delamination are significative sources of risks for metal components and threaten their functions. From this reason, the surface pre-treatment is carried out. One of the surface pre-treatment process is a powder coating. The application of powder coating is described in the paper by experiments. To obtain reliable results of surface pre-treatment process by powder coatings in practice, we create methodology based on process safety management, which is based on management of risks connected with process. We follow both, the partial risks and the integral risk evaluated by checklist.

Key words: Metal components, corrosion, delamination, risk, surface pre-treatment, powder coating, process safety management, check list.

1. Introduction

For the needs of practice, the surfaces of metal components and objects need to be safe, so that they do not endanger the people who use them and so that they perform the tasks for which they are intended. It is a fact that metal surfaces are significantly damaged by corrosion and delamination, i.e. disconnection of surface layers from the inner layers. Both of these phenomena significantly reduce the service life of both equipment and objects. The size of the damage depends on both, the method of use and the aggressiveness of the environment in which the metal components or objects are located. Therefore, a pre-treatment of surfaces is carried out.

The submitted article examines the mitigation of risks caused by corrosion and delamination by help of pre-treatment of the surface of metal samples using a coating of powdered plastics. The article describes selected methods of powder coating using three pre-treatment methods, namely:

- blasting,
- phosphating
- and zircon-based nano passivation.

The aim of the experiments is to find out which surface pre-treatment leads to better resulting surface properties, i.e. higher stability, abrasion resistance and durability in terms of:

- mechanical tests,
- resistance to corrosion and subsequent delamination of the surface or coating

and to create recommendation for using these procedures in practice.

In the experiment, all samples, with surface pre-treatments used, were exposed in the salt chamber for the same exposure time, and after the exposure we evaluated the mechanical properties, corrosion and delamination of the coating. The properties in question were evaluated according to the relevant standards.

To obtain reliable results of surface pre-treatment process by powder coatings in practice, we create methodology based on process safety management, which is based on management of risks connected with process. We follow both, the partial risks and the integral risk evaluated by checklist.

The results show interesting findings for certain types of surface pre-treatment and it is not clear which pre-treatment is the best overall. Analysis of experiments showed that some sample pre-treatments reduce impacts of corrosion and delamination of coatings more than others. Some pre-treatments proved to be very good in terms of mechanical properties and others very good in terms of corrosion and delamination of the coating. From a practical point of view, the pre-treatment of coatings with powdered plastics should be carried out considering the application and the environment in which they will be used. In all cases, it is necessary, however, to ensure safe process of pre-treatment.

2. Summary of Knowledge

2.1. *Corrosion and Delamination*

The authors Fontana (1987) and Shaw (2006) state that corrosion is often defined as the spontaneous process of degradation or deterioration of a material due to interaction with its environment. There is often a misconception that this definition should be reserved for metallic materials only. But the reality is that some non-metallic materials, such as ceramics and plastics, are also subject to corrosion. Unfortunately, corrosion cannot be avoided, but it can be studied and learned to prevent it for a longer period of time, thereby extending the life of the product. The main driving force behind corrosion processes is Gibbs energy or free enthalpy. This embodied energy should be minimized because the less energy there is in a material, the more equilibrium state it is in. In manufacturing, energy is always added to the material. If there is a high amount of energy in the material, it tries to bring itself back to a more comfortable oxidized state. It does this by releasing electrons (oxidation).

Shaw (2006) and Schweitzer (2010) state that mankind is mainly concerned with corrosion because of three main factors, which are safety, economics and security. Nowadays, for example, safety is dealt with very often for old bridges, which have usually been poorly protected against corrosion. This is also why coatings are nowadays applied to galvanized bridge structures, or powdered plastics. The fact that corrosion must also be tackled from an economic point of view is demonstrated by dozens of studies have been carried out for decades, showing that the annual direct cost of corrosion to industry is approximately 3.1% of gross national product. It is estimated that up to 50% of the costs associated with associated with the problem of unwanted oxidation of materials is caused by atmospheric corrosion. All these studies have led to the development of new ways to protect material surfaces.

Thouless (1991) states that the mechanism of coating failure is dependent on many factors. These factors are the properties of the coating, the interface and the substrate and also the stress distribution. When a coating is subjected to residual tensile stress, there are at least three possible mechanisms by which failure can occur. The coating may crack by the formation of cracks throughout the thickness of the coating, which is the case with a brittle coating. Tougher coatings can fail by delamination along the interface or even by crack propagation within the substrate. The failure mechanism associated with compressive stresses in the coating involves simultaneous buckling and delamination.

Chen and Bull (2011) states that according to the definition of ASTM (D907-70), adhesion is a state in which two surfaces are held together by interfacial forces, which may consist of valence forces or lock forces or both. These forces can be Van der Waals forces, electrostatic forces or chemical bonds via the coating/substrate interface. Delamination is a process in which a layer is separated from the substrate or (fiber) matrix in composite materials. Which can be caused by mechanical or thermal stress, shock waves, corrosion, electrostatic forces, etc. It can be detected by indirect techniques such as acoustic emission or reflectivity, but it is best observed directly by microscopy (light, electron, atomic force, acoustic, etc).

2.2. *Surface Pre-Treatment*

During the production processes, the surfaces of the products are contaminated with various contaminants. These impurities impede the correct application of the subsequent surface treatment and must be removed from the surface. Kreibich (1996) gives that impurities can be divided into:

- foreign impurities
- and impurities proper.

Foreign impurities are impurities that have adhered to the surface, for example, during turning, these are various emulsions, coolants, but also fats, lubricants and can best be removed by degreasing surgery.

On the contrary, impurities proper are impurities that are bound on the surface by chemical reactions, such as scales from production after hot forming.

Therefore, it is necessary to properly clean the surface of the product before any application of the surface treatment so that the subsequent surface treatment anchors well. Adhesion can be influenced by a suitably selected surface pre-treatment (Kreibich 1996).

Mechanical surface pre-treatment can be used, which cleans the surface and at the same time creates an anchoring profile, which is important for powder plastic applications. Mechanical surface pretreatments include operations that use kinetic energy to pre-treat the surface. They aim not only to clean the surface, but to create the necessary anchoring profile, improve mechanical properties, increase corrosion resistance, or create the necessary surface appearance. This category of pre-treatment includes blasting, tumbling, grinding, polishing and brushing technologies (Kreibich 1996).

Chemical surface pre-treatment has two properties (Kreibich 1996). The first property increases the corrosion resistance and the second property increases the adhesion of organic coatings. The works (ITS 2020, Kreibich 1996, Narayanan 2005) state that chemical surface pre-treatments are usually carried out after mechanical pre-treatments. These are technologies of lubrication, pickling, phosphating, chromium-plate and passivation using the nanotechnologies (CGPC 1999, Kreibich 1996, Narayanan 2005).

For powder coating technology, chemical pre-treatment is an important step. It is possible to encounter different views of chemical pre-treatment, where it can be created by three different technological processes. The first is only degreasing itself, the second is degreasing and creating a conversion layer in one neck, e.g. ferric phosphating and the third method consists of successive degreasing operations and creating a conversion layer, which is used, for example, by zircon-based nano-passivation (Dunham 2013, ITS 2020).). In recent years, zirconia conversion coatings have proven to be an excellent replacement for ferric phosphate pretreatments (Dunham 2013).

3. Process Safety Management

Process Safety Management (PSM) has different versions in the world. It represents complex procedure and requires a multidimensional approach that blends technology and their management (Prochazkova 2017). Process Safety Management is connected with safety culture and for its safety assessment, the checklist is often used (US DOE 1996).

In the European Union the process safety management is related to the storage and handling of hazardous chemicals (EU 2012) to limit risks. In the United Kingdom, the Control of Major Accident Hazards (COMAH) Regulations 2015 cover PSM (UK 2015). It is addressed in specific standards for the general and construction industries.

4. Method of Powder Coating

CP (2023) states that barrier protection is the oldest and most widely used type of corrosion protection of the base material. The principle is that the coating applied to the substrate prevents contact between the base with the corrosive environment. However, this method of material protection fails in the moment, when the insulating coating is breached, as the surface of the base material immediately becomes contact with the corrosive environment and corrosive processes are initiated. It is therefore necessary to emphasize the correct technological procedures in the production of the protective coating to meet the necessary adhesion to the base material, sufficient thickness and mechanical properties. ***From this it follows that process of powder coating must be safe, in order that the results with high quality would be ensured*** for practice needs.

Barrier protection provided by powder plastic coatings is primarily intended for corrosion protection and design purposes and its mechanical properties are limited. There are many ways to apply powder plastics. These are methods of application using a tribo gun, or using high voltage, or fluidized (Kreibich 1996).

In works (CGPC 1999, Kreibich 2010, Pélissier 2020) it is stated that in this technology the powder is transported through a tube to a gun, where an electrode is placed connected to a source of high voltage (30 to 100 kV) and low current, which ultimately gives less energy. This electrode releases electrons out of the gun, which form a cloud of ions called a corona in the area around the hole. The air-transported powder-colored particles, when released from the gun hole, pass through an ionized area where the ions become trapped on a large number of particles, which become electrically charged, Figure 1. The potential difference between the paint particles and the substrate creates a coating of powdered plastic.

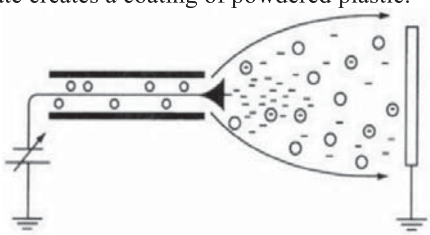

Fig. 1. Scheme of electrostatic charging of powder (Kreibich 2010).

The works (CGPC 1999, Kreibich 2010, Pélissier 2020) state that electro-kinetic charging of powder plastics is an older technology than electrostatic charging. The principle consists in rubbing air-transported particles of powder paint against the wall of the tube in the gun, which is made of polytetrafluoroethylene (Teflon). The charged particles are attracted to the earthed base material, Figure 2. Since there are not so large potential differences between the particles and the substrate in this process, there is no risk of a Faraday cage effect.

The amount of powder paint particles is regulated by the amount of compressed air. According to works (CGPC 1999, Kreibich 2010, Pélissier 2020), it is the oldest method of applying powder plastics to substrate. This method involves immersing the part in a fluidizing bed, which is the space where the powder paint is located in the fluidized state. It gets into this state by blowing clean air in the lower part of the bed through the porous bottom. Basically, there are two types of fluid powder coating, with or without electrostatic forces.

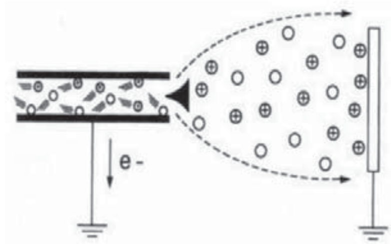

Fig. 2. Scheme of electro-kinetic charging of powder (Kreibich 2010).

Non-electrostatic application always requires the substrate to be preheated to a temperature of around 230 to 450 °C. Therefore, sample is immersed in a fluidized bed where the powder is melted to the surface for 2 to 10 seconds. After removing the substrate, the paint hardens and a coating is formed, on which, however, some adhering particles are undissolved, so the part continues to the furnace (Figure 3).

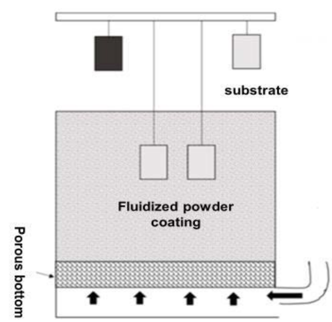

Fig. 3. Scheme of fluidized bed coating of powder plastic (Pélissier 2020).

In the second variant, electrostatic forces help to attach the powder to the surface, thanks to charged powder particles. In this variant, preheating of the part may or may not be included. The larger the preheating, , the thicker the coating layer will form.

5. Data and Methods Used

5.1. *Experiment Description*

The aim of the experiments is to evaluate:

- which surface pre-treatment powder plastic coatings most of all mitigates corrosion and delamination,

- what is the adhesion of the surface treatment after individual surface pre-treatments.

Samples of unalloyed structural steel S355J2 with dimensions of 150x100x3 mm were used for the experiments. Three surface pre-treatments were used:

- The first surface pre-treatment was mechanical blasting.
- The second surface pre-treatments were chemical-based, namely:
  - surface pre-treatment by phosphating technology
  - and surface pre-treatment based on nano passivation with zircon.
- The third surface pre-treatment consisted in applying the polyester powder coating executed to the samples using the electrostatics.

The samples were made in the company PZP KOMPLET, a.s. and in the laboratories of the Faculty of mechanical engineering in Praha. The resulting coating quality was affected by the pre-treatment used and the curing conditions, which were the same for all samples Curing took place in a hot air furnace for 20 minutes at a temperature of 180 ^{0}C.

The quality assessment of the coating:

- was carried out by corrosion test in neutral salt spray according to (ČSN EN ISO 9227)
- and the degree of delamination and corrosion in the vicinity of the cut were carried out according to (ČSN EN ISO 4628-8).

5.2. *Tool for Ensuring Process of Powder Coating Quality*

As it is mentioned in chapter 4, the process of powder coating must be safe so the high-quality results may be obtained. To ensure high quality of this process, i.e. experiment results and correct recommendations for practice, we follow stable conditions at which experiment is done; i.e. we mitigate risk of unacceptable phenomena. For this we use check list method (Prochazkova et al. 2019). According to rules given in (Prochazkova 2013) we:

- compiled the special check list in Table 1,
- used the scale for its evaluation given in Table 2.

Table 1. Checklist for the cleaning process; Y-YES, N-NO.

Question	Y	N
Was the same base material used for all experiments?		
Has the pH of the degreasing bath been measured?		
Were the correct mechanical surface pre-treatments used?		
Was the blasting abrasive clean?		
Was the size of the abrasive examined?		
Was the temperature in the phosphatize bath correct?		
Was there a subsequent sufficient rinse?		
Was degreasing before subsequent nano passivation sufficient?		
Has zircon nano passivation been well done?		
Was there a subsequent sufficient rinse?		
Were the correct testing methods used?		
Was the coating thickness measured for the samples?		
Was testing carried out and evaluated according to standards?		
Was industrial salt used to test for corrosion?		
Was the right adhesive used to test adhesion?		
When testing adhesion, was the adhesive well cured?		
Were the samples taken under defined lighting conditions?		
Was there a subsequent sufficient rinse?		
TOTAL		

Table 2. Value scale for determining the level of risk in measurement; the risk level $\boldsymbol{r = n/N}$, where $\boldsymbol{n}$ is the number of answers NO and $\boldsymbol{N}$ is the total number of questions.

Risk rate	Values *r in* %
Extremely high – 5	More than 95 %
Very high – 4	70 - 95 %
High – 3	45 - 70 %
Medium – 2	25 – 45 %
Low – 1	5 – 25 %
Negligible – 0	Low than 5 %

6. Evaluation of Results

We separately present the real technical results and recommendation for ensuring the quality results of powder coating process.

6.1. *Evaluation of Experiment Results*

The corrosion test in neutral salt spray was carried out according to the standard (ČSN EN ISO 9227). The standards (ČSN EN ISO 9227, ČSN EN ISO 4628-8) state that at the beginning of this test, a cut of 100 mm long and 0.5 mm wide according to ČSN EN ISO 17872 is made in the middle of each specimen. The cut must be made up to the base material. Subsequently, the edges of the sample are covered with self-adhesive tape around the entire circumference to prevent corrosion from spreading from there, which would distort the results.

All samples were placed in racks located in the salt chamber. There is an atmosphere in the salt chamber into which the 5 % sodium chloride solution shown in Figure 4 is sprayed. The samples were checked sequentially at intervals of 24 h, 48 h, 72 h, 168 h, 240 h, 480 h, 720 h and 1000 h. In addition, some samples were evaluated for the degree of corrosion and delamination around the cut according to ČSN EN ISO 4628-8. For samples 1.10, 2.10 and 3.10, these properties were evaluated after 480 hours. In addition, after 720 hours for samples 1.11, 2.11 and 3.11 and after 1000 hours in neutral salt mist for samples with index 1.12, 1.13, 2.12, 2.13 and 3.13.

Fig. 4. Corrosion chamber S-400 M-TR (Labortechnik 2023).

The standards (ČSN EN ISO 9227, ČSN EN ISO 4628-8) state that for some samples it is appropriate to evaluate the degree of delamination after removal from the corrosion chamber. Coating delamination describes the adhesion of a coating after corrosion tests. In neutral salt mist, a powder coating is released from the substrate over time away from the cut. In order to determine the value of delamination, it is necessary to remove the loose coating on the surface of the substrate. The coating is removed manually with a spatula. The distance of the exposed sample from the cut is then measured at intervals of 10 mm. From these values calculate the degree of delamination d:

$$\boldsymbol{d} = \frac{\boldsymbol{d_1} - \boldsymbol{w}}{\boldsymbol{2}} \tag{1}$$

where d_1 is the average total area of delamination, in millimeters; and w width of the original cut in millimeters.

After the delamination is evaluated, the same samples are evaluated for the degree of corrosion, or corrosion around the cut. The first step for successful evaluation is to remove all coating on the surface of the samples. The coating is removed using a paint stripper that is applied to the entire surface of each sample. Subsequently, with the help of a spatula, all coating is removed from the surface. After cleaning the samples, the corrosion distances of the base material from the section are measured at intervals of 10 mm. These values are averaged and then inserted into the formula for the corrosion degree:

$$\boldsymbol{c} = \frac{\boldsymbol{w_c} - \boldsymbol{w}}{\boldsymbol{2}}, \tag{2}$$

where w_c. is the average total width of the corrosion area in millimeters; and w width of the original cut in millimeters. The values obtained in the experiment are shown in Table 3.

Table 3. Evaluation of the degree of delamination and corrosion around the cut (Tlaskal 2022).

Time [h]	Sample No.	d- degree of delamination	c- degree of corrosion
480	1.10	22.5	1.1
	2.10	9.75	0
	3.10	19.85	20.3
720	1.11	27.25	2.6
	2.11	24.45	4.5
	3.11	24.25	27.35
1000	1.12	34.75	3.4
	1.13	34.75	0.55
	2.12	34.75	9.8
	2.13	24.65	4.55
	3.12	34.75	34.75
	3.13	34.75	34.75

The evaluation of the degree of delamination around the section in Table 3 shows that all samples with different pre-treatments show very similar properties, only sample 2.10 with ferric phosphate after 480 hours in neutral salt spray had 2 times less delamination than the other two types of samples. The same ferric phosphate sample also performed best in the corrosion assessment around the cut after 480 hours. The best results from the corrosion test were found on a surface with zircon-based nano passivation. The worst levels of under- corrosion were found in samples with pre-treatment in the form of surface blasting. In these samples, corrosion occurred all over the surface after 1000 hours in the corrosion chamber, as shown in Figure 5.

Fig. 5. Photographs of samples after an exposure time of 1000 hours in the Salt chamber. From the left, a sample with nano passivation pre-treatment, in the middle a sample with phosphating pre-treatment and on the right a sample with pre-treatment by blasting (Tlaskal 2022).

Another test of the powder plastic coating to the base material was a tear test according to (ČSN EN ISO 4624). This test indicates how much stress the joint can withstand, in this case it is the cohesion of the powder plastic to the metal material or bottom layer created in the pre-treatment. Using the obtained values, samples can be compared with different pre-treatments (Figure 6). By help to the tear adhesion test according to standard ČSN EN ISO 4624, the following information about different coating behavior under different surface pre-treatments was obtained. From the measured values, the average strength value was obtained, when the joint broke. All three different types of samples received almost the same numbers. For a zircon-based nano passivation coating it was 8.50 MPa, for ferric phosphating it was 9.24 MPa and the blasted surface showed an average strength of 8.51 MPa.

Fig. 6. Sample No 3.7 after tear test.

The results of present experiments had the high quality in consequence of applying the risk management using the Table 1 and Table 2; the risk rate was 12%, i.e. low risk rate (Tlaskal 2022).

6.2. *Recommendation for Practice*

The first experiments with the application of surface pre-treatments showed that the results of the experiments were greatly influenced by the quality of individual operations and very by external conditions; when repeating the same experiments, a significant dispersion of results were observed (Tlaskal 2022).

Because our goal is to create a methodology for practice, which it needs reliable results, we applied the safety management process and the dispersion of results was significantly reduced for all the methods used.

Based on the above-mentioned results, we are preparing a methodology for industrial practice based on process management safety. For experimenting in laboratories, we obtained lessons learned that we must always consider that the sequence and quality of operations during the experiment and especially external conditions very significantly affect the results. Therefore, to obtain reliable experiment results, the principles of risk management aimed to safety must be applied. In this direction, we introduce special lessons for students in our university.

7. Conclusion

The aim of present study was:

- to compare individual types of surface pre-treatment by powder plastic coatings,
- to find principles for methodology for using this procedure in practice.

The evaluation of experiment results shows:

- the degree of delamination ended approximately the same for all samples,
- the best results from the corrosion test were found on a surface with zircon-based nano passivation,
- the worst levels of corrosion were found in samples with pre-treatment in the form of surface blasting,
- method of surface pre-treatment by powder plastic coatings must be chosen according to real application in practice.

In all cases for obtaining the reliable results of surface pre-treatment process by powder coatings in practice it is necessary to respect principles process safety management, which are based on management of risks connected with process. It is necessary to follow both, the partial risks and the integral risk evaluated by checklist. Therefore, in this direction we create methodology for industrial practice.

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